INDUSTRIAL
HEATING

A COMPREHENSIVE GUIDE TO HEAT TREATMENT

VOLUME 2

A collection of articles by Dan Herring,
The Heat Treat Doctor®

A SPECIAL MESSAGE FROM DAN HERRING, THE HEAT TREAT DOCTOR®

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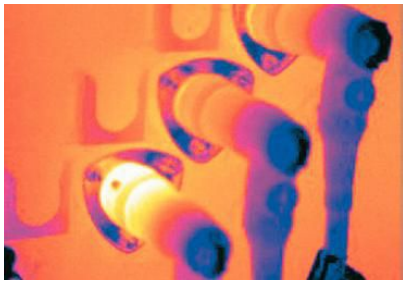

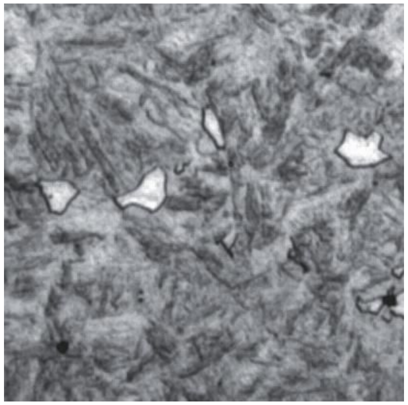

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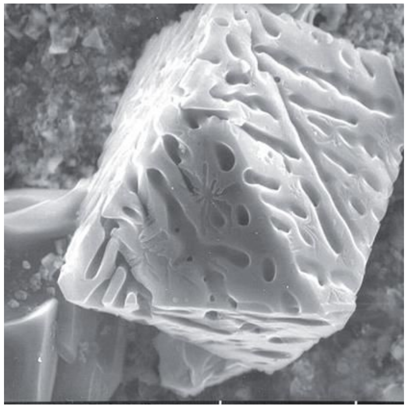

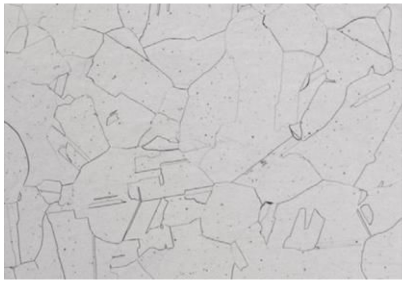

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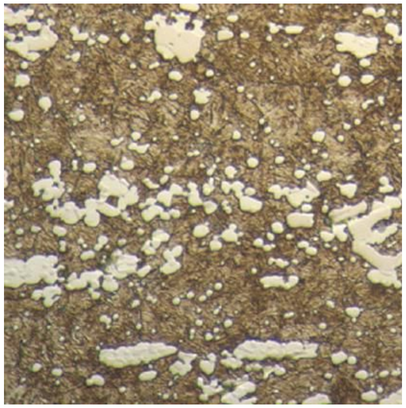

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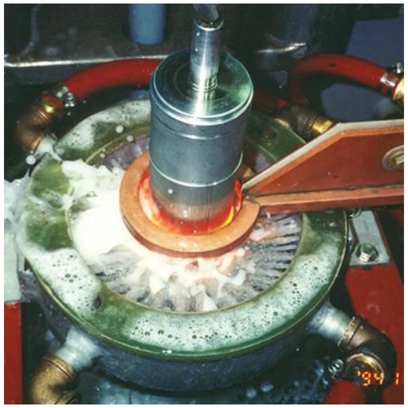

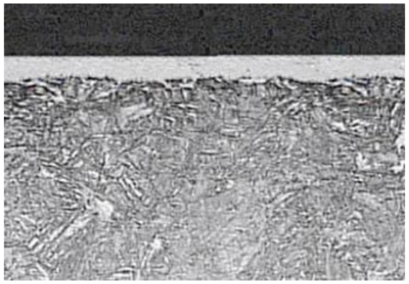

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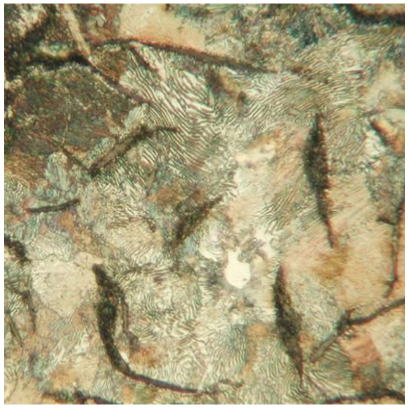

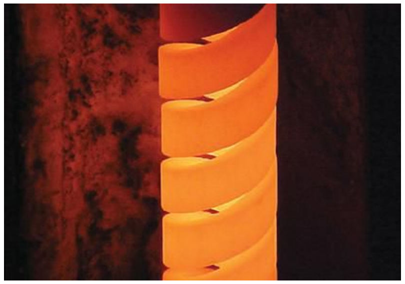

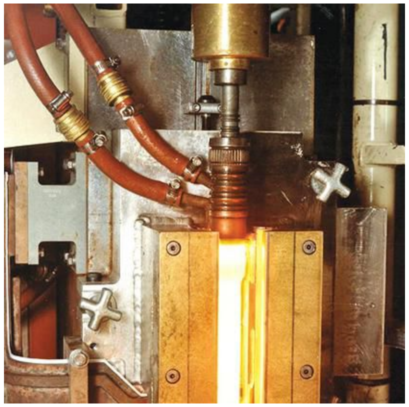

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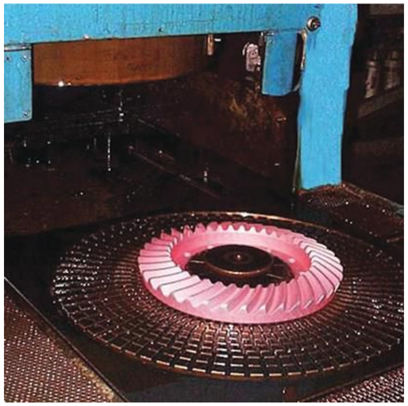

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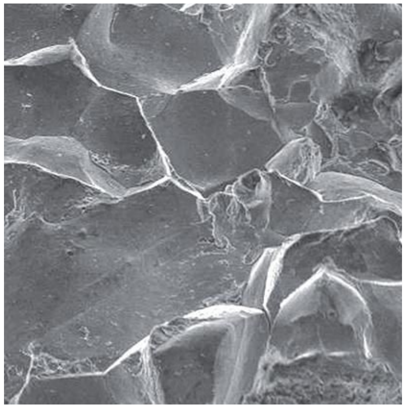

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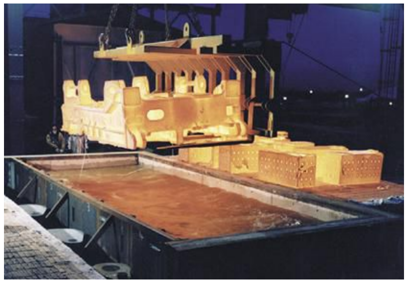

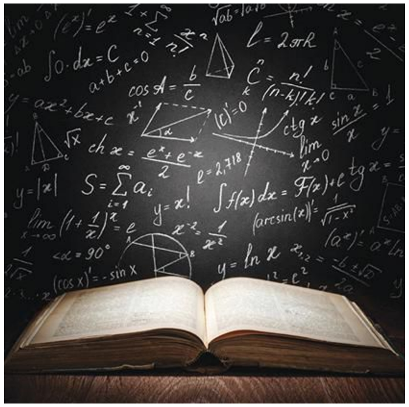

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A Heat Treater's Book Guide

There is an old adage that says, "If you don't know it, know where to find it." Given the information age in which we live, with the Internet at our fingertips and "ask Siri" or "Alexa, what is…" at our beck and call, solving problems and getting answers to our questions seems faster and easier than ever before. As heat treaters, metallurgists and engineers, however, we know the information we find must be filtered, checked and verified as to its accuracy and applicability. As such, we must also have reference books available to us. Let's learn more.

When it comes to understanding any subject, and heat treatment in particular, having sources you can trust is invaluable. Establishing a good technical reference library (Fig. 1), whether at work or at home (or both), is a good way to research the information you find, because solving everyday problems requires both an understanding of the root cause and an understanding of the underlying principles behind it. In addition, one must be able to uncover the entire scope of technical issues confronting us so as to find practical solutions and know why a particular phenomenon took place so that corrective action may be taken to avoid its reoccurrence in the future.

Presented below are the "top 5" books in each category that are considered "must have" reference books. Newer editions may exist in some cases, but exercise caution to ensure their contents are equal to or better than the originals. A more comprehensive listing can be found in the Reference 1.

General Reference[1,2,3]

1. Herring, Daniel H., *A Comprehensive Guide to Heat Treatment, Volumes 1 & 2*, e-Books, Industrial Heating/BNP Media, 2018, (www.industrialheating.com)
2. *Handbook of Mechanical Alloy Design*, George E. Totten, Lie Xie and Kiyoshi Funatani (Eds.), Marcel Dekker Inc., 2004, ISBN 0-8247-4308-3, 734 pages
3. *Mark's Standard Handbook for Mechanical Engineers, 9th Ed.*, Eugene A. Avallone and Theodore Baumeister III (Eds.), McGraw Hill Book Co., 1978, ISBN 0-07-004127-X
4. *Metals Handbook, 1948 Ed.*, ASM International,

1948, 1,332 pages
5. Wegst, C. W., *Stahlschlussel (Key to Steel)*, 20th Ed., Verlag, 2004, ISBN 3-922599-20-6, 720 pages

Atmosphere Heat Treating[1,2,3]

1. Herring, Daniel H., *Atmosphere Heat Treatment, Volume I*, BNP Media Group, 2014, ISBN 978-0-692-28393-6, 714 pages
2. Herring, Daniel H., *Atmosphere Heat Treatment, Volume II*, BNP Media Group, 2015, ISBN 978-0-692-51299-9, 803 pages
3. *Steel Heat Treatment Handbook: Equipment and Process Design, 2nd Ed.*, George E Totten (Ed.), CRC Press, 2007, ISBN 978-0-8493-8454-7, 713 pages
4. Parish, Geoffrey, *Carburizing: Microstructure and Properties*, ASM International, 1999, ISBN 0-87170-666-0, 247 pages
5. Haga, L. J., *Practical Heat Treating*, Metal Treating Institute

Furnace Atmospheres – Generation (old but relevant)[11,12]

1. *Metals Handbook, Vol. 2, Heat Treating, Cleaning and Finishing, 8th Ed.*, ASM International, 1964, LOC 27-12046, 708 pages
2. Hotchkiss, A. G. and H. M. Webber, *Protective Atmospheres*, John Wiley & Sons, 1953, LOC 53-8521, 341 pages

Fig. 1. A few bookshelves with some of The Doctor's favorite reference books.

Vacuum Heat Treating[1,2,3,14]

1. Herring, Daniel H., *Vacuum Heat Treating, BNP Media*, 2014, ISBN 978-0-9767565-0-7, 512 pages
2. Herring, Daniel H., *Vacuum Heat Treating Volume II*, BNP Media, 2016, ISBN 978-0-692-76738-2, 1,076 pages
3. *Vacuum Technology: Practical Heat Treating and Brazing*, Roger Fabian (Ed.), ASM International, 1993, ISBN 0-87170-477-3, 253 pages

4. Brunner Jr., William F. and Thomas H. Batzer, *Practical Vacuum Techniques*, Robert E. Krieger Publishing Co., 1974, ISBN 0-88275-146-8, 198 pages
5. *The Vacuum Technology Book*, Pfeiffer Vacuum Inc., 2008

Applied Energy[1,2,3,8]

1. Haimbaugh, Richard E., *Practical Induction Heat Treating*, ASM International, 2001, ISBN 0-87170-743-8, 332 pages
2. Rudnev, V., D. Loveless, R. Cook, and M. Black, *Handbook of Induction Heating*, Marcel Dekker, 2003
3. *ASM Handbook, Volume 4C: Induction Heating and Heat Treatment*, Valery Rudnev and George E. Totten (Eds.), ASM International, 2014, ISBN 978-1-62708-012-5, 820 pages
4. Tudbury, Chester A., *Basics of Induction Heating, Vol. 1 and 2*, 1960, LOC 60-8958, 132 pages, (Vol. 1) and 121 pages, (Vol. 2)
5. Semiatin, S.L. and D. E. Stutz, *Induction Heat Treatment of Steel*, ASM International, 1986, ISBN 978-0-087170-21101, 308 pages.

Quenching[1,2,3,7,9,13]

1. Totten, G. E., C. E. Bates and N. A. Clinton, *Handbook of Quenchants and Quenching Technology*, ASM International, 1992, ISBN 0-87170-448-X, 507 pages
2. Lišcˇic´, B. and H.M. Tensi, L.C.F. Canale and G.E. Totten, *Quenching Theory and Technology 2nd Ed.*, CRC Press, 2010, ISBN 978-0-8493-9279-5, 725 pages
3. N.I. Kobasko, M.A. Aronov, J. Powell and G.E. Totten, *Intensive Quenching Systems: Engineering and Design*, ASTM International, 2010, ISBN 978-0-8031-7019-3, 253 pages
4. *Theory of Quenching and Quenching Technology: A Handbook*, B. Lišcˇic´, H. M Tensi, and W. Luty (Eds.), Springer-Verlag,1992, ISBN 978-36620-1598-8, 484 pages
5. Totten, G.E. and D.S. MacKenzie, , *Handbook of Aluminum: Vol. 1 - Physical Metallurgy and Processes*, Marcel Dekker Inc., 2003, ISBN 978-0-8247-0494-0 (c.f. Chapter 20 on Aluminum Quenching), 1,310 pages

Heat Treatment (Practice)[1,2,3]

1. *Heat Treater's Guide: Practices and Procedures for Irons and Steels, 2nd Ed.*, Harry E. Chandler (Ed.), 1995, ASM International, ISBN 0-87170-520-6, 903 pages
2. Herring, Daniel H., *A Comprehensive Guide*

to Heat Treatment, Volumes 1 & 2, e-Books, Industrial Heating/BNP Media, 2018, (www.industrialheating.com)
3. *Steel and Its Heat Treatment: Bofors Handbook*, Karl-Erik Thelning (Ed.), Butterworths, 1973, ISBN 0-408-70651-1, 570 pages
4. Bullen D.K., and the metallurgical staff of Battelle Memorial Institute, *Steel and Its Heat Treatment, Vol. I: Principles, Processes, Control*, John Wiley & Sons, 1938, 445 pages
5. Bullen D.K., and the metallurgical staff of Battelle Memorial Institute, *Steel and Its Heat Treatment, Vol. II: Engineering and Special Purpose Steels*, John Wiley & Sons, 1939, 491 pages

Heat Treatment (Theory)[1,2,3]

1. Krauss, G., *Steels: Heat Treatment and Processing Principles*, ASM International, 1990, ISBN 0-87170-370-X, 497 pages
2. *ASM Handbook, Volume 4A: Steel Heat Treating: Fundamentals and Processes*, Jon L. Dossett and George E. Totten (Eds.), ASM International, 2013, ISBN 978-1-62708-011-8, 768 pages
3. *ASM Handbook, Volume 4B: Steel Heat Treating Technologies*, Jon L. Dossett and George E. Totten (Eds.), ASM International, 2014, ISBN 978-1-62708-025-5, 528 pages
4. *ASM Handbook, Volume 4D: Heat Treating of Irons and Steels*, Jon L. Dossett and George E. Totten (Eds.), ASM International, 2014, ISBN 978-1-62708-066-8, 641 pages
5. Grossmann, M. A. and E. C. Bain, *Principles of Heat Treatment, 5th Ed.*, ASM International, 1964, 302 pages
6. *Steel Heat Treatment Handbook: Metallurgy and Technologies, 2nd Ed.*, George E Totten (Ed.), CRC Press, 2007, ISBN 978-0-8493-8455-4, 831 pages
7. Burgess C. O., *Heat Treating of Gray Iron*, Gray Iron Founders' Society, Inc., 1984, 121 pages.

Ferrous Metallurgy[1,2,3]

1. Krauss, George, *Steels: Processing, Structure, and Performance*, ASM International, 2005, ISBN 0-87170-817-5, 613 pages
2. *Making, Shaping and Treating of Steel, The 9th Ed.*, Harold E. McGannon (Ed.), AISE (United States Steel Corp.), 1971, 1,420 pages
3. Llewellyn, D. T. and R. C. Hudd, *Steels, Metallurgy & Applications, 3rd Ed.*, Butterworth Heinemann, 1998, ISBN 0-7506-3757-9, 389 pages
4. *Mechanical Properties of Alloy Steel: Republic*

Alloy Steels Handbook, Booklet Adv. 1009, 1961, 84 pages

5. *Alloying Elements and Their Effect on Hardenability: Republic Alloy Steels Handbook*, Booklet Adv. 1009, 1961, 46 pages

Transformation Diagrams[1,2,3]

1. Atkins, M., *Atlas of Continuous Cooling Transformation Diagrams for Engineering Steels*, ASM International, 1977, ISBN 0-87170-093-X, 260 pages
2. *Atlas of Isothermal Transformation Diagrams*, United States Steel Corp., 1951, 143 pages
3. *I–T Diagrams: Isothermal Transformation of Austenite in a Wide Variety of Steel, 3rd Ed.*, United States Steel Corporation, 1963, 183 pages
4. *Atlas of Isothermal Transformation Diagrams (Supplement)*, United States Steel Corp., 1953, 529 pages
5. *Atlas of Time-Temperature Diagrams for Irons and Steels*, George F. Vander Voort (Ed.), 1991, ASM International, ISBN 0-87170-415-3, 766 pages
6. *Atlas of Time-Temperature Diagrams for Nonferrous Alloys*, ASM International, 1991

Nonferrous Metallurgy (Aluminum)[1,2,3,7,13]

1. *Aluminum, Properties and Physical Metallurgy*, John E. Hatch (Ed.), ASM International, 1984, ISBN 0-87170-176-6, 424 pages
2. *Handbook of Aluminum, Vol. 1: Physical Metallurgy and Processes*, George E. Totten and D. Scott Mackenzie (Eds.), Marcel Dekker, Inc., 2003, ISBN 0-8247-0494-0, 1,296 pages
3. *Handbook of Aluminum, Vol. 1: Alloy Production and Materials Manufacturing*, George E. Totten and D. Scott Mackenzie (Eds.), Marcel Dekker, Inc., 2003, ISBN 0-8247-0896-2, 1,296 pages
4. *Aluminium-Schlüssel: Key to Aluminium Alloys, 7th Ed.*, Dr.-Ing. Werner Hesse (Ed.), Aluminium-Verlag Marketing & Kommunikation GmbH, Düsseldorf, 2011 (in German and English)
5. *Heat Treater's Guide: Practices and Procedures for Nonferrous Alloys, 2nd Ed.*, Harry Chandler (Ed.), ASM International, 1996, ISBN 978-0-87170-565-5, 669 pages

Nonferrous Metallurgy (Other)[1,2,3,13]

1. Polmear, I. J., E. Light Alloys: *Metallurgy of the Light Metals*, Arnold Publisher, 1990, ISBN 0-34049-175-2, 278 pages
2. *Heat Treater's Guide: Practices and Procedures*

for Nonferrous Alloys, 2nd Ed., Chandler, Harry E. (Ed.), ASM International, 1996, ISBN 0-87170-565-6, 669 pages
3. *Superalloys, A Technical Guide, 2nd Ed.*, M. J. Donachie and S. J. Donachie (Eds.), ASM International, 2002, ISBN 0-87170-749-7, 439 pages
4. Sims, Chester T. and William C. Hagel, *Superalloys, The.*, John Wiley & Sons, 1972, ISBN 0-471-79207-1, 614 pages
5. *Titanium, A Technical Guide*, Matthew J. Donachie Jr. (Ed.), ASM International, 1988, ISBN 0-87170-309-2, 469 pages

Powder Metallurgy[1,2,3,15]

1. MPIF 35 P/M Structural, *Material Standards for PM Structural Parts*, Metal Powder Industries Federation, 2016
2. German, Randall M., *Sintering Theory and Practice*, John Wiley & Sons, 1996, ISBN 0-471057-86-X, 550 pages
3. German, Randall M., *Powder Metallurgy of Iron and Steel*, John Wiley & Sons, 1998, ISBN 0-471-15739-2, 496 pages
4. *High Temperature Sintering*, Howard I. Sanderow (Ed.), Metal Powder Industries Federation (MPIF), ISBN 0-918404-97-5, 400 pages
5. German, R.M. and Bose, A., *1997: Injection Molding of Metals and Ceramics*, Metal Powder Industries Federation, ISBN 978-1-87895-461, 413 pages

Joining (Brazing)[1,2,3,4]

1. *Brazing Handbook, 5th Ed.*, American Welding Society, 2007, ISBN 978-08717-104-6-8, 740 pages
2. Peaslee, Robert L., *Brazing Footprints, Case Studies in High Temperature Brazing*, Wall Colmonoy Corporation, 2003, ISBN 0-9724479-0-3, 299 pages
3. Schwartz, Mel M., *Brazing*, ASM International, 1967, ISBN 0-87170-246-0, 455 pages
4. *ASM Handbook, Volume 6: Welding, Brazing and Soldering, 10th Edition*, ASM International, 1993, ISBN 978-0-87170-382-8, 1,299 pages
5. Roberts, Phillip M., *Industrial Brazing Practice, 2nd Ed.*, CRC Press, 2013, ISBN 146-65677-4-0, 460 pages
6. *Aluminum Brazing Handbook, 4th Ed.*, The Aluminum Association, Inc., 1998, 84 pages

Joining (Soldering, Welding)[1,2,3,4]

1. Manko, Howard H., *Solders and Soldering*, McGraw-Hill Book Company, 1964, ISBN 07-0-

39895-X, 323 pages
2. Linnert, George E., *Welding Metallurgy; Carbon and Alloy Steels Vol.1 Fundamentals*, 4th Ed., American Welding Society, 1995, 478 pages

Tool Steels[1,2,3]

1. Roberts, G. A., and J. C. Hamaker Jr., A. R. Johnson, *Tool Steels, 3rd Ed.*, 1962, ASM International, 780 pages
2. Wilson, Robert, *Metallurgy and Heat Treatment of Tool Steels*, McGraw-Hill Book Company, 1975, ISBN 0-07-084453-4, 378 pages
3. Payson, Peter, *The Metallurgy of Tool Steels*, John Wiley & Sons, 1962, LOC 62-16155, 349 pages
4. Palmer, Frank R. and George V. Luerssen, *Tool Steel Simplified*, Carpenter Technology Corp., 1977, ISBN 0-31055-805-6, 555 pages
5. *The Tool Steel Trouble Shooter: An Analysis of 107 Tool Failures*, Bethlehem Steel Corp., 1964, 125 pages

Heating Methods (Electric & Gas)[1,2,3,6]

1. *NFPA 86 Standard for Ovens and Furnaces*, National Fire Protection Agency [latest edition]
2. Reed, Richard J., *North American Combustion Handbook, Vol. 1, 2nd Ed.*, North American Mfg. Co., 1978, ISBN 0-96015696-1-4, 501 pages
3. Reed, Richard J., *North American Combustion Handbook, Vol. 2, 2nd Ed.*, North American Mfg. Co., 1986, ISBN 978-0960-159-63-5, 501 pages
4. *IHEA Heat Processing Manual*, Industrial Heating Equipment Association [latest edition]
5. *Combustion Technology Manual*, Industrial Heating Equipment Association [latest edition]

Metallurgy (Theory)[1,2,3]

1. Brandt, Daniel A. and J. C. Warner, *Metallurgy Fundamentals, 4th Ed.*, Goodheart-Willcox Co. Inc., 1999, ISBN 1-56637-543-6, 256 pages
2. *Practical Data for Metallurgists*, 19th Ed., The Timken Co., 2017, 100 pages
3. Dieter Jr., George E., *Mechanical Metallurgy*, McGraw-Hill Book Company, 1961, LOC 61-11385, 615 pages
4. *Metallurgy for the Non-Metallurgist*, Harry E. Chandler (Ed.), ASM International, 1998, ISBN 0-87170-652-0, 284 pages
5. Reed-Hill, Robert E., *Physical Metallurgy*, D. Van Nostrand Co. Inc., 1964, 630 pages
6. Bamford, T. G. and H. Harris, *The Metallurgist's Manual, (8 Volumes)*, Chapman & Hall Ltd., 1937, 256 pages

Metallurgy (Practice)[1,2,3]

1. *Martensite*, G. B. Olson and W. S. Owen (Eds.), ASM International, 1992, ISBN 0-87170-434-X, 331 pages
2. Bhadeshia, H. K. D. H., *Bainite in Steels, The Institute of Materials*, 1992, ISBN 0-901465-95-0, 451 pages
3. *Boron in Steel*, S. K. Banerji, and J. E. Morral (Eds.), The Metallurgical Society of AIME, 1980, ISBN 0-89520-363-4, 215 pages

Metallography[1,2,3,5]

1. *Metals Handbook, Vol. 7, Atlas of Microstructures of Industrial Alloys, 8th Ed.*, ASM International, 1972, LOC 27-12046, 366 pages
2. Vander Voort, George F., *Metallography Principles and Practice*, ASM International, 1984, ISBN 0-87170-672-5, 752 pages
3. *Applied Metallography*, George F. Vander Voort (Ed.), Chapman & Hall, 1986, ISBN 0-042228-836-0, 301 pages
4. Samuels, L. E., *Light Microscopy of Carbon Steels*, ASM International, 1999, ISBN 0-87170-655-5, 502 pages
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Final Thoughts

Readers are encouraged to contact the writer (dherring@heat-treat-doctor.com) with other reference books that they have found to be particularly helpful in solving heat-treating, metallurgy and engineering problems associated with the subjects listed (or others). As we update the list in the future, our library will continue to grow to the benefit of all.

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What is Good Heat Treating? A Look Ahead…

I believe we can all agree that heat treating should strive to produce the best product quality possible (Fig. 1). But just how do we go about doing this, in other words, how does one define "good" heat treating? Let's learn more.

First, it is important to realize that there are many types of heat treatments and that each may require a different characterization of "good." Quality heat treating could refer (in no particular order) to:

- Technique – Be a master of what you do, use existing methods wisely and develop new tools to accomplish the task at hand faster, easier and more efficiently.
- Insight – Strive to achieve a major process or product breakthrough, conceptualize and simplify, understand the underlying principle, technique or approach.
- Theory – Use a systematic scientific approach to unify and generalize the existing knowledge in our field and to speculate on new ideas or innovations.
- Problem solving – Understand root cause and strive to achieve a major breakthrough on an important problem facing the industry.
- Discovery – Find the relevance of an unexpected and intriguing new heat-treat phenomenon, connection or method.
- Taste – Keep heat treating interesting and vibrant while dealing with the mundane. Always be aware of the bigger picture, the important topics, questions and solutions.
- Application – Extend the relevance of our science to other fields to further their advancement.
- Exposition – Promote a detailed and informative understanding of a relevant heat-treating method or approach.
- Pedagogy – Write and teach so as to enable others to learn and do heat treating more effectively, contribute to good heat-treatment education.
- Vision – Be aware of the long-term implications of what is being done today and think "out of the box" whenever possible.
- Public relations – Be an ambassador for who we are and what we do. Showcase our successes, address the challenges that face the industry and emphasize the rewards of what we do.

Fig. 1. Alloy bar stock exiting a roller hearth furnace. *(Photo Courtesy of SECO/WARWICK Corporation)*

Next, we must understand that it is extremely important to share our knowledge throughout the industry and to embrace the following criteria for heat-treating excellence (again, in no particular order):

- Rigor – In everything we do, be correct with all the details, think the process through, understand the consequences and be complete.
- Beauty – Never stop being amazed with what we can accomplish, and never fail to take on the difficult challenges so as to advance our science.
- Usefulness – Be sure that what we do today will allow the next generation of heat treaters to work smarter, faster and be more accurate.
- Creativity – Do not back away from radically new or original ideas, never stop questioning the status quo.
- Strength – Offer solutions based on a sound understanding of both scientific principles and solid practical experience.
- Elegance – Strive to achieve difficult results with a minimum of effort.
- Growth – Grow intentionally and avoid haphazard growth not rooted in sound thinking.
- Profitability – Make a profit so as to fuel continued growth.
- Cost – Control and contain cost wherever possible without being so constrictive that R&D programs or investments in new technology or equipment limit growth.
- Savings – Do the easy things quickly and efficiently, and tackle the hard things in such a way as to minimize rework. Be sure that savings are real and quantifiable.
- Conservationism – Be good stewards of those things that really matter, such as energy and the environment, to keeping our industry strong.
- Cherishment – Passing on our heritage, our history and our practices to the next generation of heat treaters.

The diversity and multifaceted nature of heat treating is very healthy for the manufacturing community as a whole, as it allows us to pursue many different solutions to the challenges we face and exploit many different ways in which to accomplish a given task – along with encouraging different types of heat-treating talent – from the common sense shop-floor heat-treat operator to the academic. Our individuality is a desirable trait, but we must be aware that it can become detrimental to pursue only one or two of our quality goals at the expense of the others. What we must be careful to avoid is:

- Heat treating that becomes increasingly ornate and baroque, in which individual results are generalized and refined for their own sake and not the common good. In other words, the industry drifts aimlessly without a definitive direction or sense of progress.
- Heat treating that becomes filled with "rules of thumb" and methods or beliefs without rigor, documentation or an understanding of the scientific principles on which they are founded.
- Heat treating that consists of ad hoc methods (in other words, for self-centered purposes) to solve a problem or collection of problems that have no unifying theme, connections or purpose.
- Heat treating that becomes stagnant and overly theoretical or overly practical, continually recasting and unifying previous results in increasingly technical or formal frameworks but not generating any exciting new breakthroughs as a consequence.
- Heat treating that reverses or rejects methods or accomplishments just because they were "not invented here or now" and continually presents shorter, simpler or more elegant ideas and explanations of results but does not generate any truly original ideas and new results beyond what is already known.

In each of the scenarios above, the fear is that heat treating will exhibit a flurry of activity and only progress in the short term. The risk of a decline of relevance, a failure of competitiveness or a failure to attract younger engineers to the industry in the longer term is unfounded. Fortunately, it is a hard field to stagnate in, given that we are constantly being challenged and reinvigorated by our connection to the rest of science and to the manufacturing community as a whole. This self-correcting mechanism helps to keep heat treating and metallurgy balanced, unified, productive and vibrant.

Be the Best of the Best

The very best examples of sound heat-treating practices do not merely fulfill one or two of the criteria for heat-treating excellence given at the beginning of this article, but are more importantly part of a greater story, which then unfurls to generate many further examples of good heat-treating practice in vastly diverse areas. Indeed, from a historical context, the evolution of our craft through time and the interactions of heat treaters with the entire manufacturing, academic and engineering community ensures success. Good heat treatment is not merely measured by one or more of the "local" qualities listed previously (though these are certainly important and worth pursuing and debating), but also must be considered in the grand or "global" scheme of things. How does good heat treating fit into the bigger picture, either by building upon earlier achievements or encouraging the development of future breakthroughs?

One Man's Opinion

It seems to me that the pursuit of intangible promises of some far-distant future is at least as important as any aspect of heat treating in the here and now. As we continue to strive unabated toward the more concrete and obvious aspects of heat-treat quality listed above, we must not sacrifice our vision of what lies ahead. Thus, I believe that good heat treatment is more than simply a matter of running a particular process or solving the immediate problem before us. It is about continuing to build theories, mentoring the next generation and educating those who are here now and those who are soon to come while making arguments for improvements in materials, controls, equipment, methodology and the like. Achieving all of these tasks and debating which ones should have higher priority or are more deserving of funding should occupy an inordinate amount of our mental energies. We should always be aware of a possibly larger context into which one's results could be applied.

Lastly, remember to communicate – from the shop floor to the halls of academia. Jealously guarded secrets and hidden knowledge are counterproductive and limit our ability to achieve the "critical velocity" necessary for the heat-treating industry to remain competitive in a global economy. We must always remember to offer our customers the best and most cost-effective technology but never forget that although necessity is what has driven them to us, it is our performance, our vision and our science that will keep them coming back. I still "see" a furnace without walls and a heat-treating process taking place before my very eyes employing intelligent sensors, allowing us to make real-time decisions that will produce the perfect part. Until that time, "good" heat treating is what we need.

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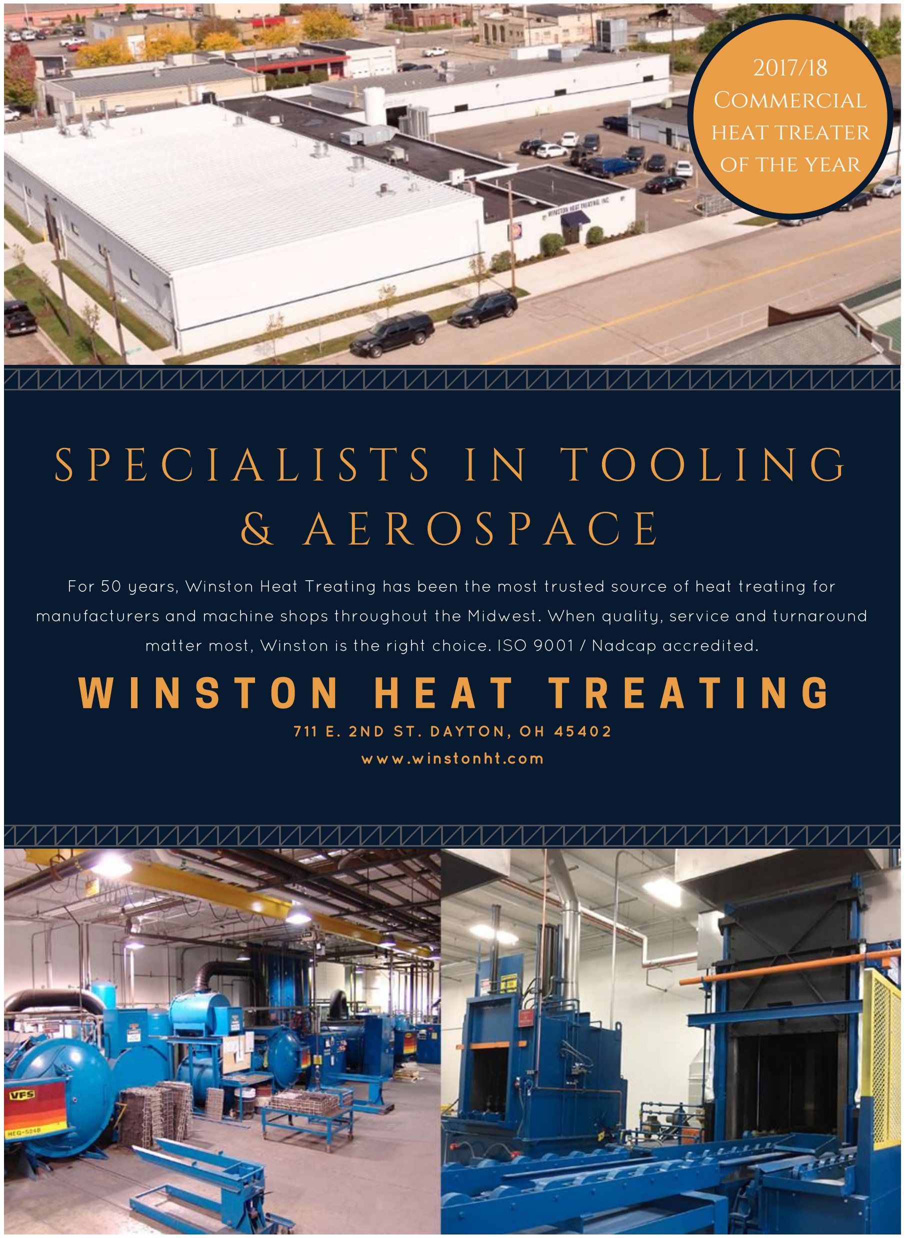
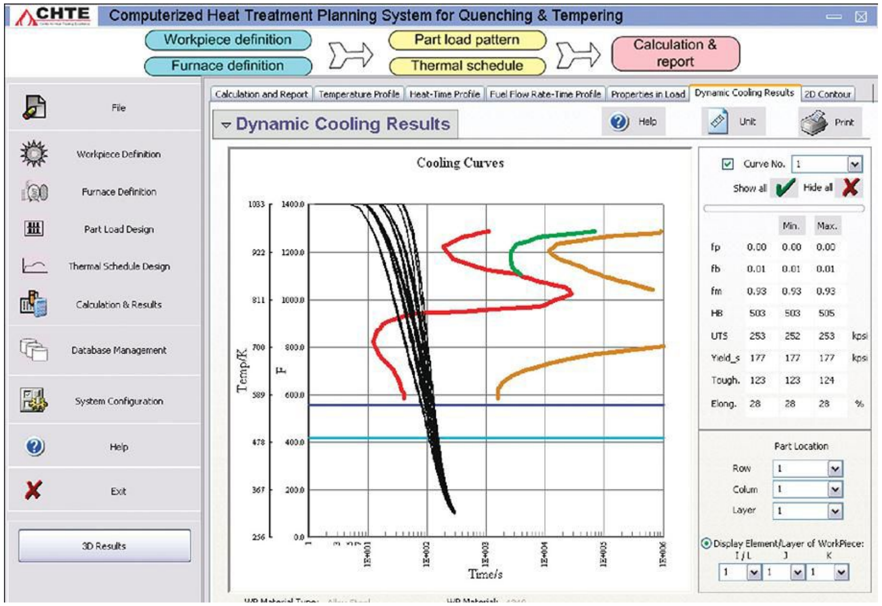


Improving the Heat Treating Process

Heat treating is alive and well, a $20 billion-a-year industry that continues to grow and prosper despite a changing manufacturing base and increased global competition. In the short term, there are many resources available to help the heat treater do his job. You might be asking yourself as I have about who is looking out for our future? Let's learn more.

Academic industry partnerships

One of the groups that looks ahead and is working to find ways to improve the heat treating process is the Center for Heat Treating Excellence (CHTE), established at Worcester Polytechnic Institute in 1999 (Worcester, Mass.; www.wpi.edu/academics/research/chte). The center is an alliance between heat treaters and university researchers to collaborate to identify and address short- and long-term needs of the heat treating industry. The center's goal is to advance heat treating technology by applying fundamental research to solve real-world problems. A unique aspect of this and similar alliances is that the research projects are industry-member driven, so the work directly benefits heat-treating industry profitability, know-how and public image.



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		Min.	Max.	
fp	0.00	0.00	0.00	
fb	0.01	0.01	0.01	
fm	0.93	0.93	0.93	
HB	503	503	505	
UTS	253	252	253	kpsi
Yield_s	177	177	177	kpsi
Tough.	123	123	124	
Elong.	28	28	28	%

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Fig. 1. Heat treating planning system

CHTE maintains a dialogue with various technical societies, government agencies and other research facilities to ensure that the needs of the heat treating community are well understood, and it continually communicates its research results to the industry as a whole through public forums and via the Internet.

To help the heat treating industry maintain its position as a cost-competitive technology, the center pursues research and develops innovative processes related to:

- Energy-efficient equipment
- Processing with minimal part distortion
- Diffusion related process optimization
- Environmental friendly by-products/emissions
- Adaptability/flexibility for advanced materials
- Process control and intelligent sensor development
- Outcome prediction based on heat treating modeling and simulation
- Equipment/process integration into manufacturing

Examples of active research projects

Programs underway at CHTE cover a broad spectrum of heat treating interests as illustrated in the following examples.

Making the heat treating process more efficient. The goal of this project is modeling and simulation of heat treating processes in batch and continuous furnaces. By looking at loading patterns of parts and thermal cycling of furnaces, this project helps to optimize a given heat treating process by creating a heat treating planning system. The software combines heat-transfer data (convection, radiation and conduction), models the type of furnace being used and inputs materials information into a mini-package, which can be used on the shop floor in daily production (Fig. 1).

Today, these systems have been validated in more than 20 case studies and are now running at some CHTE-member companies. Successful application of the software has reportedly resulted in a 20% energy and cycle time saving through optimization.

Data mining is the process of discovering meaningful correlations, patterns and trends by sifting through large amounts of data using pattern recognition technology in combination with statistical and mathematical techniques. Traditionally, production of new materials and the development of new processes have been done by experiment or by trial and error either in the laboratory or in the heat treat shop. Costly and lengthy characterization of data follows to determine the validity of material properties and the interaction of process variables. Today, using data mining techniques and fast computers, the process can be vastly simplified.

At CHTE, a project involved with computational estimation of heat-transfer curves relies on data mining techniques to help build new graphs from old ones (Fig. 2). Existing experiments are clustered based on their resulting graphs. The clustering criteria

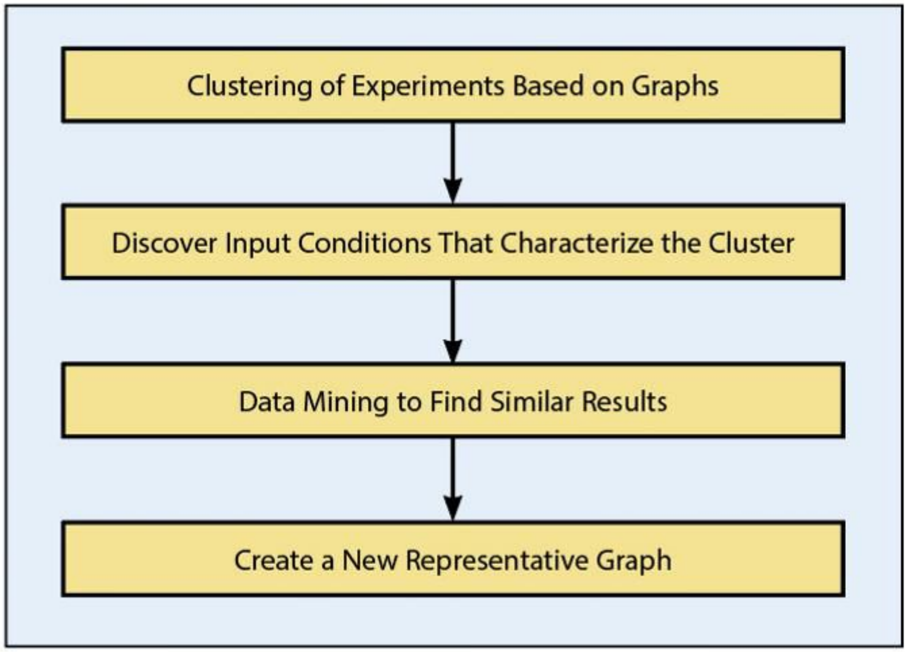

Fig. 2. Knowledge discovery and data mining

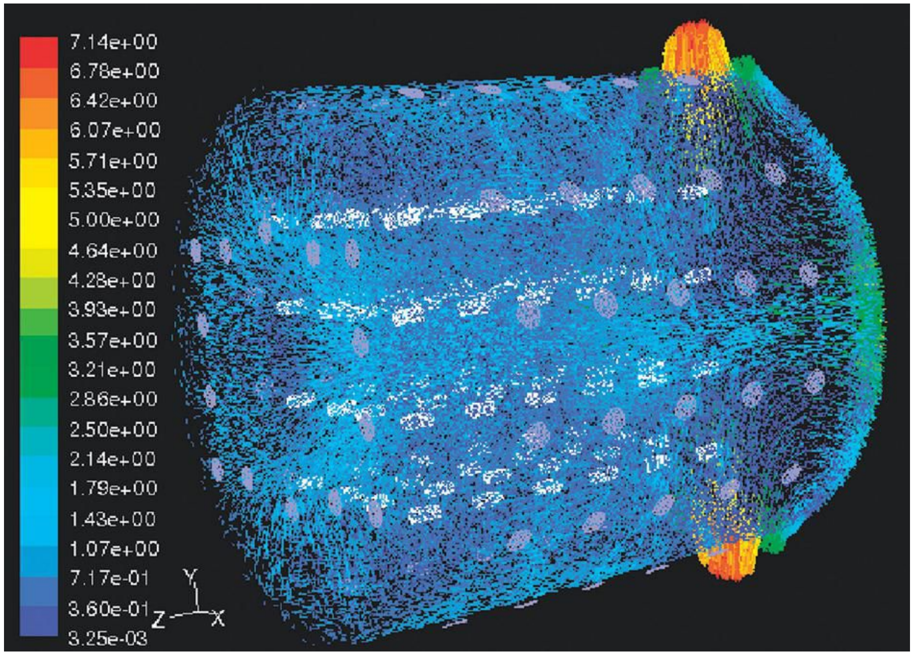

Fig. 3. Gas-flow simulation

is analyzed and applied to characterize the cluster, which represents the discovered knowledge. Data mining can then be applied so, for example, given the input conditions of an experiment, you can estimate the resulting graph, or given the desired graph in an experiment, you can estimate a set of input conditions that were used to obtain it.

Understanding high-pressure gas quenching. The goal of the CHTE gas-quenching project is to develop a theoretical and experimental understanding of the variables that control the high-pressure gas quenching (HPGQ) process. In addition, gas quenching capability is to be compared to that of other quenching media.

A combined theoretical-numerical-experimental approach has been employed to achieve the objectives. Numerical simulation was used to explore effects that are difficult to obtain by means of experimental testing. Computational fluid dynamics (CFD) simulation (Fig. 3) has been used to study the gas-flow field and the heat-transfer coefficient variation in the quench chamber using a commercially available software package (FLUENT from Fluent Inc., Lebanon, N.H., USA; www.fluent.com). A systematic study of the effects of key variables such as gas type, gas pressure, fan speed, alignment of parts and quench chamber geometry/type on heat transfer capability is now underway.

Material modeling to predict phase transformation, residual stress distribution, distortion and mechanical properties corresponding to quenching process defined by the key variables is being done using a finite element code (DANTE/ABAQUS, Deformation Control Technology Inc., Cleveland, Ohio; www.deformationcontrol.com). Finally, experimental tests are being performed to validate results from the theoretical analysis and the numerical simulation.

For further information on CHTE, contact Professor Diran Apelian (dapelian@wpi.edu).

Other academic/industry partnerships doing interesting work that might be of interest to heat treaters include Advanced Steel Processing and Products Research Center (ASPPRC) at the Colorado School of Mines (http://www.mines.edu/research/aspprc/) and The Thermal Processing Technology Center at the Illinois Institute of Technology (tptc.iit.edu).

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How to Best Communicate Your Needs to the Heat Treater

The goal of this article is to provide a structure for communicating your heat-treating requirements to your commercial heat treater (Fig. 1). It is written from the viewpoint of the company receiving the raw material stock or components for heat treatment, with the target audience being a manufacturer – big or small – who sends out steel products to be heat treated. Let's learn more.

Commercial heat treaters spend much of their time trying to clarify what their customer's expectations are on each order they process. Clearly defining the nature of the materials and the heat-treating requirements is the first step in making sure your heat treater will be able to meet or exceed your expectations. If possible, these requirements should be discussed with your heat treater before you start manufacturing the parts, not after they are final machined. It is also important to provide written documentation to the heat treater, including a duplicate copy sent with the materials/components, that represents the final agreed-upon treatments and specifications.

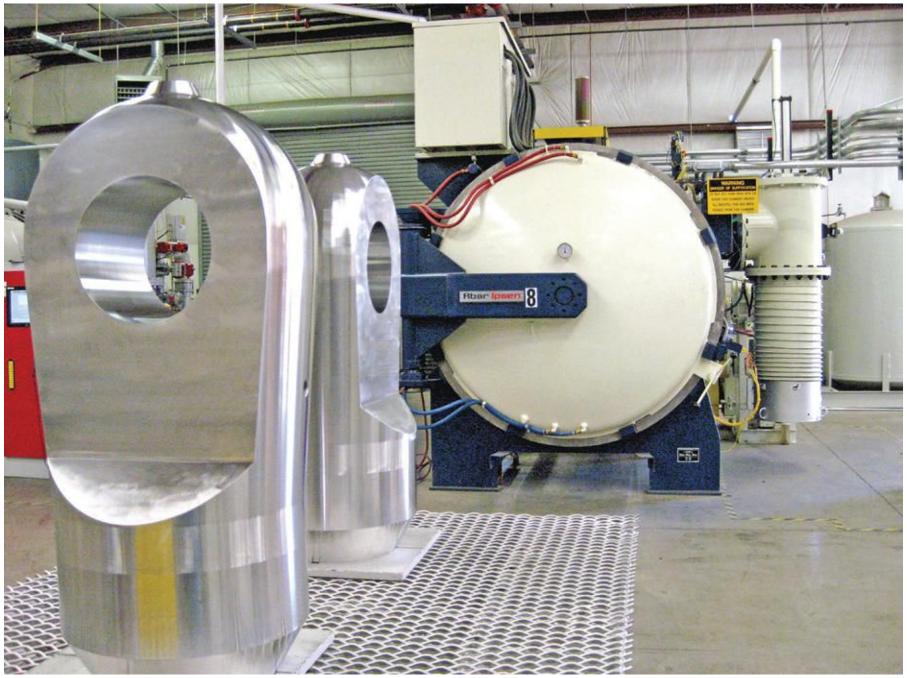


Fig. 1. Typical Commercial Heat Treat Shop

Material Grade

The first piece of information you need to supply is the material grade, typically the AISI or SAE number. Examples of these designations include 4140, 1144, 1095, 52100, H11, M4. Other common material specification systems, such as

ASTM, DIN and JIS, are also often used in today's global marketplace. Cross-references to AISI grades should be supplied if possible.

To avoid confusion, listing trade names in place of these designations should be avoided. Examples of material trade names are Bearcat™, Airkool™ and Shock-Die™. If these are provided, the specific manufacturer's data sheets should be included in the paperwork package.

The next most important document you can provide is the material certification sheet. This is provided to you by your material supplier and includes details such as the exact chemistry of the material being heat treated (including trace elements), grain size, cleanliness of the steel, prior processing and hardenability. These are invaluable aids for the heat treater to understand how to correctly process your material.

Condition Supplied

Most steels are supplied by the mill in the annealed condition. However, the material can also be supplied in other conditions such as normalized, normalized and tempered, or hardened. In addition, the material may have come to you from the mill as a sheet, bar, rod, forging or casting. It is critical that you notify the heat treater of the condition of the steel you are sending to them. If not, it is possible that the parts may not respond properly to the heat-treating process. For example, if you send parts made from 17-4PH to a heat treater to be processed to condition H-900 and the material was purchased in condition H-1150, it will not respond properly to the standard H-900 process. Your material supplier should notify you of the "as-supplied" condition in their material certification.

Instructions and Specifications

There are three types of instructions (purchase orders) heat treaters receive from their customers:

- Commercial practices
- Customer specifications
- Industry specifications

Examples of each of these are as follows:

- Commercial: 440C material, heat treat to 58-60 HRC
- Customer: 440C material, process per BPS-4602 (Bell Process Specification)
- Industry: 440C material, process per AMS 2759/5 (Aerospace Material Specification)

For a commercial purchase order, the heat treater is able to choose their own process (process temperatures, equipment type, atmosphere, soak durations, etc.). This often gives heat treaters the ability to combine orders and reduce processing time and cost. It is fair to argue that this type of purchase order gives the heat treater too much freedom.

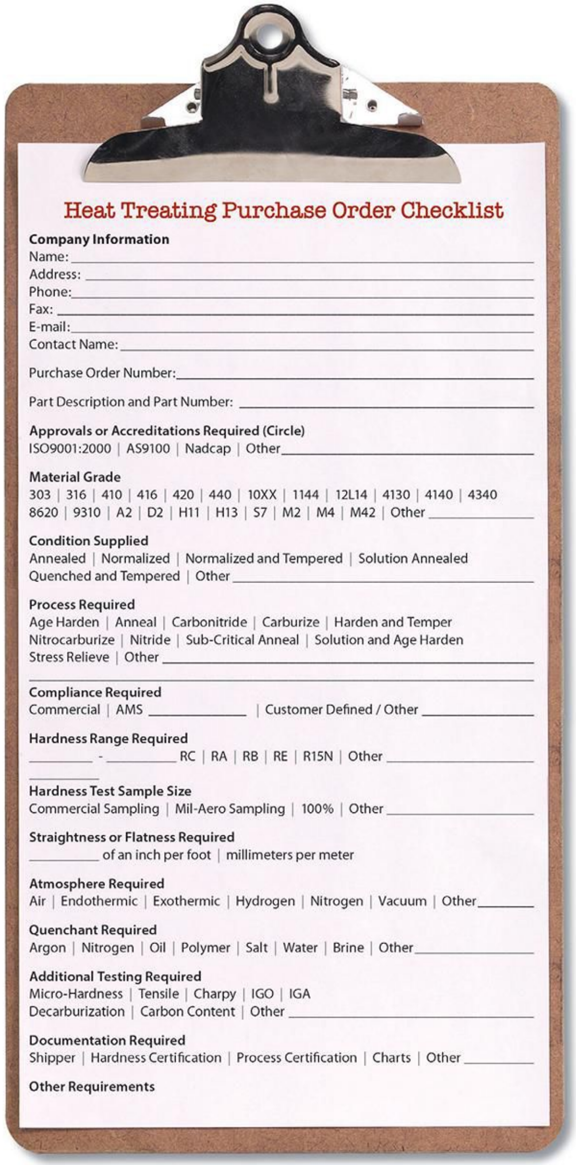
Purchase orders that call out specific customer or industry specifications are usually much more stringent. They may also bring other specifications and requirements into play as well. It is important for customers to realize that calling out a customer or industry specification on your purchase order may increase your heat-treating cost and lead time significantly.

For example, on a commercial order (Fig. 2), 440C may only require two processes (hardening and tempering) in order to meet a given hardness requirement. If the same 440C order is required to be processed per a customer or industry specification, it may require that the parts be subjected to multiple tempers and deep-freeze operations. It may also bring additional requirements (Fig. 4) into play (backfill gas dew point, furnace atmosphere, furnace pyrometry, instrument calibration, intergranular attack, decarburization, surface contamination, special documentation, additional destructive testing, larger hardness-testing sampling sizes, training, cooling rates, process-temperature setpoints, soak durations, etc.).

Fig. 2. Typical tool-and-die parts being prepared for heat treatment

Prints

It is extremely useful for your heat treater to have a copy of the current part drawing so that he can verify dimensions; note critical dimensions; understand the geometry of the part with respect to radii, sharp corners, ruling (thickest) section, the location of thin sections; surface-finish requirements and the like. Many customers also provide copies of their routers so that the heat treater can see how his processing fits into the overall scheme of the part manufacturing process. It is not uncommon for the heat treater to



Heat Treating Purchase Order Checklist

Company Information
Name: ______
Address: ______
Phone: ______
Fax: ______
E-mail: ______
Contact Name: ______

Purchase Order Number: ______

Part Description and Part Number: ______

Approvals or Accreditations Required (Circle)
ISO9001:2000 | AS9100 | Nadcap | Other ______

Material Grade
303 | 316 | 410 | 416 | 420 | 440 | 10XX | 1144 | 12L14 | 4130 | 4140 | 4340
8620 | 9310 | A2 | D2 | H11 | H13 | S7 | M2 | M4 | M42 | Other ______

Condition Supplied
Annealed | Normalized | Normalized and Tempered | Solution Annealed
Quenched and Tempered | Other ______

Process Required
Age Harden | Anneal | Carbonitride | Carburize | Harden and Temper
Nitrocarburize | Nitride | Sub-Critical Anneal | Solution and Age Harden
Stress Relieve | Other ______

Compliance Required
Commercial | AMS ______ | Customer Defined / Other ______

Hardness Range Required
______ - ______ RC | RA | RB | RE | R15N | Other ______

Hardness Test Sample Size
Commercial Sampling | Mil-Aero Sampling | 100% | Other ______

Straightness or Flatness Required
______ of an inch per foot | millimeters per meter

Atmosphere Required
Air | Endothermic | Exothermic | Hydrogen | Nitrogen | Vacuum | Other ______

Quenchant Required
Argon | Nitrogen | Oil | Polymer | Salt | Water | Brine | Other ______

Additional Testing Required
Micro-Hardness | Tensile | Charpy | IGO | IGA
Decarburization | Carbon Content | Other ______

Documentation Required
Shipper | Hardness Certification | Process Certification | Charts | Other ______

Other Requirements

Fig. 3. Heat-treating purchase order checklist

raise questions based on his experience and what he sees on the print. Remember, your heat treater has seen and dealt with literally thousands of shapes and sizes, and his opinions are invaluable.

Approvals

Customers often require that their work be sent to heat treaters who have certain approvals in place (Fig. 3). These approvals may include:

ISO9001, AS9100, TS16949, CQI-9

In order to gain these types of approvals, the heat treater must have an effective quality system in place, and it must be verified by an outside service. ISO9001, AS9100, TS16949 quality approvals are usually general in nature (quality management system, management responsibility, product realization, etc.) and are not heat-treating process specific.

Nadcap

This aerospace approval is the highest industry-wide accreditation a heat treater can achieve. Not only does it cover the quality systems above, it also includes audits on specific heat-treating processes. A portion of the weeklong audit also includes the witnessing of actual heat-treat runs. The process-related portion of the audit confirms compliance with aerospace and aerospace-prime specifications. PRI is the accrediting body for this program. More information on Nadcap can be found at www.pri-network.org.

Prime Approval

Even if a heat treater is ISO9001 and AS9100 approved as well as Nadcap accredited, they still may not be able to process work for certain aerospace primes. These include Boeing, Pratt & Whitney, Airbus, etc. These companies also require that your heat treater pass their site audits. If the parts you are sending out are related to the companies above, they can only be processed at heat-treating facilities approved by the aerospace prime.

Testing Requirements

Your purchase order or request for quotation should clearly state your testing requirements. If you are asking the heat treater to process the order to a certain AMS specification, these requirements may already be defined. Otherwise, you need to communicate what your requirements are (or what exceptions you will allow). A few examples of typical testing requirements include surface hardness, microhardness, tensile strength, surface carbon content, IGO/IGA and microstructure. Be aware that test coupons may be required to facilitate these types of tests. Also, keep in mind the consequences of the tests. For example, if an order is to be 100% Rockwell hardness tested, all of your parts are going to be returned with hardness dents or indentations on them.

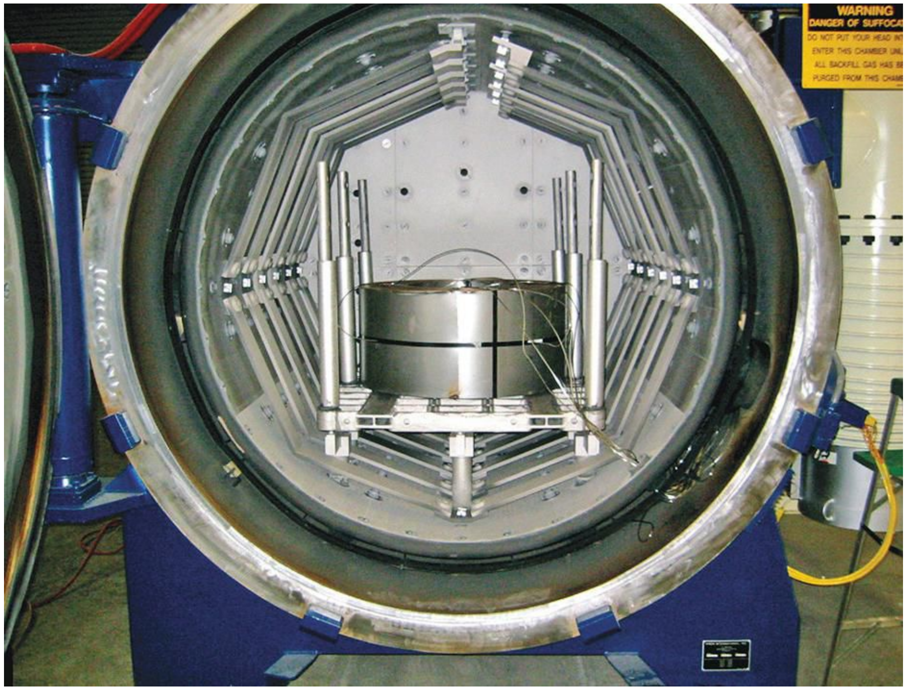


Fig. 4. Process control of steel coils in a vacuum furnace using thermocouples inserted into the load in order to monitor actual part temperature

Dimensional Requirements

To some degree, all materials will change size and shape during heat treatment. You need to plan your manufacturing process to accommodate these changes. Stating "Keep Flat" or "Keep Straight" on your purchase order is not realistic. However, there are materials and processes that can be chosen to minimize these changes. Putting a flatness or straightness callout on your purchase order is good practice, but only if it is realistic and achievable. Involving your heat treater early in the project can help in this regard. The heat treater will be able to assist in specifying the most suitable material and process sequence that will get results that meet your expectations.

Cosmetic Requirements

If your parts require special handling, please inform your heat treater of that fact. Keep in mind that individual handling and racking of parts can add significant cost. In most cases, heat treaters will handle your parts with care. However, if you require that a certain surface remain free of hardness indentations, for example, please note that instruction on your purchase order. Other cosmetic requirements may include glass beading after heat treatment, vacuum processing to keep parts clean and bright, and keeping sharp corners of parts free from nicks and dings.

Documentation Requirements

There are several levels of documentation that can be supplied by the heat treater.

- Shipping ticket only
- Product certifications (hardness, microstructure, mechanical properties, etc.)
- Process certifications
- Furnace chart-recorder data

The shipping ticket is signed by the customer and used as the proof of delivery document. Product certifications typically show the number of parts tested and their range of values. It should also state the specification the testing complied with. The process certification usually shows the process that was run (soak times, temperature setpoints, quenchants used, etc.). It will also state what specifications the process was in compliance with. For commercial work, only the first two are usually supplied.

For aerospace work, the first three are typically supplied. The furnace chart-recorder data is usually kept on file by the heat treater for a predetermined number of years. It can be made available to the customer on request. If the heat treater is running a proprietary process, however, he may be unwilling to hand this intellectual property over to the customer.

Summary

In conclusion, a true partnership needs to exist between the customer and heat treater in order to optimize the performance of the end-product. Heat treaters have their customer's best interest at heart, but they are not mind readers and have limitations that are best overcome by mutual information flow and good planning combined with knowledge and experience.

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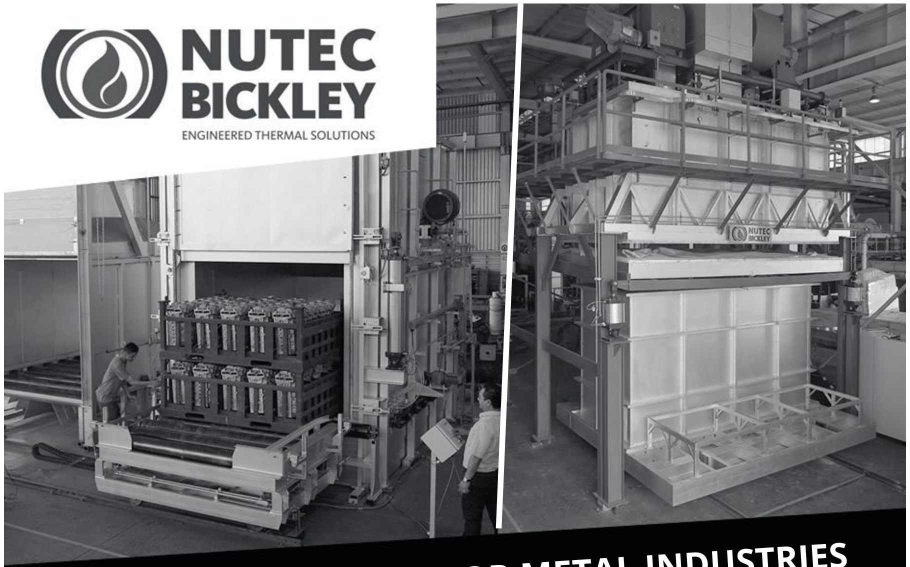

Saving Customers Time, Money and Energy

Heat treatment is an energy user and all too often an energy abuser. Given the significant amounts of energy needed to run a typical process, this translates into a huge cost to the heat treater. In addition, material choices and specification requirements often dictate cycle temperatures and times, while environmental compliance further adds to the overall cost. How can we, and how should we, go about addressing these issues? Let's learn more.

Energy[1,3,4]

The first step in any energy-management program is to understand where and how much energy is being consumed or wasted. Energy monitoring via gas totalizers or electrical power meters (including peak-demand monitoring) is a good way to start. Thermal imaging (Fig. 1) is another tool to help focus maintenance activities and identify areas for improvement.

A complete energy audit normally consists of five basic parts:[1]

- A description of the facility and all of the equipment involved
- Mapping of the current energy consumption in the plant

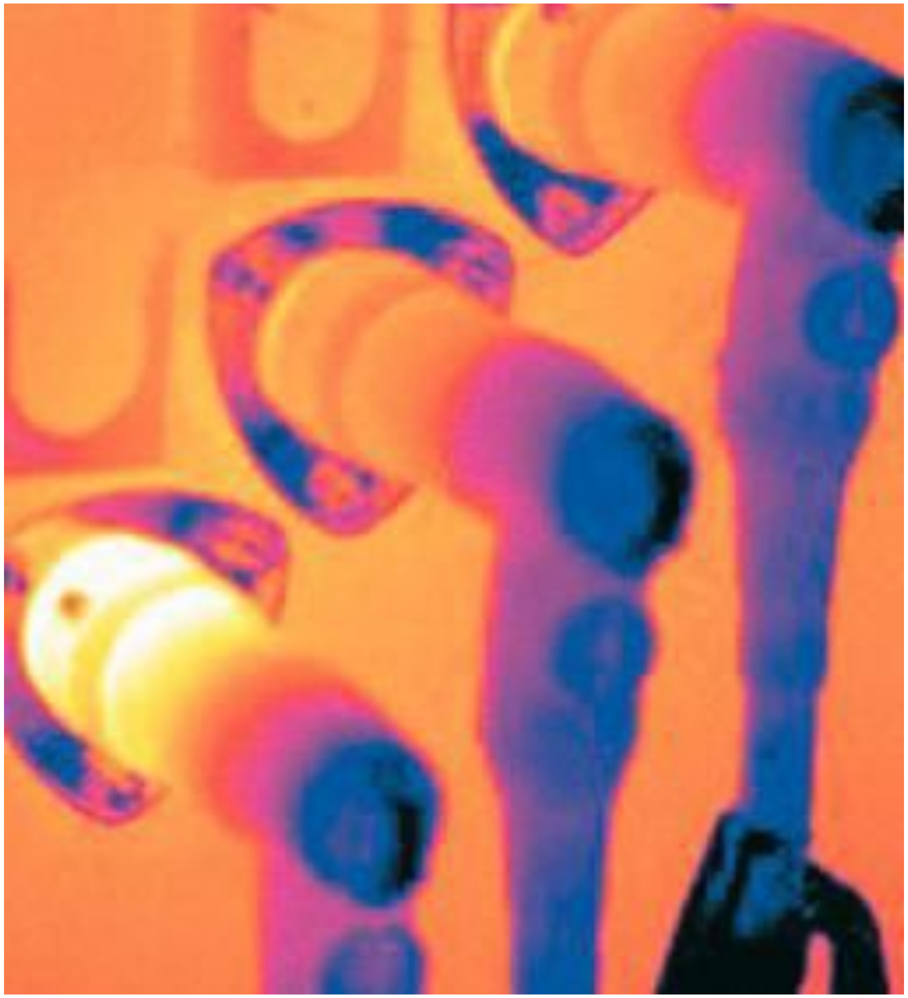

Fig. 1. Thermal image showing areas of heat loss around silicon-carbide heating elements in a mesh-belt conveyor furnace *(courtesy of Pregl Services)*

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- The short-term energy consumption in the plant
- The long-term energy consumption in the plant
- A search for energy-efficiency improvement possibilities

By way of example,[1] an energy audit was conducted at a large commercial heat treater in Europe with the following major findings:

- The plant consumed 9,590 MWh/year of electricity and 120 MWh/year of peak demand costs at the start of the audit.
- 68% of the energy was consumed by primary heat-treatment processes and equipment with the other 32% consumed in ancillary processes.
- The largest portion of the energy consumption was the main 14 integral quench-style heat-treatment furnaces. The ancillary equipment consisted of over 30 units (preheating and tempering furnaces, parts washers).
- The electricity consumption could be decreased by 7.85% (753 MWh/year) by energy conservation (housekeeping and recipe optimization) measures.
- The electricity consumption could be further decreased by 4.36% (418 MWh/year) and the peak demand eliminated by energy monitoring and energy-saving investment measures.
- The combination of the proposed energy housekeeping measures and energy-saving investment measures reduced the energy cost with savings of approximately $135,000 per year. It also resulted in a reduction of the carbon-dioxide emissions by approximately 740 tons per year.
- If all proposed measures were implemented, the total investment cost would have a payback of three years and a net present value of almost $2.1 million over 10 years using current energy prices.

Environmental

Environmental compliance is a complex subject. I've often seen heat treaters shaking their heads in disbelief as to why they must both pay for water and pay a sewer tax to dispose of it having done nothing more than heat it a few degrees. These types of issues have led to a philosophical debate within the heat-treatment industry as to what constitutes good environmental policy. Stark contrasts can be seen in different countries throughout the world.

In the U.S., for example, environmental policy is viewed as obeying the rules and regulations mandated by federal, state and local governmental agencies. By contrast, many corporations strive to be "good neighbors," often imposing more stringent environmental policies than those mandated by law.

In Europe, there is a strong cultural emphasis placed on environmental issues, which are at the forefront when considering equipment purchases and the way in which heat-treat plants are managed. Emissions are not only carefully monitored, but companies often strive to outdo one another so as to be rewarded rather than penalized (e.g., the carbon tax) for their environmental choices.

In Asia, as societies continue to evolve, environmental policies will follow suit. For example, one heat treater in Japan has coy fish swimming in his cooling-water system, and each employee is responsible for keeping his fish alive and healthy! By contrast, the smog that surrounds cities in China is a clear indication that we still have more work to do.

Process Optimization

Traditionally, when a load is heat treated in a furnace, the only measurements available to track the cycle progress are furnace control thermocouples or, in some instances, part workload thermocouples. The goal is to heat the entire load to a specific temperature and soak at that temperature for a given period of time.

Ideally, the user would like to know the temperature at the center of the load and the moment that all of the piece parts reach that temperature. If known, cycle times can be optimized. Most heat-treat recipes soak the load longer than necessary to ensure meeting metallurgical and mechanical properties. This adds cost and reduces throughput.

There are currently three principal methods for ensuring a stable and uniform load temperature.

- Manual intervention – visual inspection of the load during processing. This can be done by observing the load through a peep sight or, in some rare instances, by opening the furnace door(s). This method is dependent on the skill of

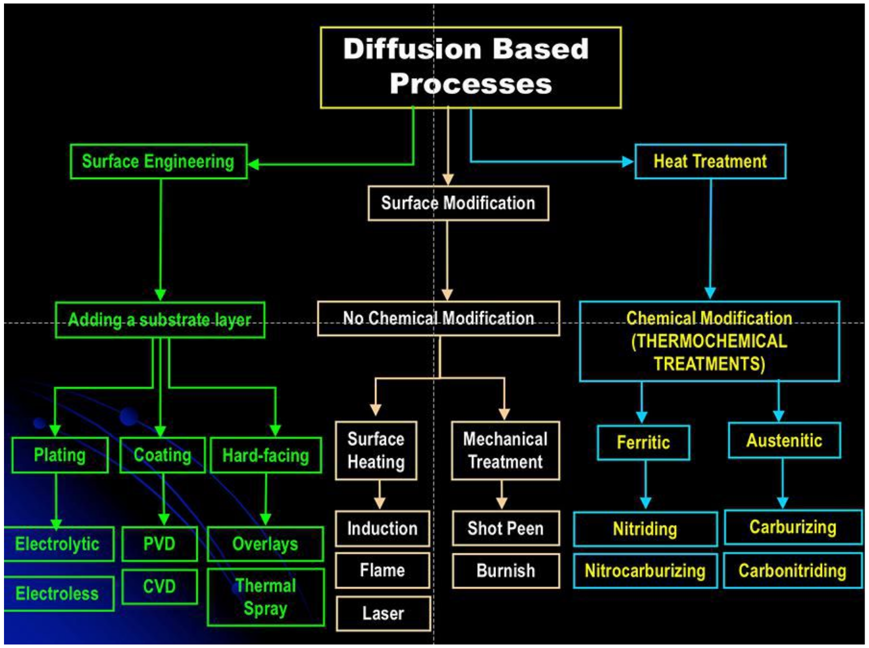

Fig. 2. Categorization of diffusion-related processes

the operator and can disrupt furnace operation.

- Standard automatic control – where the heating process parameters are calculated and the soak time is over-specified to ensure uniformity.
- Process supervision to monitor energy consumption as well as all of the usual parameters, ensuring that the required heating practice is achieved without wasted time and energy.

Companies are creating modeling algorithms and so-called visual supervisors[2] that calculate furnace parameters, allowing for improved temperature uniformity. This in turn allows the cycle time (regardless of the load) to be optimized. This steady-state method results in reduced cycle times and energy costs.

Finally, optimization of all diffusion-based processes (Fig. 2) can be achieved by increasing process temperature as a means of reducing cycle time. In this case, cost reduction can be achieved by such methods as:

- Modifying material chemistries
- Improving process control to minimize case-depth variation
- Controlling distortion so as to avoid post-heat-treatment machining operations
- Choosing more-efficient diffusion processes for the application

Cost[1]

Cost containment is a huge issue for the heat treater. The net present value is the difference between the investment and the present value of future cost savings due to the investment. The present value of future cost savings is calculated with an interest rate, usually the cost of capital. The net present value is calculated according to:

$$(1) \qquad NPV = \sum_{i=1}^{i=t} \frac{a_i}{(1+r)^i} - I$$

where NPV is the net present value, t is the time frame for the calculation, a_i is the annual cost saving due to the investment, r is the cost of capital and I is the total investment cost.

This method considers the time value of money and accounts for all cash flows during the time frame for the calculation. It can be used to evaluate an investment and to compare the profitability between investments. A good investment will have a high net present value.

Another way to compare investments is to calculate the net present value ratio, which is the net present value divided by the initial investment according to:

$$(2) \qquad NPVR = NPV / I$$

where NPRV is the net-present-value ratio, NPV is the net present value and I is the total investment cost. A higher NPRV indicates a more profitable investment.

Summary

In order for heat treating to be the most cost-effective solution to manufacturing, we must continue to evolve in the areas of technical innovation; improved up-time productivity; and reduction of energy, environmental and process costs.

Simply reducing cost by heretofore traditional methods (e.g., labor reduction, quality relaxation, deferred reinvestment or delayed introduction of new technology) will no longer keep the industry competitive. Applying conservation methods and negotiating more-favorable energy contracts are a good start.

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What Happens to Steel During Heat Treatment? Part One: Phase Transformations

If we were traveling from one city to another, we would need to input our destination into a GPS locating device, use an Internet website such as MapQuest® to plot our route or simply pull out a map to help us find our way. The Iron-Carbon, or perhaps more accurately the Iron-Iron Carbide phase diagram (Fig. 1), is nothing more than a roadmap for heat treaters. A phase diagram simply helps us predict what will happen to the internal structure of the steel. Let's learn more.

Heat treating of steel involves phase transformations. These can be diffusion-dependent transformations such as the formation of pearlite and diffusionless transformations such as when martensite forms. Let's begin by reviewing the phases identified in Figure 1 – namely ferrite, austenite, cementite and pearlite.

At room temperature, the most stable form of iron is ferrite, also known as α-iron ("alpha" iron), which has a body-centered cubic (BCC) crystal structure. Ferrite is a fairly soft material that can dissolve only a very small amount of carbon – no more than 0.021% at 1670°F (910°C) and only 0.008% at room temperature. Ferrite is the phase that exists below the upper critical temperature of a steel with less than 0.80% carbon.

The formation of austenite or γ-iron ("gamma" iron) begins when steel is heated above its lower critical temperature, the Ac_1 (A_1) line shown on the diagram. The structure is fully austenitic above the Ac_3 (A_3) or Ac_{cm} line. Austenite has a face-centered cubic (FCC) crystal structure and can contain up to 2.03% carbon at 2110°F (1154°C) or approximately 100 times greater than the maximum limit for ferrite. Carbon strengthens steel and gives it the ability to be hardened by heat treatment.

As carbon-rich austenite cools, the mixture attempts to revert back to the ferrite phase, resulting in some areas having an excess of carbon. One way for carbon to leave austenite is for a phase called cementite to precipitate out – leaving behind iron that is pure enough to take the form of ferrite – creating a cementite-ferrite mixture (Fig. 2). Cementite, or iron carbide, is a hard, brittle intermetallic compound consisting of iron and carbon at 6.7% (at this percentage, every fourth atom is a carbon atom). Its chemical symbol is Fe_3C. Carbon in the form of graphite is the stable phase – the phase having minimum free energy. Thus cementite is metastable – it simply forms before graphite can develop and will

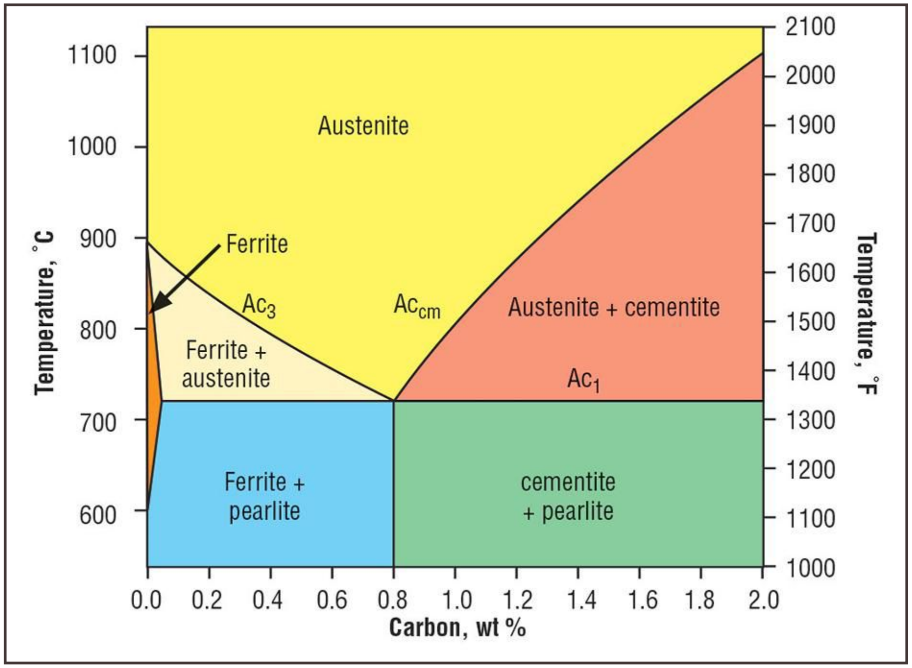

Fig. 1[1] Simplified Iron-Iron Carbide phase diagram

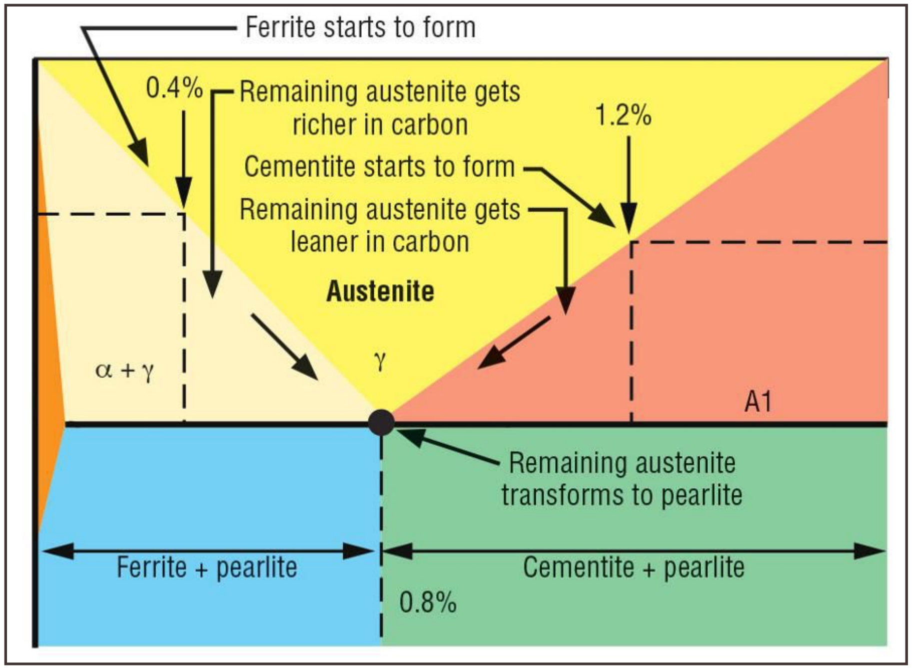

Fig. 2[2] Section or the Iron-Iron Carbide Diagram Showing the Cooling Transformation of Two Different Carbon Steels from Austenite to Mixtures of Ferrite and Cementite."

decompose into graphite given the right conditions.

The formation of pearlite involves the mixture of two phases, iron and iron carbide, and is a growth-dominated process requiring a combination of both high temperatures and long times. Pearlite is not a true phase. The iron takes the crystalline form of ferrite while the iron carbide takes the crystalline form of cementite, and the resulting structure consists of alternating lamellar bands, or plates. The distance between these plates as well as their thickness is dependent on the cooling rate of the material. Fast cooling creates thin plates that are close together and slow cooling creates a much coarser structure possessing less toughness. A fully pearlitic structure occurs at approximately 0.8% carbon, the eutectoid point on the phase diagram.

Under certain cooling conditions, another phase known as bainite can form. The formation

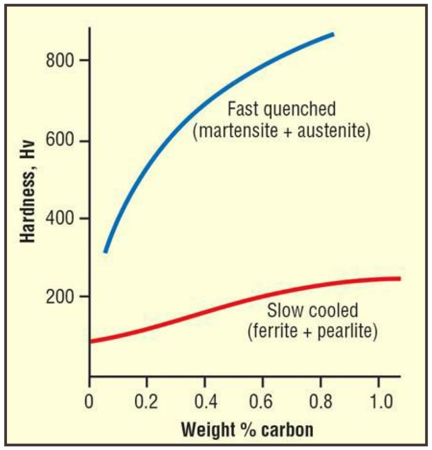

Fig. 3[3] Hardness variation by phase present

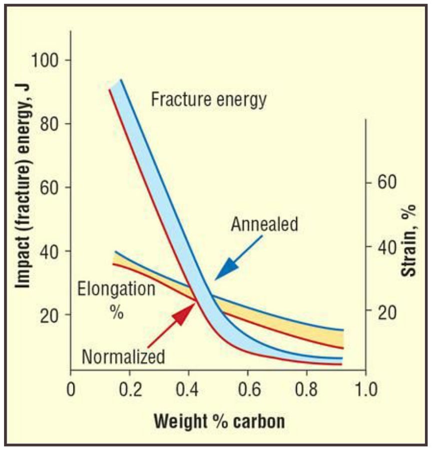

Fig. 4[3] Impact property variation by phase change

of bainite is dominated by the nucleation process, resulting in a fine dispersion of ferrite and cementite, and is typified by intermediate hardness and good toughness. Bainite forms as needles or plates depending on the temperature of the transformation. Pearlite and bainite transformations compete with one another, and once a portion of the steel microstructure has transformed to one or the other, it is not possible to transform between them without reheating to form austenite. Bainite will normally not form on continuous cooling to room temperature because all of the austenite will have transformed to pearlite by the time the bainite transformation is ready to begin. For this reason it is not shown on the phase diagram. Fast cooling followed by interrupted quenching and an isothermal hold above the martensite start (M_s) temperature is required to form bainite.

Martensite is usually considered to be a grain structure, not a phase. For this reason, martensite is not shown on the phase diagram of the iron-carbon system. Martensite is characterized by an angular needle-like or plate-like structure. Alloys with less than 0.6% carbon form lath martensite. Alloys with more than 1.0% carbon form plate martensite, and alloys between 0.6 to 1.0% carbon form mixtures of the two in varying degrees.

The hardness of martensite is dependent on its carbon content. You might be surprised to know that pure martensite is soft and ductile. As a result of heating and rapid cooling, however, we form martensite with interstitially dissolved carbon in a supersaturated state, resulting in very high hardness – about four to five times the strength of ferrite. A minimum of 0.4% carbon is needed to form martensite. When austenite is quenched, the carbon is “frozen” in place as the crystal structure changes. Since the carbon atoms are too large to fit in the interstitial vacancies, a distortion of the crystal structure occurs, creating a body-centered tetragonal (BCT) crystal structure.

The formation of martensite involves nucleation and growth, but the mechanism is not controlled by diffusion. Rather, it is primarily a shear process similar to twinning. Martensite formation also produces some dilatation. While the nucleation of martensite is thermally activated, the growth process is not – the glide of transformation dislocations shears the material, forming thin plates. These plates grow along specific crystallographic directions until either they encounter an austenite grain boundary, another martensite plate or until enough martensite has formed causing the growth reaction to cease. Because martensite plates form at approximately the speed of sound – much too fast for carbon to diffuse out of the austenite – martensite and austenite have identical chemical compositions. The martensite phase is metastable and wants to decompose into ferrite and cementite on heating.

By raising the temperature to a few hundred degrees, martensite will decompose into bainite. The decomposition of martensite starts by forming bainite at the boundaries of the martensite needles. If the temperature is high enough, this bainite can coarsen into lower (fine) pearlite and eventually upper (coarse) pearlite.

Mechanical properties are affected by which phase(s) are present in the microstructure. Hardness (Fig. 3) is the measurement with which heat treaters most often relate. However, one must be careful. For example, increasing cementite concentration will lead to an increase in hardness but with a resultant loss of ductility.

Elongation is a measure of ductility (Fig. 4). We see that the ductile behavior decreases in a rather linear fashion with increasing carbon content. Once the matrix contains only a small percentage of ferrite, the ductility approaches zero. Impact energy is a fairly direct measure of brittleness. Low energy means the material is brittle and will fracture easily. We see impact values decrease steeply and then taper off around 0.5% carbon. For hypereutectoid steels (those having greater than 0.80% carbon), the fracture energy is relatively constant and quite low. Essentially, the fracture toughness for these steels is determined by the fracture properties of cementite.

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What Happens to Steel During Heat Treatment? Part Two: Cooling Transformations

Once we've heated a piece of steel to elevated temperature, it must be cooled in order to complete its transformation into a useful engineering material. Understanding cooling transformations is another important responsibility of the heat treater. Let's learn more.

Types of Cooling Transformation Diagrams

Transformation diagrams are simply another form of roadmap to allow us to predict a steel's response to heat treating. There are two main types of cooling transformation diagrams, and it is important that we understand what each is and how we can use them.

Time-Temperature Transformation (TTT) Diagrams

Also known as isothermal transformation (IT) diagrams, TTT diagrams measure the rate of transformation at a constant (isothermal) temperature (Fig. 1). In other words, once a part is austenitized, it is rapidly cooled to a lower temperature and held at that temperature while the rate of transformation is measured. The different types of microstructures produced (ferrite, pearlite, bainite, martensite) are then indicated on the diagram together with the holding times required for each transformation to begin and end.

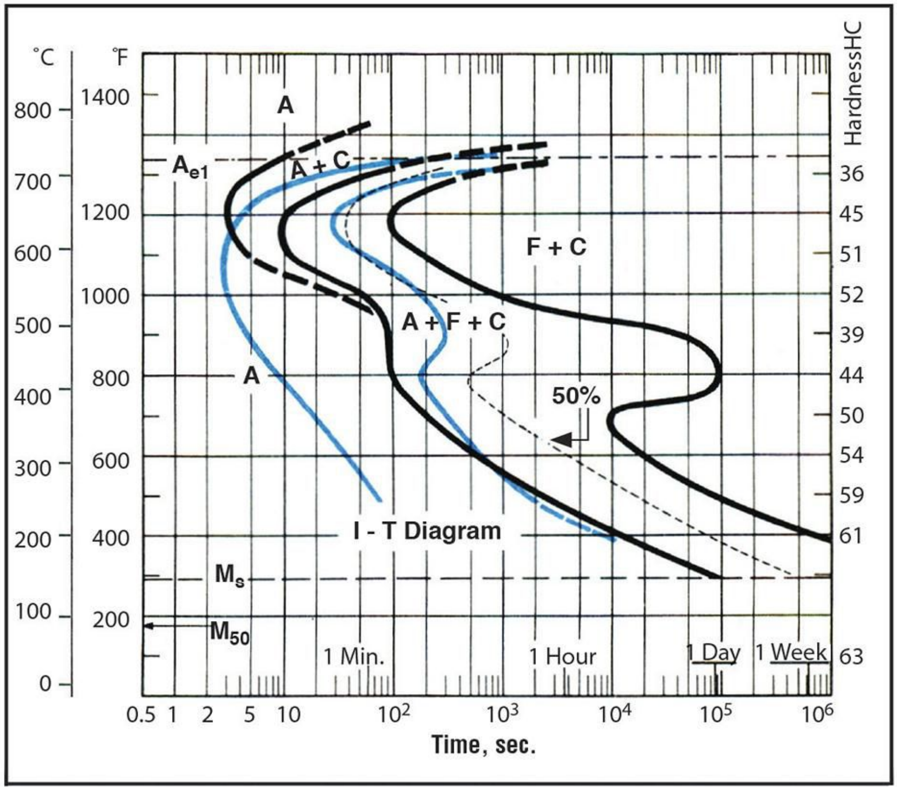

Fig. 1[1] TTT Diagram (52100 Steel)

Continuous Cooling Transformation (CCT) Diagrams

Also known as cooling transformation (CT) diagrams, CCT diagrams measure the degree of transformation as a function of time for a constantly changing (decreasing) temperature (Fig. 2). In other words, a sample is austenitized and then cooled at a predetermined rate, and the degree of transformation is measured using such techniques as dilatometry, magnetic permeability or other physical methods.

A Closer Look at TTT Diagrams

TTT diagrams are useful in planning heat treatments and in determining the critical cooling rate on quenching, which is the cooling rate at which one just avoids the nose of the TTT curve. If the austenite-to-martensite transformation is incomplete, retained austenite usually transforms during tempering into the transformation product indicated on the TTT diagram.

In general, TTT diagrams allow us to gain limited information on the influence of alloying elements on transformations during continuous cooling by comparing the temperatures at which the transformation products occur.

A Closer Look at CCT Diagrams

CCT diagrams provide a useful tool for predicting the microstructure achieved during a typical quench after heat treating. We can measure (or calculate) the rates of cooling at any point within a steel part (surface, core, mid-radius).

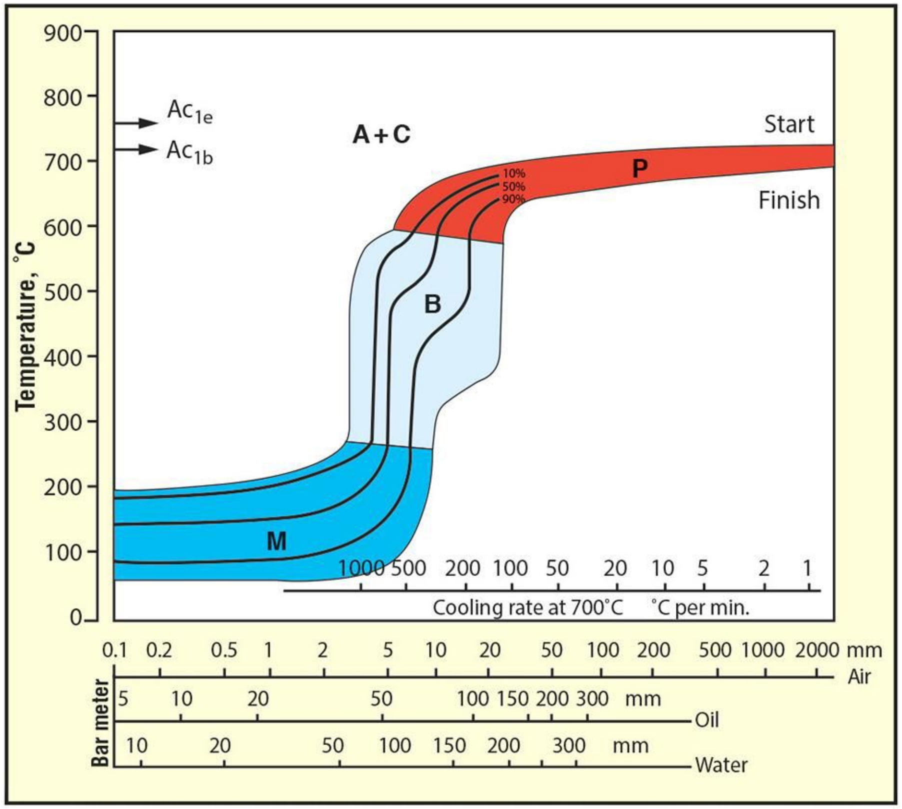

Fig. 2[2] CCT Diagram (52100 Steel)

CCT diagrams have been developed for many steel compositions by using various experimental methods. One such method is the use of Jominy bars with thermocouples attached along the length of the bar.

Microstructures are correlated with the cooling rates calculated from the thermocouple readings. A CCT diagram that indicates the transformation products obtained at various cooling rates is then developed from the Jominy information. Thus the hardenability of steel allows us to develop an engineering approach to understanding the effects of quenching in various cooling media (see "Jominy Testing, The Practical Side," - October 2001).

CCT diagrams show the approximate proportions of the major phases and the hardness of the microstructures obtained. The effect of tempering on hardness levels is often shown as well. The hardenability effect of the steel can be seen directly from the diagram – low-hardenability steels show early transformation, mainly from the upper left-hand side of the diagram (to ferrite and pearlite or bainite). By contrast, high-hardenability steels exhibit curves in the lower right-hand side of the diagram with austenite changing predominately to martensite over a wide range of part thicknesses and quenching rates.

A Comparison of TTT and CCT Diagrams

Despite the general similarity in shape between CCT and TTT diagrams for identical steels, the data is presented differently. On CCT diagrams the products of transformation (martensite-bainite-pearlite) are indicated along the bottom of the diagram, whereas they are shown on the right side of the TTT diagram. Phase changes are recorded within the starting and finishing boundaries on CCT diagrams, whereas on TTT diagrams these regions indicate the transformation phase themselves.

Although similar in shape to TTT diagrams, the nose of the CCT diagram is shifted down to the right, indicating that more time is available for martensite transformation than is shown on the corresponding TTT diagram. TTT diagrams actually err by indicating a faster cooling rate than necessary to form 100% martensite on quenching. This error is usually on the conservative side since the goal of most heat-treatment operations is to produce 100% martensite.

Appropriate Cautions

CCT diagrams mainly refer only to the center of a bar, but the microstructures at other positions can be inferred. For example, the microstructure produced on cooling at some mid-radius position in a larger diameter often corresponds to that produced at the center of a bar of smaller diameter – a so-called equivalent diameter – with similar microstructures being produced by similar cooling rates.

CCT diagrams usually refer to the average chemical composition. Variations in composition can lead to considerable differences in microstructure and properties. There are also critical thickness ranges where slightly slower or faster cooling rates produce significant changes

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in the predominant microstructure indicated. Changes in carbon and manganese content can have pronounced effects on these thickness ranges. A major difficulty in constructing CCT diagrams is the interpretation of transformation behavior. Martensite and bainite are affected by compositional changes in the parent austenite that may have resulted from any prior ferrite formation or carbide precipitation at higher temperatures.

Prior heat treatment can affect grain size and hence modify the subsequent transformations on cooling. The austenitizing temperature may affect the austenite composition of steel that contains strong carbide-forming elements. Consequently, undissolved carbides may be present. These considerations should be taken into account when using or adapting CCT diagrams. Heating by applied energy (induction, flame, laser) with rapid heating and short thermal cycle times has a drastic effect on the condition of the austenite and as a result the accuracy of the CCT diagrams. Welding is another process that is very difficult to predict using these types of diagrams.

Another factor is quench severity and the degree of agitation, the effects of which can only be determined experimentally. Air cooling is normally the main criterion for developing these diagrams. Water – not brine quenching – is represented as a standard medium-to-fast quenching medium.

Summing Up

Few heat-treatment processes involve isothermal transformation, and most microstructures are produced as a result of continuous cooling operations. If the rate of cooling is slow, the microstructure corresponds more closely to that indicated on the upper portion of a TTT diagram. Faster cooling rates, however, deviate considerably during the transformation process.

While the heat treater should be aware of both types of transformation diagrams, the use of CCT diagrams is often more directly applicable to microstructures that are produced at the center of a heat treated part under real-world conditions. For this reason they are a valuable tool.

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Boron in Steel

Boron addition to steel is a complex subject, and it does not always make our lives as heat treaters easier. Let's learn more.

The Boron Hardenability Effect

An outstanding feature of boron steels is the improvement in hardenability produced by the addition of even a minute quantity of boron. It is generally accepted that a hardenability peak is reached when the quantity of boron is between 3 and 15 ppm[1]. If an excessive amount of boron (>30 ppm) is present, the boron constituents become segregated in the austenite grain boundaries, which not only lowers hardenability, but also may decrease toughness, cause embrittlement and produce hot shortness. The affect of boron on hardenability also depends on the amount of carbon in the steel. The effect of boron increases in inverse proportion to the percentage of carbon present.

Boron must be in its atomic state to improve hardenability, which means that care must be taken during steel production for the boron to be effective. Boron may also become ineffective if its state is changed by incorrect heat treatment. For example, high austenitizing temperatures must be avoided as well as temperature ranges where certain boron precipitates occur.

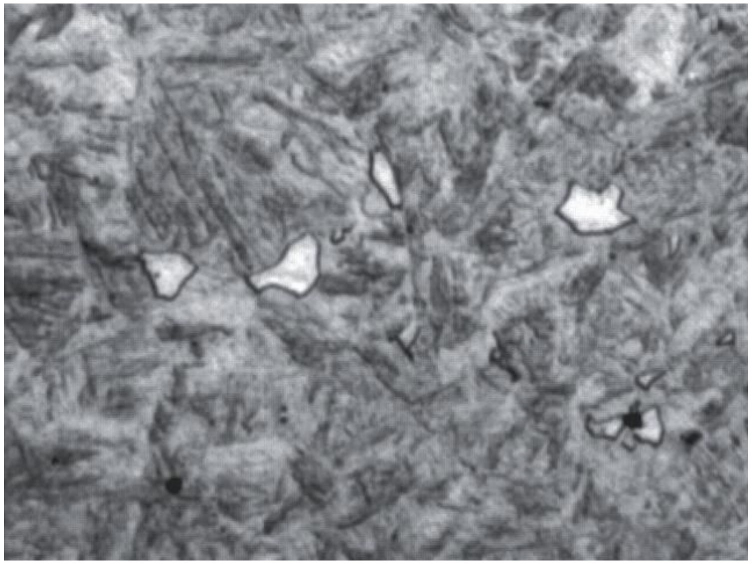

Fig. 1. Core ferrite (500X)

Hardenability is highly dependent on the behavior of oxygen, carbon and nitrogen present in the steel. Boron reacts with oxygen to form boron oxide (B_2O_3); with carbon to form iron borocementite ($Fe_3(CB)$) and iron borocarbide ($Fe_{23}(CB)_6$); and with nitrogen to form boron nitride (BN). Loss of boron by oxygen is prevented by making the boron addition to

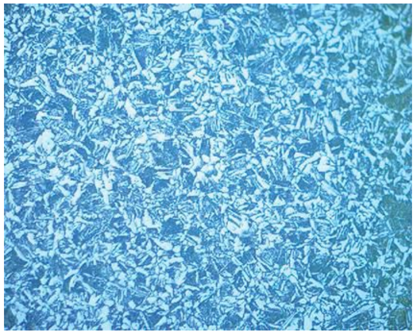

Fig. 2. Core microstructure (500X)

Fig. 3. Core microstructure (500X)

silicon-aluminum killed steels and by using good ladle and mold practices. Strong nitride formers (titanium, aluminum, zirconium) protect the boron from reaction with nitrogen. For example, if nitrogen is fixed by using titanium, satisfactory hardenability is obtained in the temperature range up to 1830°F (1000°C) provided that the steel contains about 5-20 ppm of boron.

The hardenability of boron steel is also closely related to austenitizing conditions and is generally said to decrease by heating above 1830°F (1000°C). Boron steel must also be tempered at a lower temperature than other alloy element steels of the same hardenability.

Case History 1 – Commercial Heat Treater

Problem description: A commercial heat treater reported low core hardness and localized core ferrite (Fig. 1) after routine inspection of sample lots of steel parts carburized in separate loads. Inspection of subsequent loads using the same cycle failed to show a recurrence of the ferrite condition.

Background: Parts were carburized in a batch integral quench furnace – in one case at a temperature of 1675°F (915°C) and in the other at 1700°F (925°C). Either temperature should have been sufficient to completely dissolve ferrite into austenite (per the iron-carbon phase diagram).

Attempted solutions: The first step was to send parts out for chemical analysis and to compare the chemistry to customer specification and to the material certification. The material chemistry was found to be within specification.

Next, the furnace temperature and temperature uniformity were checked and found to be within acceptable limits. No unusual variation in case depth was found, which would be indicative of austenitizing temperature variation in the carburizing chamber. Additional steps involved increasing load sample size to nine pieces/load and complete microstructural checks. Loading patterns and load weight were monitored.

As an added precaution, carburizing temperature was increased to 1725°F (940°C), necessitating additional checks to look for dimensional changes associated with this higher temperature. Careful monitoring of case depth was also needed because quite often increasing diffusion temperature can add variation in shallow case depths.

Samples containing core ferrite were reheated in a vacuum furnace at temperatures of 1675°F (915°C) and 1800°F (980°C). Core ferrite did not dissolve completely at 1675°F (915°C), but austenitizing at 1800°F (980°C) finally put the ferrite back into solution.

Case History 2 – Captive Heat Treater

Problem description: Ferrite was observed in the core microstructure of 15B21 parts along with variations in core hardness from 93 HRB to 40 HRC.

Background: Parts were initially carburized at 1725°F (940°C) and oil quenched from 1550°F (840°C). A surface hardness of 58–61 HRC was obtained with an effective case depth (50 HRC) of approximately 0.055 inch (1.40 mm). Microstructural examination of parts at 93 HRB (Fig. 2) revealed the presence of ferrite in the core.

Attempted solutions: The first step was to send parts out for chemical analysis (Table 1) and to compare the chemistry to customer specification and to the material certification. The material chemistry was found to be within specification.

The next step was to run parts in different furnaces. A total of seven batch integral-quench and pusher furnaces were used in an attempt to rule out such variables as temperature, temperature uniformity, carburizing uniformity, furnace atmosphere, atmosphere control, quenchant and quench severity. All parts run produced similar results.

Finally, material from a different steel lot was processed to the same set of heat-treatment parameters. The result was consistent core hardness in the range of 34 HRC (Fig. 3).

Table 1 Chemistry Comparison

Element	Specification	Part 1	Part 2	Ladle Analysis
Carbon	0.19 – 0.23	0.17	0.19	0.20
Manganese	0.8 – 1.10	0.90	0.94	0.91
Phosphorous	0.040 max	0.021	0.022	0.009
Sulfur	0.050 max	0.012	0.014	0.015
Silicon	0.15 – 0.35	0.22	0.24	0.24
Nickel	-	0.10	0.13	0.10
Chromium	-	0.16	0.19	0.16
Molybdenum	-	0.016	0.024	0.02
Copper	-	0.21	0.17	0.28
Aluminum	-	< 0.01	< 0.01	0.0019
Boron	0.0005 – 0.003	0.0016	0.0023	0.0017
Titanium	-	0.023	0.043	0.035
Niobium	-	< 0.01	< 0.01	-
Vanadium	-	< 0.01	< 0.01	-
Lead	-	< 0.01	< 0.01	-

Note: Nitrogen content was not specified.

Lessons Learned

The heat treater can control many of his process and equipment variables. He/she can design robust boost/diffuse carburizing cycles, minimize retained austenite and intergranular oxidation as well as control carbide distribution. The heat treater can also ensure that the equipment is operating properly, is well maintained and has good temperature uniformity. He/she can load parts in a manner as to maximize heat transfer and aid uniformity of quenching. Sometimes all of this is not enough, however, since the heat treater has limited control over material properties (e.g. alloy segregation, as-received grain size, steel cleanliness).

Both the commercial and captive heat treater in these case studies spent considerable resources and countless hours in an attempt to rule out a process or equipment problem and to find the root cause of the phenomenon. While most heat-treating problems are not material issues, these case studies clearly pointed to the material as the root cause of the problem. Despite the fact that the Jominy readings were within an acceptable range for the steels in question, parts would not harden.

As it turns out, the titanium to nitrogen (Ti:N) ratio in the steel heat is a critical factor. When specifying boron steels, many companies believe a 4:1 ratio (or greater) is needed to avoid the problem of "ineffective" boron. In many cases the effective boron content is not the total boron content.

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Steel Cleanliness: Inclusions in Steel

Steel cleanliness has become a topic of great interest to the heat treater given the reality that steel is a global-sourced commodity. Often, the type and distribution of inclusions have a significant influence on mechanical properties. It's time to review this subject so that we can continue to better understand the steel we process. Let's learn more.

The composition, size, number and distribution of inclusions determine the cleanliness of steel. Very large nonmetallic inclusions of any sort are undesirable, while very fine dispersions can be either helpful or harmful. Larger inclusions are responsible, to a greater or lesser extent, for directional properties, notch sensitivity, creep and reportedly poor fatigue properties.

There are a number of rating systems and methods to determine inclusion count, and these are commonly presented in terms of severity (Table 1). Common test methods include ASTM E 45 (Standard Test Method for Determining the Inclusion Content of Steel) and ASTM E 2142 (Standard Test Methods for Rating and Classifying Inclusions in Steel Using the Scanning Electron Microscope).

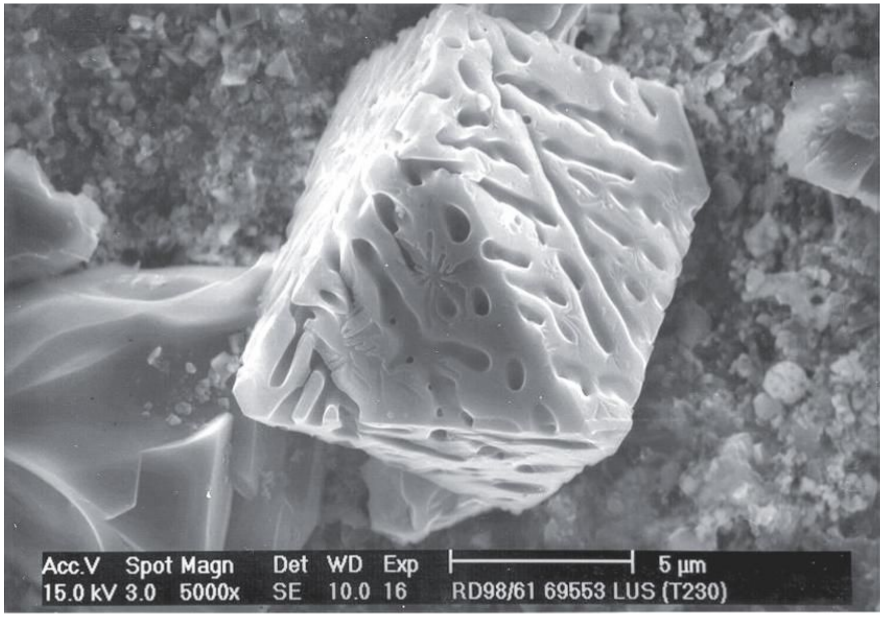

Fig. 1. Typical nonmetallic inclusion

Where Do Inclusions Come From?

The steelmaking process (tap, ladle treatment and continuous casting) is the source for most nonmetallic inclusions (Fig. 1), although indigenous (natural) sources, alloy additions and exogenous (foreign) sources such as refractory or slag also contribute. Most are formed because admixture dissolubility decreases during cooling and consolidation.

Table 1. Minimum values for inclusion severity rating levels for measurements in micrometers

Severity	A	B	C	D
0.5	37.0	17.2	17.8	1
1.0	127.0	76.8	75.6	2
1.5	261.0	184.2	176.0	4
2.0	436.1	342.7	320.5	9
2.5	649.0	554.7	510.3	16
3.0	898.0	822.2	746.1	25
3.5	1181.0	1147.0	1029.0	36
4.0	1498.0	1530.0	1359.0	49
4.5	1898.0	1973.0	1737.0	81
5.0	2230.0	2476.0	2163.0	100

Notes: Test Method E 45 Rating Limits (μm at 1× or count)
For expression in other units, see Test Methods E 45, Table 2

How are Inclusions Classified?

Nonmetallic inclusions are typically divided into the following groups: oxides (Fig. 2), sulfides, silicates, nitrides and phosphides. Inclusions bonded with oxygen are further divided into free oxides, spinels (compound oxides in bi- and tri- configurations) and silicates (pure or admixed).

Types of Inclusions

Inclusions fall into the following general categories:

- $CaO\text{-}SiO_2\text{-}Al_2O_3$
- $Fe_xMn_{1-x}O\text{-}SiO_2\text{-}Al_2O_3$
- $Fe_xMn_{1-x}O\text{-}SiO_2\text{-}CryAl_{2-y}O_3$
- Fe_xO_y (oxides)
- Fe_xO_y (oxides) + transition metals (lanthanides, Ti, Zr, Hf, V, Nb, Ta)
- $MgO\text{-}SiO_2\text{-}Al_2O_3$
- $MnO\text{-}SiO_2\text{-}Al_2O_3$
- MnS (sulfides)
- Se + Te

To facilitate identification, inclusions can be grouped based on three predominate optical characteristics: color, reflectivity and reaction to polarized light (Table 2). Some inclusions fall into multiple categories.

Here's a brief overview of the more common types of inclusions:

1. Alumina (Al_2O_3) is found in steel in which aluminum has been added typically to control grain size or as a deoxidant. It can also be carried into steels from refractories in which case the inclusions tend to be large and isolated. Optically, they appear

as stringers, often with "comet tails" due to polishing.

2. Aluminum sulfide (Al_2S_3) is found in steels deoxidized with excess amounts of aluminum. These inclusions are less soluble in steel than manganese sulfide and separate out earlier, forming large particles.
3. Chromite ($MnO{\cdot}Cr_2O_3$ or $FeO{\cdot}Cr_2O_3$) is a spinel found in chromium steels or where ferrochromium has been added. Particles are angular with a tendency to segregate.
4. Chromium sulfide (Cr_2S_3) is generally found in 1% and higher chromium steels. In cast steel they are globular, triangular or rectangular in shape and often mistaken for iron sulfide (FeS).
5. Fayalite ($2FeO{\cdot}SiO_2$) is found in steels deoxidized with silicon. Two types are typical – translucent and glassy.
6. Graphite (C) is one of the principal elements in cast irons.
7. Iron aluminate ($FeO{\cdot}Al_2O_3$) is a spinel found in steels deoxidized with aluminum. Inclusions are found in cast steels singly and as laths or tabular crystals and are often observed with iron oxide (FeO) inclusions.
8. Iron nitride (Fe_2N, Fe_4N) is found in steels high in nitrogen (or in nitrided steels), and it's not normally classified as a nonmetallic inclusion. Occurs as needles or bands that tend to follow the grain boundaries.

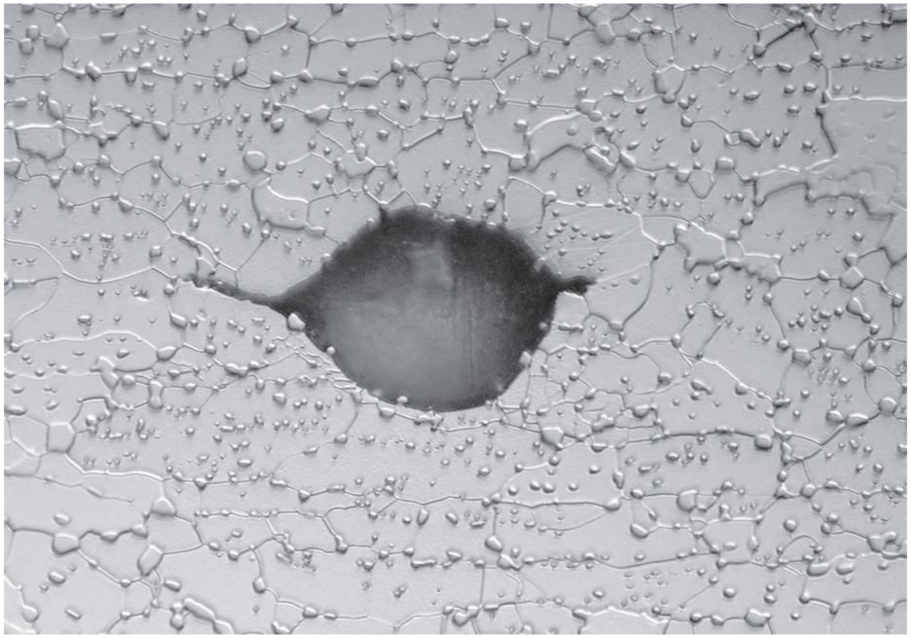

Fig. 2. Typical oxide found in SAE 1050 steel austenitized at 1550˚F and salt quenched (1250X, 2% Nital, oblique illumination)

9. Iron oxide is generally found with manganese oxide. In cast materials inclusions are globular, dendritic or angular in shape. Often mistaken for MnS (silver nitrate whitens MnS but leaves FeO unattacked).
10. Iron sulfide is found in low-manganese and high-sulfur steel (Mn:S ratio<4). Can cause hot-shortness due to its low melting point, 988˚C

(1810˚F). In cast material it is often observed at grain boundaries.

11. Manganese-iron-silicates are a complex inclusion type in which the silicates can be either acidic (large amounts of silicon with some manganese going into solution) or basic (average amounts of silicon and average-to-low amounts of manganese).
12. Manganese sulfide is the most common type of inclusion encountered. According to analysis based on the steel ingots containing 0.01-0.15% sulfur, the morphology is typically classified into three types:
    a. Type I are silicon-killed steels (or steels incompletely killed with aluminum, zirconium or titanium). MnS are globular with a wide range of sizes and often duplex with oxides.
    b. Type II are aluminum-killed steels with no excess of aluminum having a dendritic structure (called grain-boundary sulfide because MnS is forms on grain boundaries).
    c. Type III are found if excessive amounts of aluminum are present or if magnesium is used as a deoxidant. They are angular and always form as monophase inclusions.

Massive spherical inclusions are found in high-sulfur, free-machining steels. In this type of steel, low

Table 2. Classification of Inclusions

Group Number	Chemical Formula	Mineralogical Name	Optical Classification [1,2]
I	$2FeO.SiO_2$	Fayalite	Inclusions are dark and transparent in ordinary reflected light; dark in polarized light between crossed Nicols when the stage of the microscope is rotated.
	$MnSiO_3$	Rhodonite	
	$MnFe_2SiO_4$	Manganese iron-silicate	
	SiO_2	Silica (or silicon dioxide)	
II	$FeO.Al_2O_3$	Hercynite (or iron aluminate)	Inclusions are dark and translucent in ordinary reflected light; dark in polarized light between crossed Nicols when the stage of the microscope is rotated.
	MnO	Manganese oxide	
	SiO_2	Tridymite (or silica)	
	$Mn_3+2Al_2(SiO_4)_3$ or $2MnO.Al_2O_3.3SiO_2$	Spessartite (or manganese-aluminum-silicate)	
III	Al2O3	Alumina	Inclusions are dark and translucent in ordinary reflected light; bright in polarized light between crossed Nicols when the stage of the microscope is rotated.
	AlN	Aluminum nitride	
	(Fe_2SiO_4)	Fayalite	
	Fe_2SiO_4	Iron silicate	
	$MnFe_2SiO_4$	Manganese iron-silicate	
	$Mn(SiO_4)_x$	Manganese meta-silicate	
	MnO	Manganese oxide	
	$3Al_2O_3.2SiO_2$	Mullite	
	SiO_2	Silica (quartz)	
	$2MnO.SiO_2$	Tephroite	
IV	Fe_2SiO_4	Iron silicate	Inclusions are light and translucent in ordinary reflected light; dark in polarized light between crossed Nicols when the stage of the microscope is rotated.
	MnS	Manganese sulfide	
	$Mn_3+^2Al_2(SiO_4)_3$ or $2MnO.Al_2O_3.3SiO_2$	Spessartite	
V	Cr_2O_3	Chromium oxide	Inclusions are light and translucent in ordinary reflected light; bright in polarized light between crossed Nicols when the stage of the microscope is rotated.
	Fe_2O_3	Haematite (or ferric oxide)	
	Fe_2SiO_4	Iron silicate	
	$MnFe_2SiO_4$	Manganese iron-silicate	
	$Mn(SiO_4)x$	Manganese meta-silicate	
VI	Al_2S_3	Aluminum sulfide (golden yellow to tan)	Inclusions are opaque and colored in ordinary reflected light; dark in polarized light between crossed Nicols when the stage of the microscope is rotated.
	$(Fe, Mg)Cr_2O_4$	Chromite (grey)	
	Cr_2S_3	Chromium sulfide (tan and grey to yellowish-pink)	
	$FeO.Ti_2O_3$,	Ilmenite (various shades of grey to fawn)	
	Fe_4N	Iron nitride (white)	
	FeO	Iron oxide or wüstite (grey)	
	Fe_3O_4	Magnetite (grey)	
	Ti(CN)	Titanium carbonitride (yellow to pink)	
	Ti_2O_3	Titanium oxide	
	ZrN	Zirconium nitride (lemon to orange).	
VII	C	Graphite (black)	Inclusions are opaque and colored in ordinary reflected light; bright in polarized light between crossed Nicols when the stage of the microscope is rotated.
	Fe_2O_3	Haematite or Ferric oxide (grey to white)	
	Fe_2N, Fe_4N	Iron nitride (white)	
	FeS	Iron sulfide or triolite, (yellow, tan to brown)	
	TiO_2	Titanium oxide or rutile (grey)	
	Ti_2O_3	Titanium oxide (brown to purple)	
	ZrS_2	Zirconium sulfide (bluish grey to yellowish tan).	

Notes:
1. The correct setting for polarized light is that of maximum brightness.
2. The two Nicol prisms of an optical microscope are said to be crossed when the shorter diagonal of one is at right angles to the shorter diagonal of the other.

silicon contents give inclusions with the best length-to-width ratio for optimum machinability. Type I and Type III are preferred since Type II is associated with low ductility.

13. Manganese-aluminum-silicate ($2MnO \cdot Al_2O_3 \cdot 3SiO_2$) forms in the presence of manganese oxide, aluminum oxide and silica. Large inclusions have six sides, and smaller inclusions are globular in appearance.
14. Titanium carbonitride (Ti(CN)) is found in high carbon and alloy tool steels and in plain-carbon steels that have been killed with ferro-titanium. Particles are well-defined and angular and are often mistaken for titanium sulfide.
15. Titanium oxide is found in steels deoxidized with titanium or ferro-titanium. Four oxides of titanium are commonly found (Ti_2O_3, TiO, $FeO \cdot TiO_2$, Ti_2O_3). TiO appears as cubic crystals often found near Ti_2O_3 inclusions that are globular in shape and often contain iron. TiO is often confused with alumino-silicates.
16. Zirconium nitride (ZrN) is found in steel that has been deoxidized with zirconium. In cast materials, inclusions can be square, rectangular, triangular, trapezoidal or octagonal. Low zirconium content produces softer inclusions. May be found within an oxide inclusion.
17. Zirconium sulfide (ZnS_2) is found in steels deoxidized with zirconium. Inclusions with less zirconium tend to deform more easily. In cast material, the inclusions are globular.

Scanning Electron Microscopy (SEM) Methods

Since modern steels have fewer and finer nonmetallic inclusions, the use of scanning electron microscopy (SEM) has become popular for those who have this tool available. Individual inclusions often consist of more than one type, such as sulfides and silicates, so energy dispersive spectroscopy (EDS) analysis is a useful tool. EDS offers both point analysis and X-ray maps to distinguish among types of inclusions directly, not just by inference from shape.

ASTM E2142 defines three methods of inclusion analysis. Method 1 is analogous to E45 or E1122 for automatic image analysis. Method 2 uses EDS to sort the inclusions into traditional composition classifications as defined in E45. Method 3 allows customization into classes as necessary, and it is this method that is particularly useful for duplex or complex inclusions. Although the standard is written for inclusions in steel, the method could be extended to other discrete second phases or particles.

Inclusion Control

An example of manipulation of inclusions is the modification of composition and morphology of oxides and sulfides in calcium-killed steel. Some of the reported benefits include improved castability, avoidance of tearing and reheat cracking in welding applications, improved machinability and increases in both tensile ductility and tensile strength.

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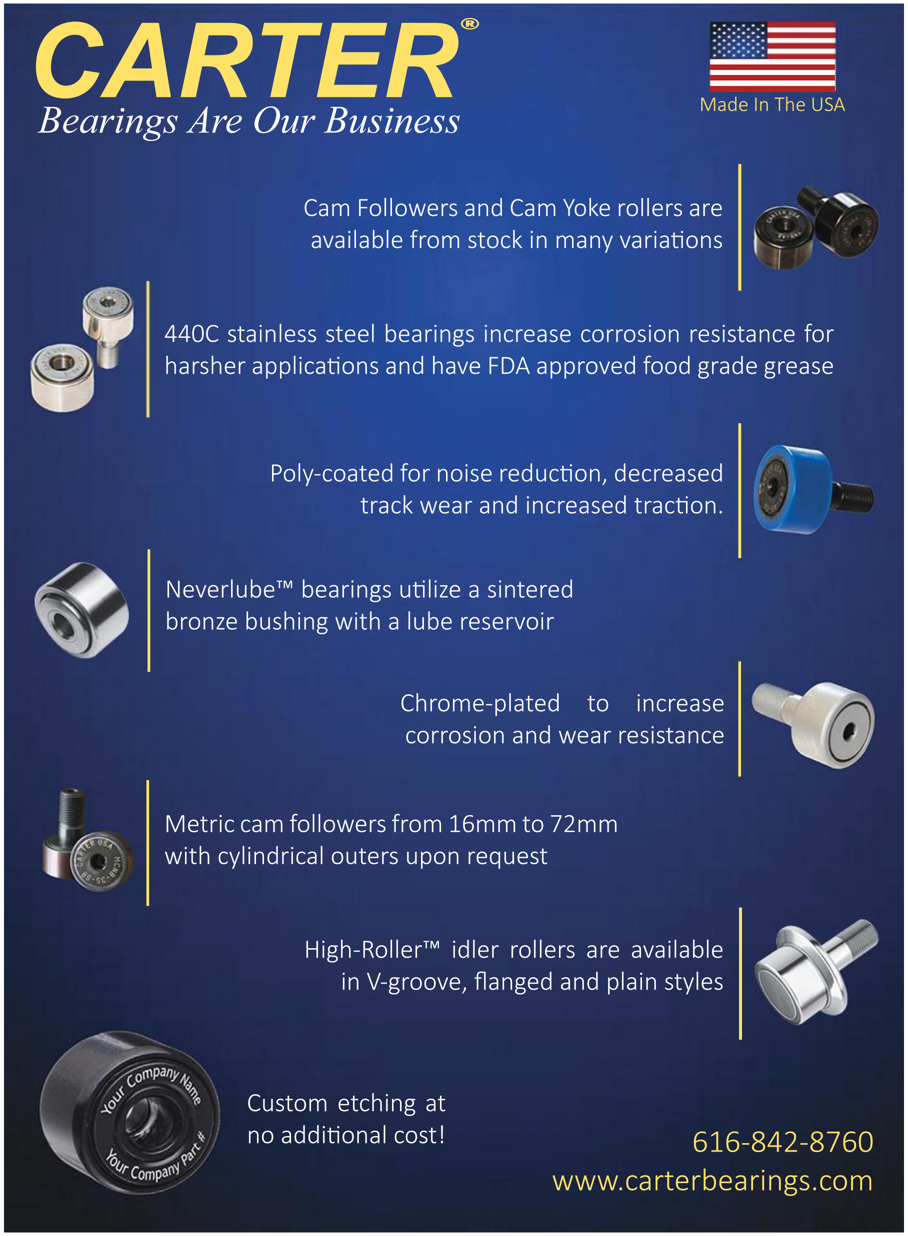

Stainless Steels Part One: Classification and Selection

Stainless steel is primarily used for applications where resistance to corrosion or heat, or both, is required. For their cost, the performance enhancement achieved by stainless steels is unmatched. In the past and continuing through today, there have been a number of ways developed to retard or prevent corrosion, the most common of these being painting or coating with metals and non-metals. Stainless steels offer an attractive alternative. Let's learn more.

A Little History

An Englishman named Harry Brearley is considered the first not only to recognize the superior corrosion resistance of an iron-based alloy containing chromium but also put this property to use for making "rustless" cutlery from a 12.8% Cr alloy. Brearley's discovery, which included a heat treatment to harden the alloy, was the result of a chance observation. While trying to prevent corrosion and fouling in rifle barrels, he alloyed iron with chromium and observed during metallographic work that these steels resisted attack by etchants. He later gave these ferritic iron-chromium alloys the name "stainless" steels. This name was also applied to the austenitic iron-chromium-nickel compositions, which were being developed into commercial products in Germany about the same time.

Today, over fifty grades have designation numbers, and there are at least another 100 non-standard compositions, which are marketed under various trade names.

Commonality Between Grades

Chromium is the common denominator among the various grades of stainless steel; all grades of stainless steel contain a minimum of 11.5% chromium. This is the minimum amount required for a stainless steel to be resistant to rusting in a non-contaminated outdoor environment with 100% relative humidity. Resistance to oxidizing acids is another property that they share.

Stainless steels are typically divided into the following major sub-categories:

- Austenitic
- Martensitic

- Ferritic
- Duplex
- Precipitation hardening
- Superalloys

Austenitic Grades

Austenitic grades (Fig. 1) are those alloys that are widely used in corrosive environments, such as the chemical and food industry. The austenitic grades are non-magnetic, have unusually good mechanical properties and do not respond to conventional quench hardening heat treatments. They consist of the iron-chromium-nickel steels, widely known as the 300 series. The 200 series are also members of this group, where some of the nickel has been replaced by manganese.

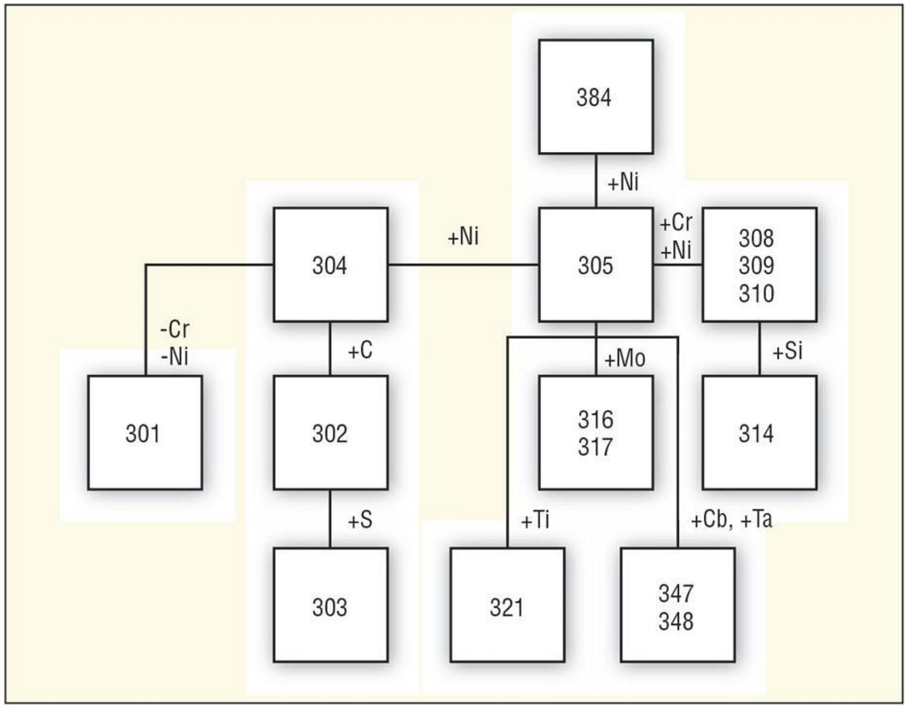

Fig. 1 Austenitic Group [2]

Martensitic Grades

Martensitic grades (Fig. 2) were developed in order to provide a group of stainless alloys that would be corrosion resistant and hardenable by heat-treating. Their crystal structures change on heating and cooling. The martensitic grades are straight chromium steels containing no nickel. They are magnetic and are used in applications where hardness, strength, and wear resistance are important considerations. Steels in this group carry the 400 series designation (as do some of the ferritic stainless steels – See Fig. 3).

The chromium content of these steels is generally lower than for the austenitic grades and, in general, the corrosion resistance of the martensitic grades is far lower than the austenitic grades (and somewhat lower than the ferritic grades).

Ferritic Grades

Ferritic grades have been developed to provide a group of stainless steels to resist corrosion and oxidation while being highly resistant to stress corrosion cracking. The structure is ferritic at all temperatures (hence the name). These steels are magnetic but cannot be hardened or strengthened by heat treatment. They can be cold worked and softened by annealing if required. Nitriding is the only heat treatment that is sometimes applied to ferritic grades. As a group, they are more corrosion resistant than the martensitic grades but generally inferior to the austenitic grades. Like martensitic grades, these are straight chromium steels with no nickel. Automotive applications are commonplace.

Precipitation Hardening Grades

These grades of stainless steels were developed primarily for use as aerospace materials but now are gaining wider commercial appeal since they are cost effective for a variety of applications and are available in a range of products (bar, rod, wire, forgings, sheet, and strip). As a class, they offer a unique combination of strength, fabricability, ease of heat treatment, and corrosion resistance not found in the other grades. They are classified as the 600 series but are also known by the

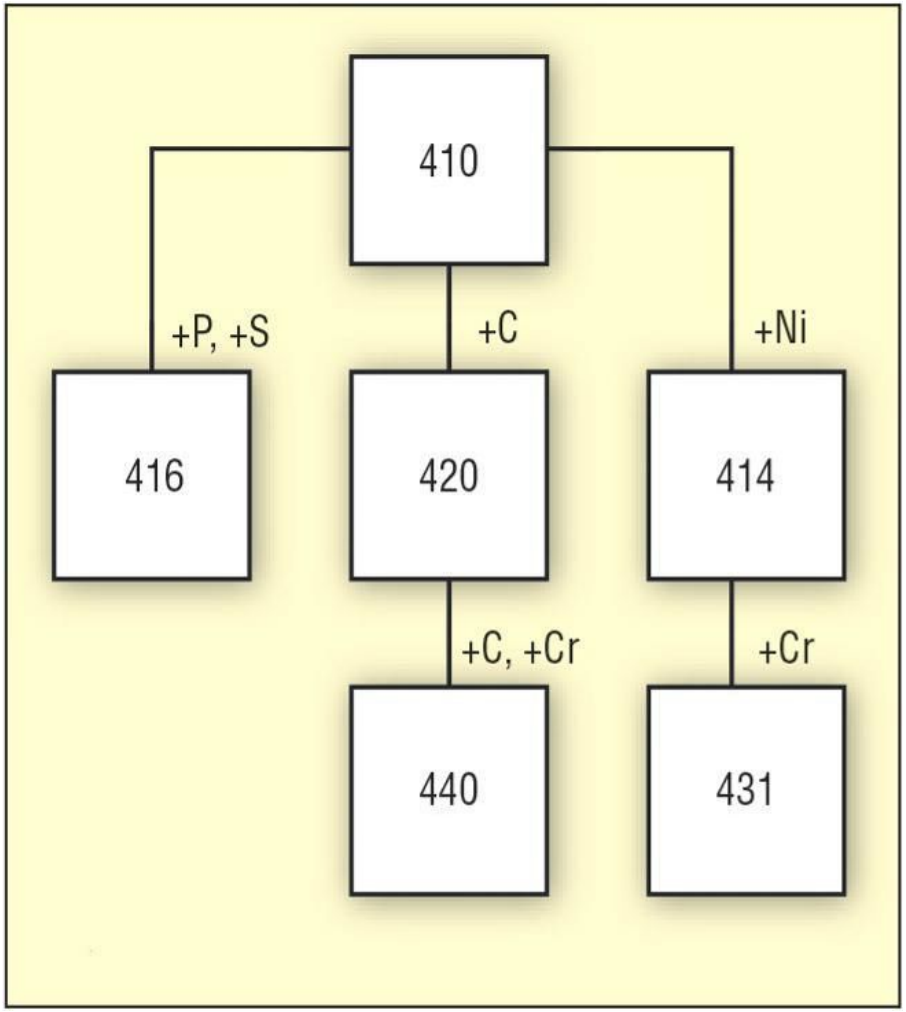

Fig. 2 Martensitic Group [2]

designations 13-8Mo, 15-5PH, 17-4PH, and 17-7PH. The austenitic precipitation hardenable alloys have, to a large extent, been replaced by the more sophisticated and higher strength superalloys. The martensitic precipitation-hardenable stainless steels are the workhorse of the family.

Duplex Grades

Duplex grades are the newest of the stainless steels. These materials are a combination of austenitic and ferritic materials. They have higher strength and superior resistance to stress corrosion cracking. These are usually mill ordered materials with designations such as 2005.

Superalloy Grades

These include a variety of alloys with very large amounts of nickel, chromium, and molybdenum, as well as other alloying elements designed to enhance performance and resist attack at high temperatures. These alloys are based on nickel, iron-nickel, and cobalt-nickel and exhibit a good combination of mechanical strength and resistance to surface degradation. They are not usually grouped with the stainless steels and have instead their own classification.

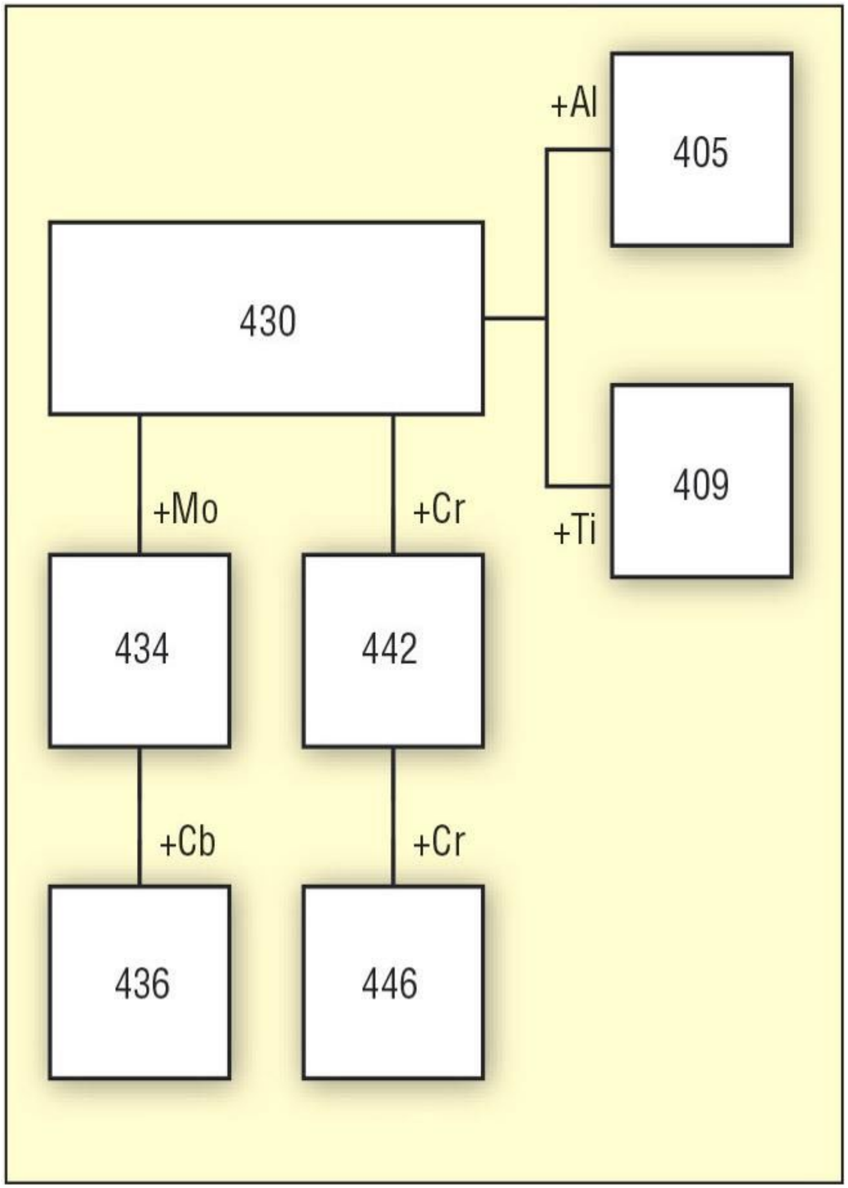

Fig. 3 Ferritic Group[2]

Considerations When Selecting Stainless Steels

The correct choice of the best stainless steel for a given application is dependent on a number of factors. Key considerations include:

1. Is the material suited for the intended service? That is, does it or can it be processed to have the required material and mechanical properties?
2. Is it economical? Consider total manufactured cost not just material cost.
3. Are the required properties achievable with the grade selected (and what the viable alternatives are)? The design engineer should consult with the material producer to assist in the selection process.
4. Can it be easily fabricated? Important considerations include ductility, formability, weldability, and machinability.
5. Is it commercially available where and when it is needed? Choose standard grades when all design and manufacturing issues are equal.
6. Are special grades the better choice? Use special grades where and when needed, especially if a property or service environment dictates. Be sure to understand if there are restrictions to the use of these special grades (e.g. FDA approval in the food industry).
7. Consider service life as well as potential problems.

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Metallic Glasses

A new type of metal may soon find its way into our everyday lives; one that is up to twice as strong as typical titanium alloys and much tougher than ceramics, yet is elastic and springy, with very high wear and corrosion resistance characteristics. This extraordinary family of materials is called metallic glass, and is currently the subject of intense research among materials scientists. Let's learn more.

What are Metallic Glasses?

Metallic glasses, also called amorphous metals, have existed in thin ribbon form since the 1960s. They are produced by rapid (on the order of 10^5-10^6 K/s) cooling from the liquid state. Metallic glasses differ from conventional metals in that they lack crystalline structure (Fig 1). The atoms in the amorphous structure are randomly arranged, like in a liquid, rather than sitting on a repeatable, orderly lattice. This lack of crystalline structure means that metallic glasses also lack crystalline defects, such as grain boundaries and dislocations. Without these "weak spots," metallic glasses have extraordinary mechanical properties, magnetic behavior, and corrosion resistance.

By comparison, grain boundaries and dislocations in metals (Fig. 2) result in much lower strength than the theoretical maximum that would be achieved if they were single flawless crystals.

Bulk Metallic Glasses

The very name metallic glass signifies a material that is made out of metal atoms that exist in a disordered, glassy state. Bulk metallic glass (BMG) is a particular type of metallic glass, just as maraging steel is a particular type of steel. There are even sub-classes of bulk metallic glasses, each having different compositions and slightly different properties.

The recent development of bulk, or three-dimensional form, metallic glasses has opened the door for use of these fascinating materials in structural applications, because bulk forms are more than 20 times thicker than ribbons (typically 40 µm). These alloys require cooling rates of only 1 to 100 K/s, so they are suitable for conventional casting (up to 1 cm, or 0.4 in.). The possibly of molding them into complex shapes will allow more precision parts and avoid the costs and waste associated with machining.

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All metallic glasses are made up of metal atoms, such as copper, nickel and titanium. The unique structure of metallic glass gives it interesting and useful properties.

Properties of bulk metallic glass

Metallic glass looks much like its metal cousins. They are gray and opaque, with a very shiny surface. The surface, though, is so smooth that paint has difficulty sticking to it. Otherwise, it is difficult to distinguish metallic glasses from ordinary metals simply by looking at them, but this similarity hides the great differences at the atomic level, which results in very different properties.

Bulk metallic glasses resist breaking when stretched, they keep their shape and they are hard to shatter. In scientific terms, they possess high elastic strain limit, high yield strength and high fracture toughness. For example, Vitreloy 1, a bulk metallic glass consisting of zirconium and titanium, has properties (Table 1) that compare favorably with its metal counterparts.

Applications of bulk metallic glass

Bulk metallic glasses have found more uses in the past 15 years or so, and have begun replacing some conventional materials such as crystalline metals, metal alloys and high-tech ceramics (Fig. 3). Among current applications are structural engineering materials, consumer electronic components, jewelry, replacement joints and recreational equipment such as skis, tennis rackets and golf club heads.

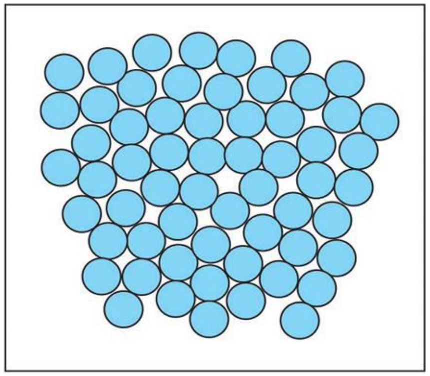

Fig. 1. In amorphous structure, atoms are arranged in a random fashion, similar to their arrangement in the liquid state.

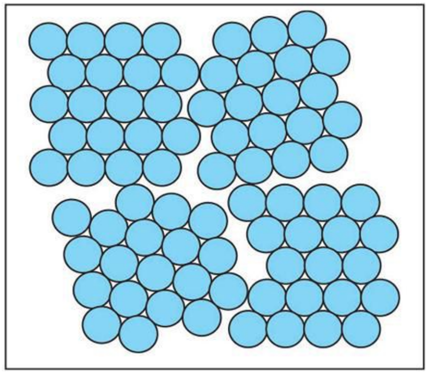

Fig. 2. Illustration of four grains in a crystalline atomic structure; atoms form an orderly lattice in each grain. Grain boundaries are one example of a crystalline defect.

Bulk metallic glass was also used on board the Genesis spacecraft, the fifth mission in NASA's Discovery program, which was launched in August 2001 to collect samples of solar wind. Unfortunately, the parachute on the Genesis sample return capsule failed to deploy on reentry in September 2004. The capsule crashed in the Utah desert but fragments from the sample return container were shipped to Johnson Space Center in early October. Scientists are

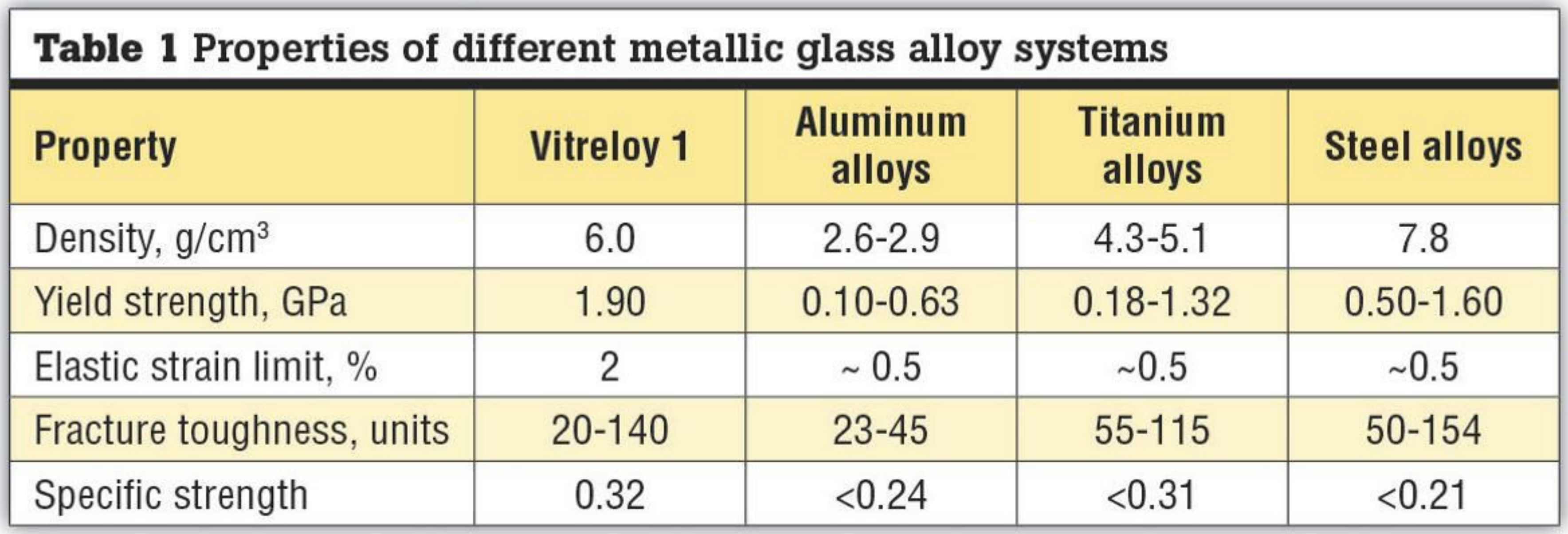

Table 1 Properties of different metallic glass alloy systems

Property	Vitreloy 1	Aluminum alloys	Titanium alloys	Steel alloys
Density, g/cm^3	6.0	2.6-2.9	4.3-5.1	7.8
Yield strength, GPa	1.90	0.10-0.63	0.18-1.32	0.50-1.60
Elastic strain limit, %	2	~ 0.5	~0.5	~0.5
Fracture toughness, units	20-140	23-45	55-115	50-154
Specific strength	0.32	<0.24	<0.31	<0.21

optimistic that they will be able to salvage useful data from the samples. The probe's solar wind collector was made of a bulk metallic glass. The bulk metallic glass plate on Genesis was designed to absorb ions coming from the sun for analysis back on Earth. Metallic glass was used because its surface dissolves evenly, alloying the trapped ions to be released in precise layers.

The future of bulk metallic glass

The discovery of bulk metallic glass little more than ten years ago was one of the most exciting recent breakthroughs in materials science. With properties (such as strength and elasticity) far superior to those of conventional metals combined with fabrication methods similar to those for plastics, bulk metallic glasses have tremendous potential.

For example, recent work at Los Alamos National Laboratory (Los Alamos, NM; www.lanl.gov) has produced a bulk metallic glass made of pure zirconium metal having, among other properties, high thermal stability. The material

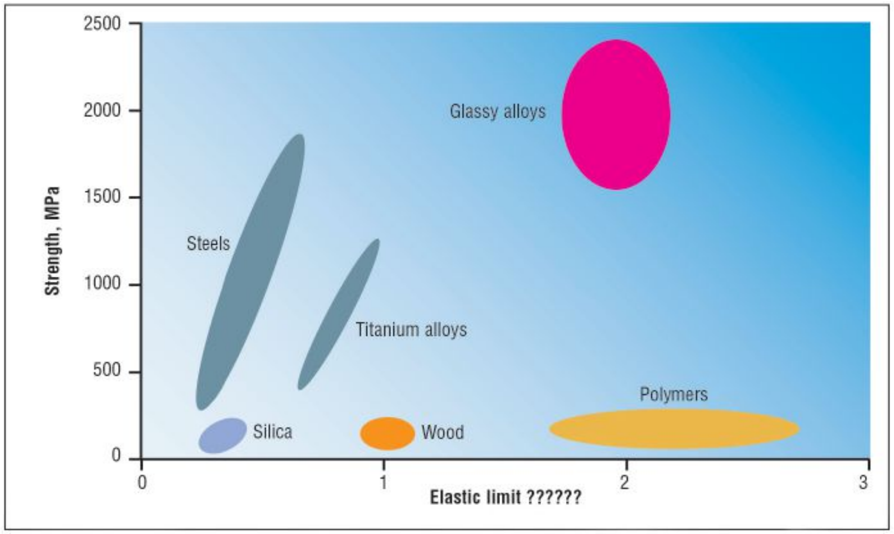

Fig. 3. Metallic glasses offer a good combination of strength and toughness compared with these selected engineering materials.

remains as glass at temperatures above 1600°F (870°C). Traditional BMGs return to a crystalline structure, and thus lose many important properties, at temperatures as low as 800°F (430°C).

Although current uses are limited to relatively a small number of applications, the potential of these new materials will open up a much wider range of new applications in the near future.

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Grain Size and Its Influence on Materials Properties

As metallurgists, we know the importance of grain size, and as heat treaters, we need to be sure we understand how grain size will affect mechanical properties especially when designing our process recipes. Let's learn more.

Grain size specifics

The grain size of carbon and alloy steels is generally understood to mean prior austenitic grain size. On heating steel through its critical range, transformation to austenite takes place. The austenite grains are extremely small when first formed, but grow in size as the time and temperature are increased. The grain size will remain small for steels held at temperatures just above the (upper) critical. At higher temperatures, however, different steels shown wide variation in grain size, depending on chemical composition and steelmaking (deoxidation and prior treatment) practices.

Steels in which elements such as Al and V have been added produce finelydispersed, not readily soluble and highly refractory carbides or oxides, and maintain a slow rate of grain growth at 925°C (1700°F) and above. Steels without these additions usually develop a relatively large austenitic grain size at temperatures somewhat below this temperature. If you overheat steels with grain refinement additions, such that the fine dispersed phases dissolve or coalesce, then you can end up with a larger grain size than for alloys where these grain refinement additions are not made.

Today, research is underway to find ways to increase carburizing temperatures as high as 1150°C (2100°F) while avoiding excessive grain growth. Microalloying elements such as Al, Nb and Ti have shown considerable promise [1].

Grain size effect on properties

Grain size has a measurable effect on most mechanical properties. For example, at room temperature, hardness, yield strength, tensile strength, fatigue strength and impact strength all increase with decreasing grain size. Machinability is also affected; rough machining favors coarse grain size while finish machining favors fine grain size. The effect of grain size is greatest on properties that are related to the early stages of deformation. Thus, for example, yield stress is

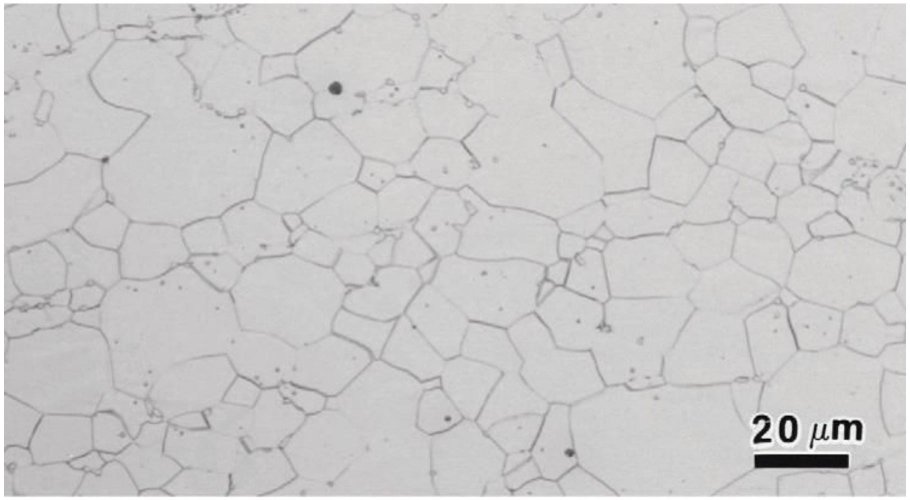

Fig. 1 Ferrite grain structure of a lamination steel; 2% nital etch.

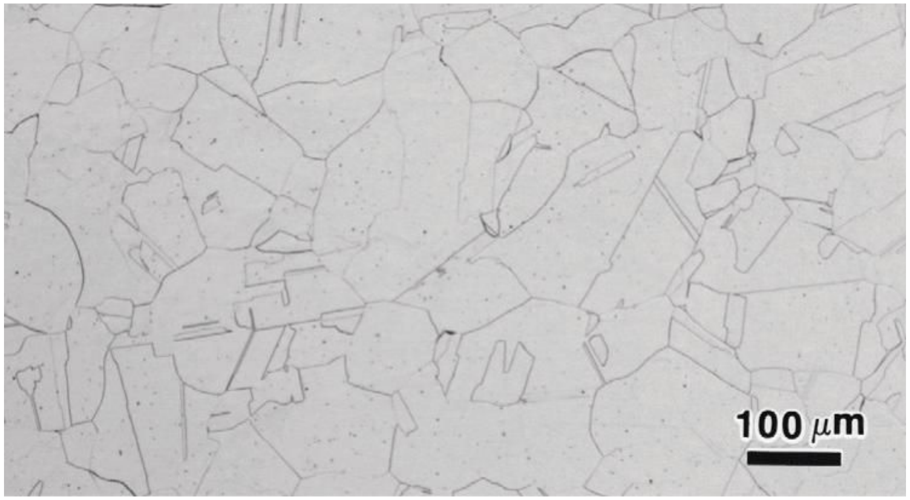

Fig. 2 Austenite grains with annealing twins in AISI Type 316 stainless steel; Kalling's number 2 etch.

more dependent on grain size than tensile strength [2, 3].

Fine-grain steels do not harden quite as deeply and have less tendency to crack than coarse-grain steels of similar analysis. Also, fine-grain steels have greater fatigue resistance, and a fine grain size promotes a somewhat greater toughness and shock resistance. Steels made fine grained by addition of aluminum have machinability inferior to those made without aluminum. Also, cold working frequently alters grain size by promoting more rapid coarsening of the grains in critically stressed areas. The original grain size characteristics, however, can usually be restored by stress relieving. Coarse-grain steels have better creep and stress rupture properties because diffusion at high temperatures is impeded by subgrain low-angle boundaries present in coarse-grain steels [4].

Measuring grain size

Grain size can be measured using an optical microscope on a transverse metallographic mount (because rolling elongates the grains) by counting the number of grains within a given area, by determining the number of grains that intersect a given length of a random line, or by comparison with reference pictures (standards). Grain size can

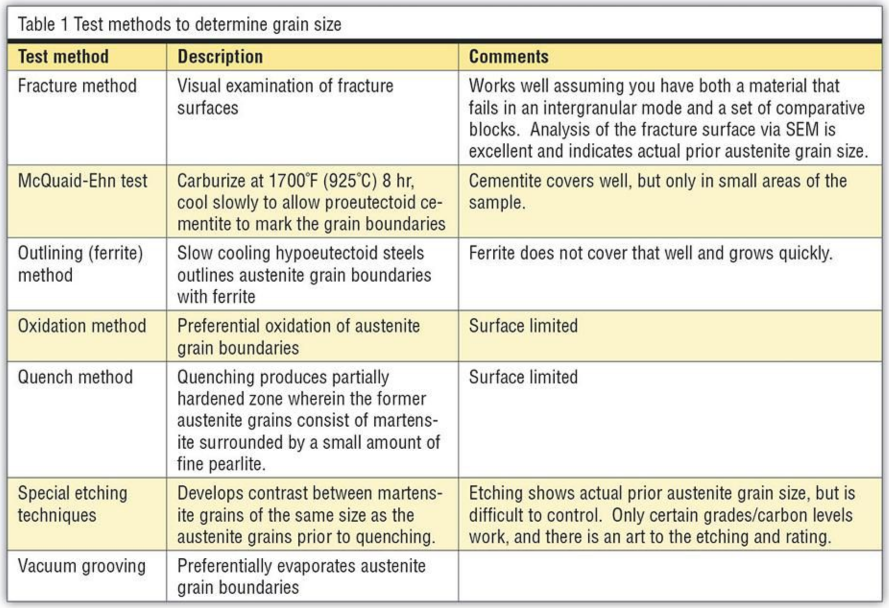

Table 1 Test methods to determine grain size

Test method	Description	Comments
Fracture method	Visual examination of fracture surfaces	Works well assuming you have both a material that fails in an intergranular mode and a set of comparative blocks. Analysis of the fracture surface via SEM is excellent and indicates actual prior austenite grain size.
McQuaid-Ehn test	Carburize at 1700˚F (925˚C) 8 hr, cool slowly to allow proeutectoid cementite to mark the grain boundaries	Cementite covers well, but only in small areas of the sample.
Outlining (ferrite) method	Slow cooling hypoeutectoid steels outlines austenite grain boundaries with ferrite	Ferrite does not cover that well and grows quickly.
Oxidation method	Preferential oxidation of austenite grain boundaries	Surface limited
Quench method	Quenching produces partially hardened zone wherein the former austenite grains consist of martensite surrounded by a small amount of fine pearlite.	Surface limited
Special etching techniques	Develops contrast between martensite grains of the same size as the austenite grains prior to quenching.	Etching shows actual prior austenite grain size, but is difficult to control. Only certain grades/carbon levels work, and there is an art to the etching and rating.
Vacuum grooving	Preferentially evaporates austenite grain boundaries	

range from 00 to 14.0 (0.5080 to 0.0028 mm) according to ASTM E112. For coarser structures, the number of grains can be counted manually within a certain area, and then calculating the grain size. In measuring austenitic grain size, the time and temperature must be constant to produce reproducible results.

The characteristics of the individual grains also add to the complication of measurement. First, the three-dimensional size of the grains is not constant and the sectioning plane cuts through the grains at random. Thus, on any cross section, a range of sizes is observed, none larger than the cross section of the largest grain sampled. Grain shape also varies, particularly as a function of grain size.

Remember that grain size measurement is also complicated by the different types of grains that can be present in metals, although their fundamental shapes are the same. In body-centered cubic (BCC) metals, such as Fe, Mo, and Cr, we have grains produced in ferritic structures (Fig. 1); in face-centered cubic (FCC) metals, such as Al, Ni, Cu, and certain stainless steels, we have grains produced in austenitic structures (Fig. 2). The grains have the same shapes and are measured in the same way, but we must be careful in describing what kind of grains are being measured.

For example, in the FCC metals, there may be so-called twin boundaries within the grains, produced by annealing or deformation. Twins are ignored if trying to define the grain size. However, if trying to establish a relationship between microstructure and properties (strength, for example), twin boundaries must be taken into consideration as they influence dislocation movement, just as grain boundaries do. There also are pearlite packet boundaries in steels. Therefore, we must recognize the intent of the work being performed.

A number of standard measurement methods can be used (Table 1), and it is important to recognize that all of them are very subjective. Software has been developed to help make grain measurement and counting easier (although it still is necessary to understand what you are trying to measure to ensure representative results). Automated systems usually use simple menus and easy "wizard" guides; use vertical, horizontal, concentric circle or diagonal line intercept methods; perform calculations according to ASTM standards; calculate average grain size from multiple images; and manually add or remove points.

ASTM standards

ASTM International (www.astm.org) has a number of standards for determining the grain size of various materials. For steels, E112: Standard Test Methods for Determining Average Grain Size applies. ASTM standards have also been introduced to deal with particular situations including:

- E930: Standard Test Methods for Estimating the Largest Grain Observed in a Metallographic Section, (ALA Grain Size) to handle the measurement of occasional very large grains present in an otherwise uniform, fine grain size dispersion
- E1181: Standard Test Methods for Characterizing Duplex Grain Sizes or for rating the grain size when the size distribution is not normal; for example, bimodal or duplex steels
- E1382: Standard Test Methods for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis.

A lower ASTM grain size number means the greater the number of grains, as shown in the following table. There also is an equation expressing the relationship between grain size and yield strength. Bending fatigue also follows a similar relationship.

ASTM grain size in terms of the number of grains

ASTM grain size number	Number of grains/in.^{2} (mm^2) at 100×
1	Up to 1-1/2
2	1-1/2 - 3
3	3 - 6
4	6 - 12
5	12 - 24
6	24 - 48
7	48 - 96
8	96 and over

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H-Band and RH-Band Steels

When and why do we use hardenability-band (H-band) and restricted-hardenability-band (RH-band) steels is a question often posed to The Doctor by design engineers. Why not use these steels all the time is a common question from heat treaters. Let's learn more.

The choice of material must be made only after careful consideration of the performance demanded by the application. For example, gears under load (Fig. 1) are subject to gradient stresses both on the active flank and at the root fillet. Proper material selection as well as heat treatment will produce strength gradients that are adequate to withstand these stresses and provide an acceptable margin of safety.

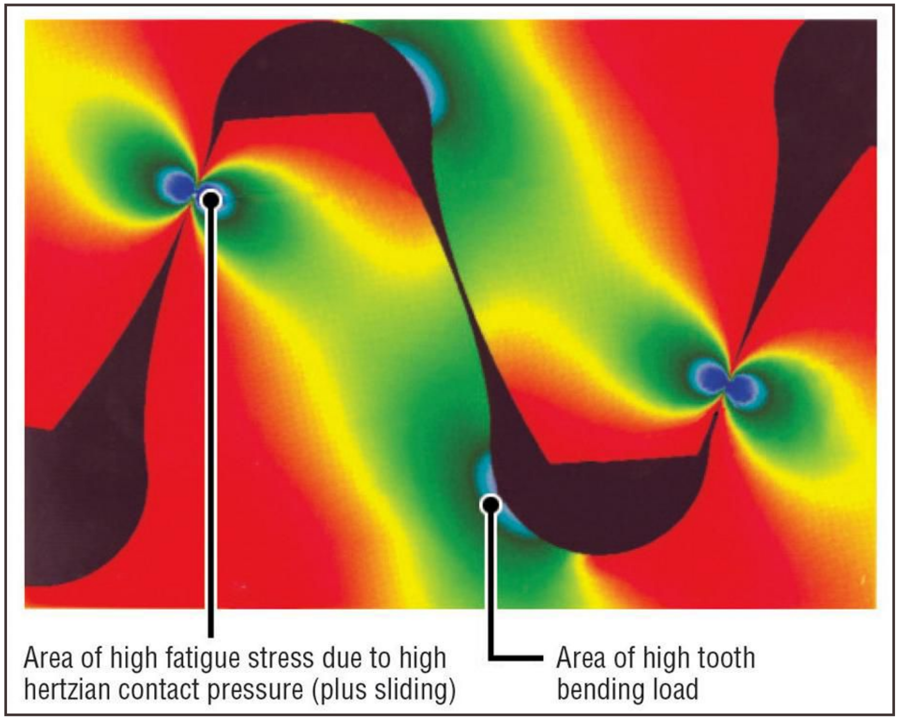

Fig. 1. Gear stress pattern

Material choice must be a balance between overall cost and required service life. The Model of Gear Engineering (Fig. 2) tells us that cost is the most important consideration among purchasers of raw material followed by material properties. From an engineering perspective, however, key design considerations require an analysis of the type of applied load, whether gradual or instantaneous, and the desired mechanical properties, such as bending fatigue strength or wear resistance. The required mechanical properties are the critical consideration and will define core strength and heat-treating requirements. Manufacturing economics plays an important role as well, and once again, cost manifested in reducing the number of manufacturing operations is paramount.

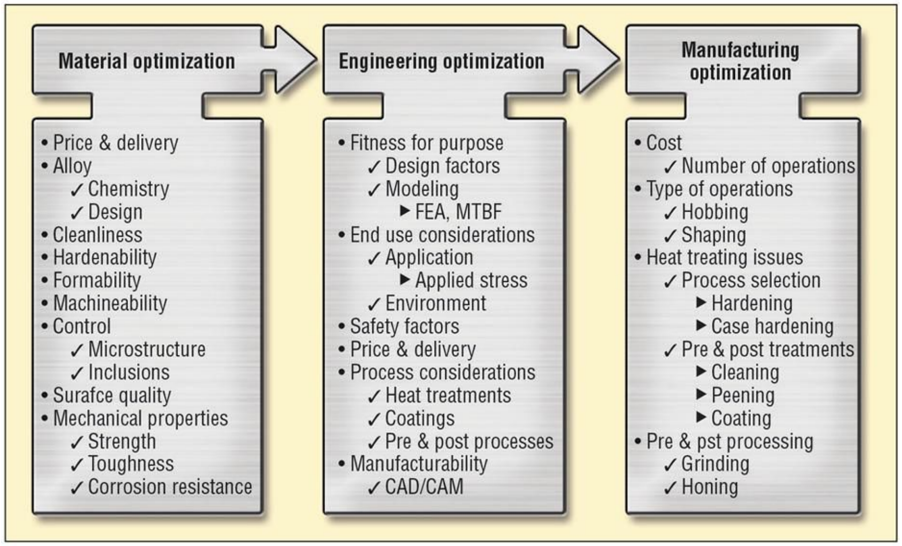

Fig. 2. Model of gear engineering

In our gear example, each area in the gear-tooth profile sees different service demands. In the root area, good surface hardness and high residual compressive stress are desired to improve bending fatigue life. On the active flank, a combination of high hardness and adequate subsurface strength are necessary for adequate resistance to macropitting and sub-case fatigue.

Proper material hardenability selection is critical to the success of any product since this affects how the material can be quenched (Fig. 3). If the material hardenability is too high, the material will be costly, susceptible to quench cracking and produce high core hardness. For a given gear-tooth pitch size, for example, the result will be more distortion and higher mid-tooth core hardness than is necessary or desirable. If the material hardenability is too low, the material will exhibit low hardness with non-martensitic transformation products (NMTP) present in the microstructure, often exhibit uneven hardness and be prone to distortion and unpredictable size changes. For gears, the core hardness needs to be in a specific range to support the case but not too high to cause cracking at the case/core interface. Many manufacturers find that the use of tighter hardenability (RH-band) steels ensures that the proper mid-tooth core hardness is consistently achieved.

Restricted hardenability carburizing steels also make distortion more predictable from lot to lot (Fig. 4 - 5). This should not adversely affect cost or availability of the raw material due to the better steelmaking processes now being used by forging and bar-stock suppliers. New steelmaking processes are yielding more consistent hardenability, and some H-band steels actually meet RH-band requirements without the added cost. Another advantage to heat treaters is that RH-band grades simplify process control and avoid “tweaking” the cycle recipe each time a load is run. Finally, in medium carbon steels using an RH-band grade, surface hardness by induction hardening has been reported to be more

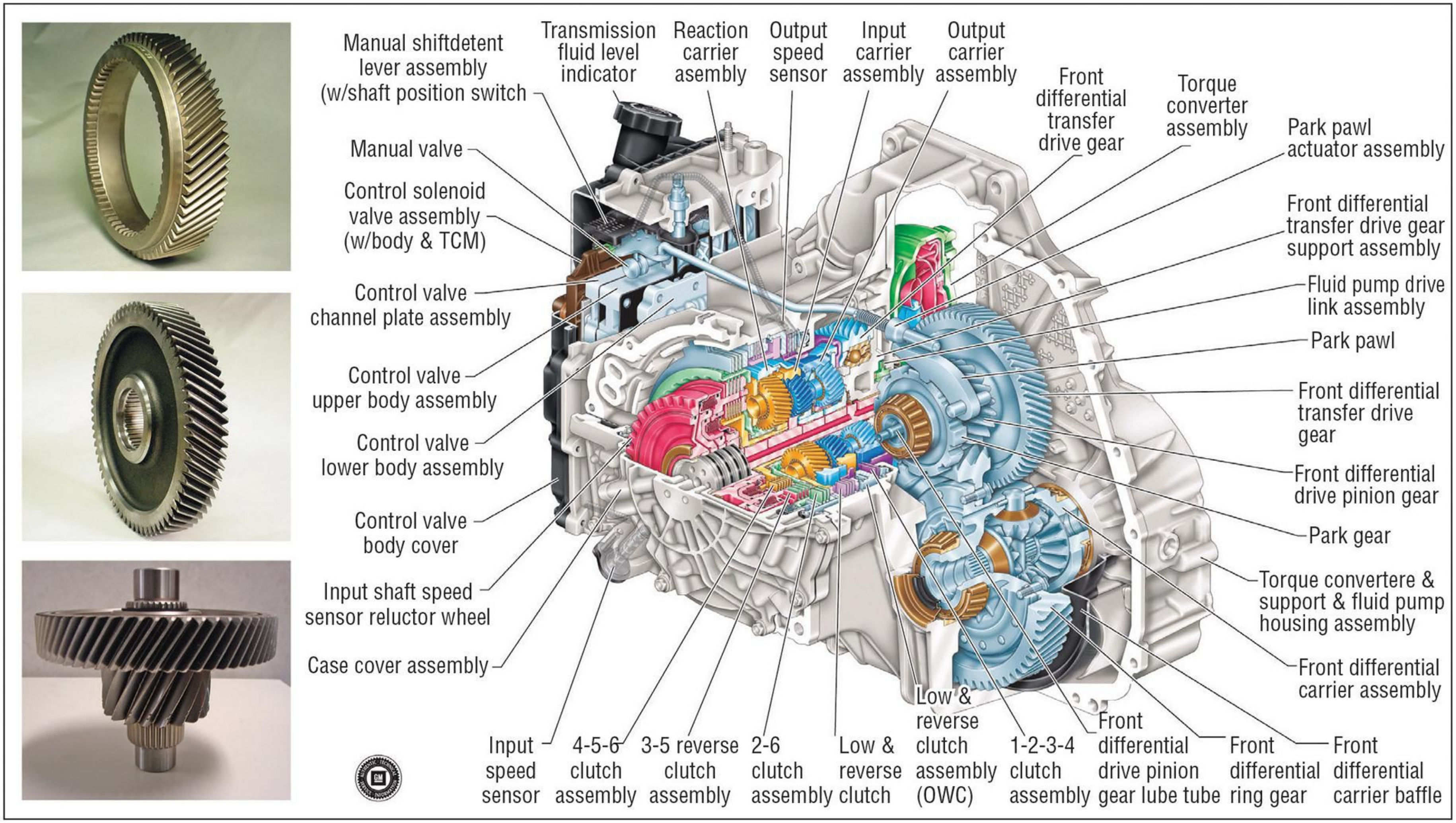

Fig. 3. 6T70/75 automotive transmission gears

Table 1. Comparison of H-band and RH-band steel chemistries

	%C	% Mn	% Si	%S	%P	%Ni	%Cr	%Mo	%Cu	%Fe
8620H	.17/.23	.60/.95	.15/.35	.25	.25	.35/.75	.35/.65	.15/.25	.35	balance
8620RH	.18/.23	.70/.90	.15/.35	.25	.25	.40/.70	.40/.60	.15/.25	.35	balance

Table 2. Comparison of H-band and RH-band steel Jominy

	J1	J4	J6	J8
8620H	41-48	27-41	21-34	N/A-30
8620RH	42-47	30-38	24-31	21-28

Fig. 4. 8620RH clutch pack for off-road vehicle transmission

Fig. 5. 8822RH truck ring-and-pinion set

achievable.

H-band and RH-band steels incorporate slightly different carbon and possibly other chemistry ranges (Table 1) and hardenability (Table 2); generally have a broader chemistry range than standard alloy steels (to allow steel producers to design the optimum alloy combination); and have a more restricted hardenability range at each Jominy distance.

In general, H-band steels offer a wide range of mechanical properties that depend on the development of tempered martensite after quenching and tempering. RH-band steels will exhibit a hardness range not greater than 5 HRC at the initial position (on the end-quench hardenability bar) and not greater than 65% of the hardness range for standard H-band steels in the “inflection” region. Generally, the RH-band follows the middle of the range corresponding to standard H-band.

From a design standpoint, the depth to which parts are hardened not only affects their serviceability but also their cost. While the hardening need be no deeper than is required to provide the strength to sustain load at any given depth below the surface, service duty plays a critical role. For example, parts loaded principally in tension and parts operating at high hardness and high stress levels should be through-hardened. In contrast, the commercial practice, used for many parts including automotive, of hardening to 80% (or more) martensite at ¾ radius of the finished part is usually sufficient. For parts used in applications in which they are moderately stressed or are expected to see low deflection under load, less depth of hardening is required.

Finally, the center of a 3.375-inch (85-mm) round of 4340H material can be austenitized and oil quenched to 80% martensite (46HRC). By contrast, when quenching to 95% martensite (55HRC), the bar size drops to a 2-inch (50-mm) round (Fig. 6).

Final Thoughts

With the vast array of domestic and foreign steels available to choose from, with standard and non-standard chemistry, the challenge of selecting the right steel for a given application seems daunting. The use of H-band steels simplifies this task because direct comparisons can be made between the various steels to quantify their response to heat treatment. Different steels are often grouped by their hardenability requirements, allowing greater selectivity within a given design and making the job of the heat treater easier.

RH-band steels provide even more confidence that, for example, core hardness within a specific range on a gear tooth can be achieved repeatedly, whereas an H-band steel might harden to the high end in one load and to the low end of the range in another load.

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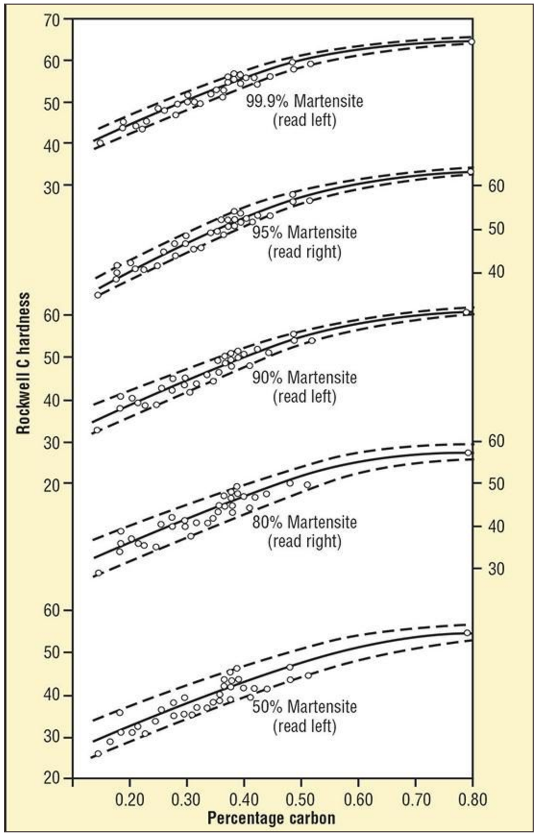

Fig. 6. Hardness of martensite structures as a function of carbon content[1]

The Heat Treatment of Light Metals

When we deal with applications where strength-to-weight ratio is a critical consideration, we often turn to solutions involving the so-called "light metals." Aluminum, magnesium, titanium and in some cases beryllium enhance engineering performance while minimizing the weight of components and structures. Most of us involved in heat treating these materials know how we do it, but it is equally important to understand why we do it. Let's learn more.

First, it is important to remember that light metals possess other physical properties that may be of importance in selection or service. These include the good electrical and thermal conductivity of aluminum, the machinability and noise dampening of magnesium or the extreme corrosion resistance of titanium. Our heat-treatment processes must retain and possibly enhance these properties.

Diffusion

As with all nonferrous alloys, the role of diffusion-related mechanisms in light metals must be understood to select the optimum heat treatment. The time/temperature relationship (Fig. 1) is critical in determining how changes will occur in the microstructures, which affect the resultant properties.

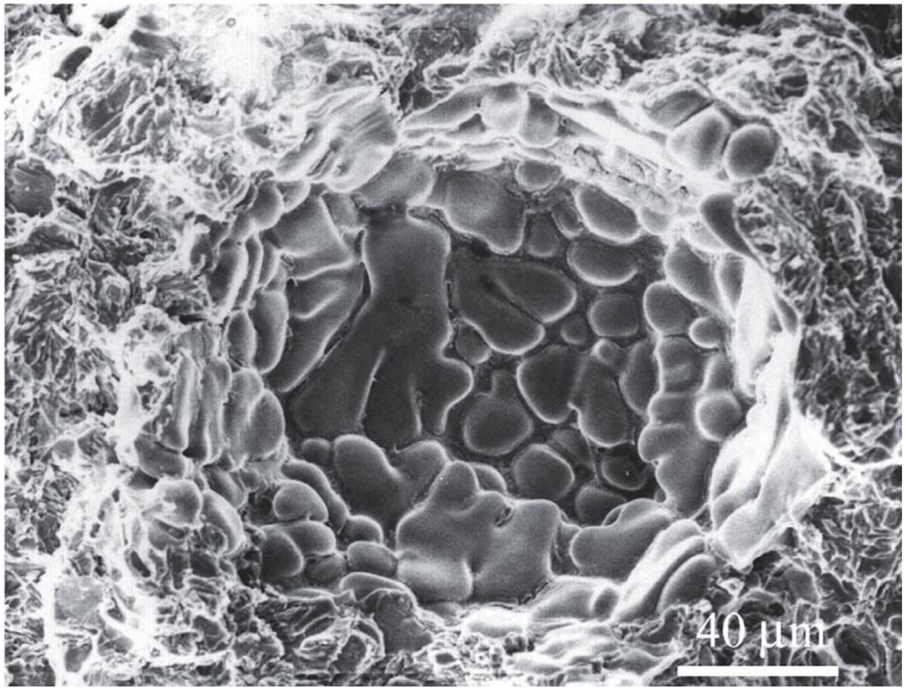

Fig. 1. Localized overheating of an aluminum-copper alloy (rosette pattern)

Diffusion can simply be thought of as the rearrangement of the atoms inside the crystal (lattice) structure of the metal. Diffusion is controlled by the rate at which atoms change

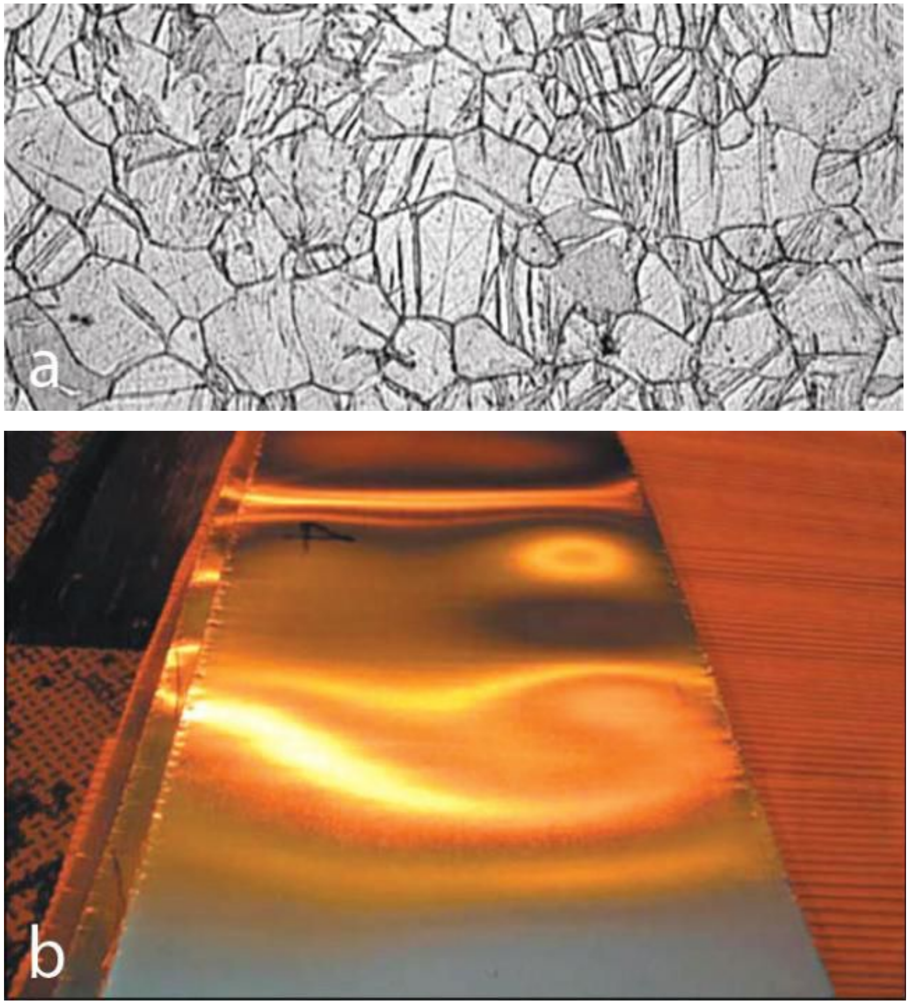

Fig. 2. Annealed AZ31 magnesium - 3% aluminum, 1% zinc alloy[2]
(a) Uniform microstructure after the 10th pass with intermediate 2-5 minute infrared heating to 750°F (400°C) with average rolling reductions of 16.5% per pass
(b) As-rolled sheet with good surface finish and minimal edge cracking

position and increases exponentially when temperature is applied. A few simple mechanisms are involved:

- **Vacancy diffusion** – the predominant diffusion mechanism in metals due to the (relatively) small amount of energy required for atom movement.
- **Chemical diffusion** – the exchange of atoms when two metals or alloys are placed in contact. Migration across the contact boundary occurs by atom exchange.
- **Interstitial diffusion** – occurs if a sufficiently small solute atom moves to a position between larger solvent atoms in an energy-favorable configuration.
- **Grain-boundary diffusion** – diffusion occurs along these defects in the crystal structure due to high interfacial energy and relatively weak bonding.

Heat Treatment

Light metals are usually heat treated either to improve mechanical properties or as a means of conditioning for specific fabricating operations. The type of heat treatment selected – annealing, homogenizing, solution treating, aging, stress relief – depends on alloy composition, the form (cast or

Fig. 3. Machined component ready for stress relief (Photograph courtesy of Solar Atmospheres, Inc.)

wrought), manufacturing methods and ultimately on the anticipated service conditions.

Annealing

In shaping of light metals by deformation – cold, warm or hot working – there is a limit to the amount of plastic deformation possible without failure by fracture. Annealing the material prior to reaching this limit places the part microstructure in a condition that can allow additional deformation to take place if necessary.

The annealing temperature for many of these metals is near the recrystallization temperature. For example, pure aluminum recrystallizes at a temperature of 300°F, and recovery/annealing temperatures normally vary from 300-650°F (150-340°C). By contrast, extensively cold-worked commercial aluminum alloys subject to recrystallization require heating for several hours into the 650-775°F (340-410°C) range.

Wrought magnesium alloys (Fig. 2) in various conditions of cold work, strain hardening or temper must be annealed for one or more hours by being heated at 550-850°F (290-455°C) depending on alloy.

By contrast, many titanium alloys are placed in service in the annealed state. The annealing of titanium and titanium alloys serves primarily to increase fracture toughness, ductility at room temperature, dimensional and thermal stability and creep resistance.

Homogenization

Most often a mill process, homogenization uses high temperatures (near the solidus) usually for prolonged periods to eliminate or reduce microsegregation – for improved workability – in castings that are to be hot or cold worked.

Solution Heat Treatment and Aging

Solution heat treating improves the strength of many light metals by causing the alloying elements to go into a solid solution and controlling the rate and extent of their return. Aging or precipitation hardening promotes full transformation of the material from its metastable state.

For example, a wide range of strength levels can be obtained in titanium alpha-beta (α-β) or beta (β) alloys by solution treating and aging. The origin of heat-treating responses of titanium alloys lies in the instability of the high-temperature beta phase at lower temperatures. Heating an alpha-beta alloy to the solution-treating temperature produces a higher ratio of beta phase. This partitioning of phases is maintained by quenching. On subsequent aging, decomposition of the unstable beta phase occurs, providing high strength.

Stress Relief

Stress relief of wrought components is performed to reduce or remove residual stresses induced by cold working (shaping, forming) and hot working (forging), straightening, welding or even cooling. The removal of such stresses helps maintain shape stability and eliminates unfavorable conditions such as the loss of compressive yield strength – the Bauschinger effect.

Stress relief of castings is particularly important to avoid residual stresses and associated warpage caused by differential cooling rates or from aggressive machining practices. Preventing stress-corrosion cracking is another reason.

For example, when magnesium extrusions are welded to hard-rolled sheet, a lower stress-relief temperature and longer time should be used to minimize distortion. It is better to use 300°F (150°C) for 60 minutes rather than 500°F (260°C) for 15 minutes. Other materials such as titanium and most titanium alloys can have a stress-relief operation performed (Fig. 3) without adversely affecting strength or ductility.

Other Factors

Good heat treating encompasses not only good maintenance practice, but also an understanding of how process and equipment variables can affect the outcome. For example, proper control of air movement promotes tight temperature uniformity, and tight seals ensure the effectiveness of a protective atmosphere or prevent air ingress under vacuum. Understanding which quench medium to employ and how to use it is important – even if it's as simple as choosing when to cool parts in still air and when to use fan cooling – as is an understanding of the variables introduced by part geometry and loading arrangement. Dimensional stability and the prediction of growth or shrinkage in normal or extreme-duty service should be considered as well.

Light-Metals Research

R&D activity is focused on better understanding of microstructures, transformations and properties at the atomic and subatomic (nano) level. This includes structure and kinetics in early stages of nucleation, dispersoid formation, recrystallization resistance and precipitation sequences. In particular, these efforts are focused on obtaining a fundamental understanding and quantitative description of reactions and processes during heat treatment to lay the foundation for improved industrial practice and design of the next generation of light alloys with improved materials properties.

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3. Additional related information may be found by searching for these (and other) key words/terms via BNP Media SEARCH at www.industrialheating.com: diffusion, recrystallization, wrought, homogenization, aging, solution treatment

Heat Treating of Aluminum Castings

The heat treatment of cast aluminium alloys (Table 1) is carried out to increase their strength and hardness and to change their physical, mechanical and metallurgical properties. Let's learn more.

Different types of castings require different thermal treatments (Fig. 1). For example, improved mechanical and physical properties can be produced in sand and permanent-mold castings by heat treatment. By contrast, some alloys – such as 443.0 that contain little or no copper, zinc or magnesium – do not respond to heat treatment and do not exhibit improvements in mechanical properties. Others, such as die castings, can only be given a stress relief (and not solution heat treated) because of their porous internal structure due to fears of surface blistering and internal porosity. Each type of heat treatment is worth understanding in more detail.

Table 1. Classification of thermal treatments of cast aluminum alloys

Suffix Designation	Heat Treatment
M	None – As cast or as manufactured
TB (T4)	Solution treated and naturally aged
TE (T5)	Artificially aged
TB7	Solution treated and stabilized
TF (T6)	Solution heat treated and fully artificially aged
TF7	Solution treated and artificially aged and stabilized
TS	Stress relieved and annealed

Heat Treatment

Solution Treatment – TB Condition (T4)

Castings are heated to a temperature just below the alloy melting point (dependent on chemical composition) and held at this temperature (dependent

Fig. 1. Cast aluminum engine block (Photograph Courtesy of Wisconsin Oven Corporation; East Troy, Wis.)

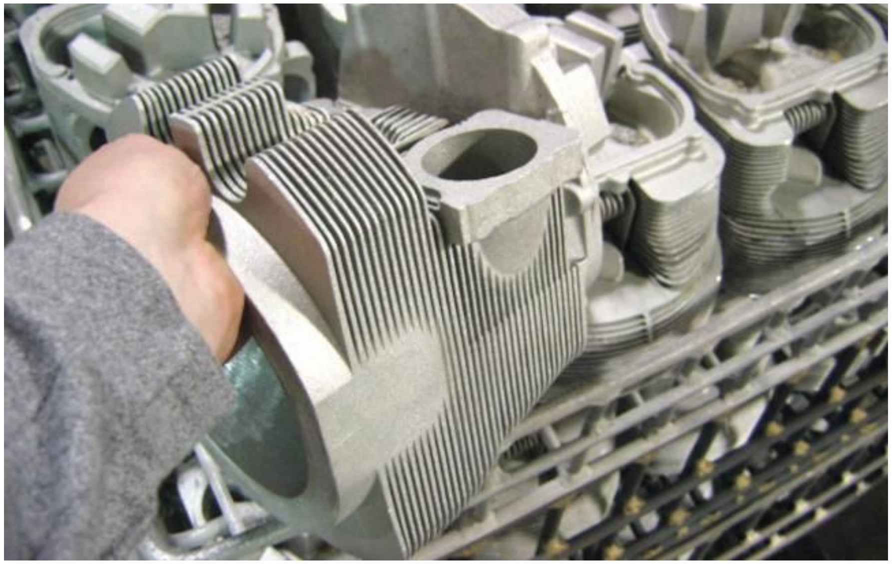

Fig. 2. Load of aluminum castings for heat treatment at a commercial shop (Photograph courtesy of Wisconsin Oven Corporation; East Troy, Wis.)

on alloy and cross-sectional thickness) a sufficient amount of time to allow the alloying elements to enter into solid solution. Upon quenching, these elements are in a super-saturated metastable state. Quench media include water, boiling water or polymer. Choice of the quenchant is often a balance between achieving mechanical properties and managing distortion while avoiding the buildup of internal stresses in the part. Although mechanical properties increase somewhat by natural aging, precipitation hardening (artificially aging) is typically employed to achieve maximum benefit to the mechanical properties.

Precipitation (Aging) – TE Condition (T5 or T51)

Artifical aging treatment is carried out at temperatures above ambient, typically in the range of 150-200˚C (300-400˚F). This type of heat treatment is done at these relatively low temperatures to eliminate growth. They are also used to stabilize the castings dimensionally (improving mechanical properties somewhat) and to improve machinability. Soak (hold) times can vary between two and 24 hours depending upon the alloy and the cross-sectional thickness of the part. Lower temperatures and longer times promote precipitation and often enhanced mechanical properties.

Solution Treated and Stabilized – TB7 Condition

After solution treatment, castings can be heated into the range of 200-250˚C (400-480˚F) for stabilization and homogenization of the alloying elements. Times and temperature vary with the type of alloy and mass of the component.

Solution Treatment and Precipitation Hardening – TF Condition (T6 or T61)

Solution treatment followed by precipitation (age) hardening produces the highest strength and mechanical properties (tensile and yield strength) while retaining ductility (elongation). Precipitation (age) hardening stabilizes the properties.

Solution Treated and Stabilized – TF7 Condition (T7 or T71)

Castings used for elevated-temperature service may benefit from a solution treatment and stabilization between 200-250˚C (400-480˚F) in order to stabilize mechanical properties when the component is exposed to temperatures close to or in this range. This heat treatment improves mechanical properties to a large degree, stabilizes the castings and usually results in a slightly lower tensile and yield strength but an increased elongation value compared to the T6 series of heat treatments.

Stress Relief and Annealing – TS Condition

Stress relief and annealing can be used to remove stresses in a casting or to soften the component for subsequent shaping or mechanical-working operations. Stress relief is typically performed between 200-250˚C (400-480˚F) while annealing is done around 300-400˚C (575-750˚F).

Alloy Selection

Aluminum casting alloys (Table 2) are numbered in accordance with a three-digit-plus-decimal designation in order to identify major alloying elements (and some alloy combinations).

The digit following the decimal in each alloy number indicates the form of product.

- "0" indicates the chemistry limits applied to an alloy casting.
- "1" indicates the chemistry limits for ingot used to make the alloy casting.
- "2" indicates the chemistry limits for ingot are different (typically tighter).

Generally, the XXX.1 designation indicates the ingot is supplied as a secondary product (e.g., remelted from scrap), whereas the XXX.2 designation suggests the ingot is produced from primary aluminum. Some alloy names are preceded by a letter so as to distinguish between alloys that differ only slightly in percentages of impurities or minor alloying elements (e.g., 356.0, A356.0, B356.0 or F356.0).

In order to choose the alloy, casting process and heat treatment, the engineer begins by understanding the service conditions under which the component part will operate. In order to achieve the desired strength, hardness, corrosion resistance, impact strength or machinability, designers have over 60 casting alloys in use today and up to five different heat treatments they can select (Table 3). Sometimes one mechanical property (e.g., yield strength) dictates alloy choice and/or casting method.

Table 3 Typical Mechanical Properties of Common Aluminum Casting Alloys

Alloy	Casting Process	Temper	Tension			Shear Strength (ksi)	Compressive Yield Strength[1] (ksi)	Brinell Hardness (BHN, 500 kg on 10 mm ball)	Endurance Limit (ksi)
			UTS (ksi)	YS[1] (ksi)	Elongation (%)				
242.0	Sand	F[2]	31	40	<05	-	-	-	-
	Sand	O[3]	27	18	1.0	21	18	70	8
	Sand	T571	32	30	0.5	26	34	85	11
	Sand	T77	30	23	2	24	24	75	10.5
	PM[4]	T571	40	34	1	30	34	105	10.5
	PM	T61	47	42	0.5	35	44	110	9.5
	Sand	T75	31	-	2	-	-	75	-
308.0	PM	F	28	16	2	22	17	70	13
319.0	Sand	F	27	18	2	22	19	79	10
	Sand	T5	30	26	1.5	24	27	80	11
	Sand	T6	36	24	2	29	25	80	11
	PM	F	34	19	2.5	24	19	85	-
	PM	T6	40	27	3	-	27	95	-
356.0	Sand	F	24	18	6.0	-	-	-	-
	Sand	T51	25	20	2	20	21	60	8
	Sand	T6	33	24	3.5	26	25	70	8.5
	Sand	T7	34	30	2	24	31	75	9
	Sand	T71	28	21	3.5	20	22	60	8.5
	PM	F	26	18	5	-	-	-	-
	PM	T51	27	20	2	-	-	-	-
	PM	T6	38	27	5	30	27	80	13
	PM	T7	32	24	6	25	24	70	11
A356.0	Sand	F	23	12	6	-	-	-	-
	Sand	T51	26	18	3	-	-	-	-
	Sand	T6	40	30	6	-	-	75	-
	Sand	T71	30	20	3	-	-	-	-
	PM	F	27	113	8	-	-	-	-
	PM	T51	29	20	5	-	-	-	-
	PM	T6	41	30	12	-	-	80	-
	PM	T7	41	30	10	28	32	90	13
A380.0	Die	F	48	24	3	31	80	21	-
A390.0	PM	F & T5	29	29	< 1.0	-	-	110	-
	PM	T6	45	45	< 1.0	-	60	145	17
	PM	T7	38	38	< 1.0	-	51.7	120	14.5
	Sand	F & T5	26	26	< 1.0	-	-	100	-
	Sand	T6	40	40	< 1.0	-	-	140	13
	Sand	T7	36	36	< 1.0	-	-	115	-
B390.0	Die	F	40.5	35	1	-	-	120	20
	Die	T5	40	38.5	1	-	-	-	-
535.0	Sand	F	40	20	13	27	35.5	70	10
712.0	Sand	F	35	25	5	26	17	75	9

Notes:
1. 0.2% offset
2. F designates “as cast”
3. O designates “stress relief or anneal”
4. PM designates “permanent mold” casting

Table 2. Cast aluminum alloy designations

Series	Alloy Type
1XX.X	99.0% Al (minimum)
2XX.X	Al + Cu
3XX.X	Al + Si-Mg or Si-Cu or Si-Mg-Cu
4XX.X	Al + Si
5XX.X	Al + Mg
6XX.X	Reserved
7XX.X	Al + Zn
8XX.X	Al + Sn
9XX.X	Reserved

Table 4. Typical applications

Alloy	Typical Applications	Remarks
242.0 / A242.0	Cylinder heads, generator housings (aircraft), pistons (aircraft, diesel, motorcycle)	Applications where strength and hardness at high temperatures are desirable.
319.0 / A319.0 / B319.0 / 320	Sand castings: crankcases (internal combustion and diesel Engines), pans (oil), tanks (gasoline and oil) Permanent mold castings: engine components (various), heads (water-cooled cylinder), housings (rear axle)	Applications where moderate strength is required. Mechanical properties are not adversely affected by slight changes in impurity content.
356.0	Sand castings: brackets, blocks (water-cooled cylinder), cases (automotive transmission), fittings, housings (rear axle), pump bodies Permanent mold castings: bodies (valve), blocks (engine), brackets (springs), elbows (fuel tanks), fittings (fuselage, tank car), hardware (marine), machine tool parts, pump parts, rudder-control supports	Applications where excellent casting characteristics are required. In the –T6 condition for marine applications where pressure tightness and/or corrosion resistance are required.
A356.0	Airframes, chassis parts (trucks), machine parts, missile components, structural parts	Applications where higher strength and higher ductility (especially elongation) are desirable.
A380.0 / B380.0	Housings (lawn mowers), heads (air-cooled cylinders), gear cases, radio transmitters	Applications for general-purpose die castings with good mechanical properties.
A390.0 / B390.0	Blocks (internal-combustion engines), brakes, cylinder bodies (compressors), pistons (internal-combustion engines), pumps	Applications where high hardness, good wear resistance and low coefficient of thermal expansion are required.
535.0	Brackets, c-clamps, computing devices, instruments, machined parts	Applications requiring strength, shock resistance, ductility and dimensional stability.
712.0	Castings (marine), farm machinery, machine-tool parts	Applications requiring good strength, shock and corrosion resistance, machinability and dimensional stability.

In Summary

In most cases, aluminum castings are chosen as the most cost-effective solution based on raw material and production cost. However, knowledge of end-use service requirements and how to optimize design efficiency and/or lower production costs in manufacturing (e.g., reproducibility) may change the design. For instance, a casting with sound design may have a size or shape that will promote distortion in heat treating. A wide variety of applications (Table 4, Fig. 2) are possible.

Casting method is also an important consideration. For example, castings required in large quantities favor permanent molds, die casting or automated sand casting (provided the size and design features of the casting and available alloys are suitable). Sand casting often is used to produce parts with hollow cavities and a complex arrangement of ribs or pockets and for parts unsuitable for casting in metal molds. Sand casting usually requires minimum tooling charge, but the unit price of the castings and the finished part can be high. Permanent mold casting requires a higher tooling charge, but the unit price is lower, particularly for longer runs. Die casting usually requires the highest tooling charge but also the lowest piece price on large quantities.

Practical Aspects Related to the Heat Treatment of Titanium and Titanium Alloys

Of the various nonferrous materials in use today, titanium and its alloys have experienced rapid growth for industrial (38%), commercial aerospace (29%) and military aerospace (23%) products. Titanium alloys are used extensively for heavily loaded aerospace components and most recently because of their excellent compatibility with composites. Interest in titanium will continue to grow as military consumption is expected to double in the next 10 years, and commercial aerospace reports that the world fleet will more than double over the next 20 years (Fig. 1). The heat treatment of these alloys is complex and demands an understanding of the end-use application, desired microstructure and process variables. Let's learn more.

Why Use Titanium?

Titanium alloys have excellent corrosion and erosion resistance as well as very high tensile strength and toughness, even at extreme temperatures. Titanium's benefits can be summarized as:

- Excellent strength-to-weight ratio
- Compatibility with carbon/epoxy materials
- Elevated temperature 350°F - 1000°F (175°C - 540°C) service capability
- Superior oxidation and corrosion resistance
- Excellent fatigue and fracture resistance
- Biocompatibility

Applications for titanium alloys include:

- Manned and unmanned aircraft (commercial and military aircraft, rotorcraft)
- Artillery (howitzers)
- Military vehicles (tanks, hovercraft)
- Naval and marine applications (surface vessels, subs)
- Turbines (power generation)
- Medical devices (implants, instruments)
- Chemical processing plants (petrochemical, oil platforms)
- Architecture (sculptures)
- Automotive (motorcycles, performance automobiles)
- Pulp and paper industry (washing and bleaching systems)
- Consumer electronics (batteries, watches)
- Sports equipment (bicycle frames, golf clubs)

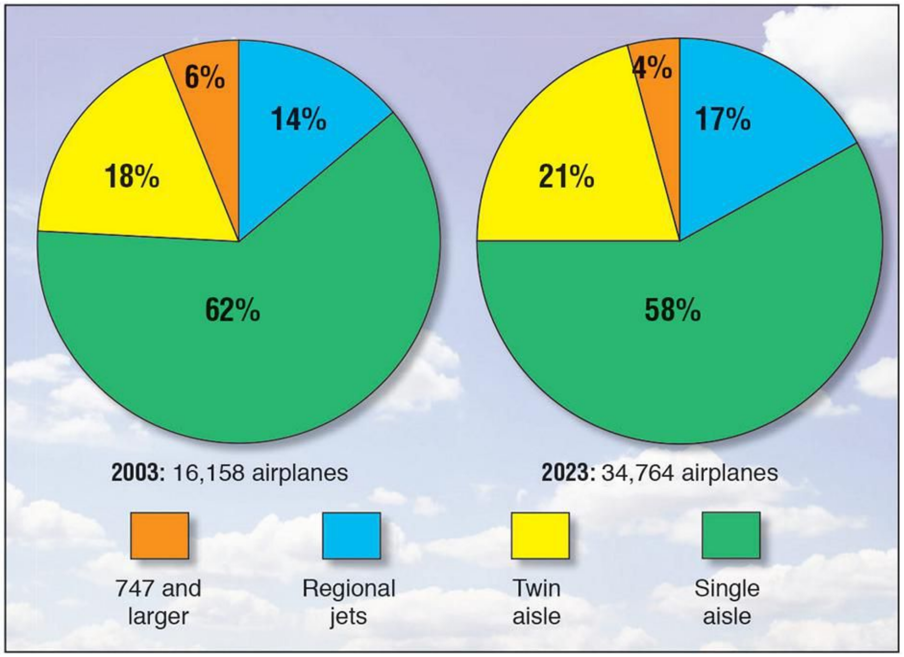

Fig. 1. Projected commercial aerospace growth [1]

Types of Alloys

Titanium alloys are classified in four main groups based on the types and amounts of alloying elements they contain:

- Alpha (α) – cannot be strengthened by heat treatment; low to medium strength, good notch toughness, formable, weldable, good creep resistance (superior to beta alloys) at somewhat elevated temperatures.
- Near alpha – medium strength and good creep resistance.
- Alpha-beta (α-β) – strengthened by heat treatment; medium to high strength, high formability, good creep resistance (but less than most alpha alloys), alloys with beta content less than 20% are weldable. The most familiar alloy in this category is Ti-6Al-4V.
- Beta (β) – strengthened by heat treatment; high strength, fair creep resistance, prone to a ductile-to-brittle transition temperature.
- Some alloying elements (Al, Ga, Ge, C, O, N) raise the alpha-to-beta transition temperature (alpha stabilizers) while others (Mo, V, Ta, Nb, Mn, Fe, Cr, Co, Ni, Cu, Si) lower the beta transition temperature (beta stabilizers).

Types of Heat Treatments

While pure titanium is soft and relatively weak, heat treating can significantly enhance its properties. Titanium and titanium alloys are heat treated in order to:

- Reduce residual stresses from fabrication (stress relieving)
- Produce an optimum combination of ductility, machinability, and dimensional and structural stability (annealing)
- Increase strength (solution treating and aging)

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- Optimize specific properties such as fracture toughness, fatigue strength, and high-temperature creep strength
- Create specific material conditions

Standard heat treatments include:

- Annealing – increases fracture toughness and ductility (at room temperature) and improves dimensional stability and creep resistance. Annealing may be necessary following severe cold work and to enhance fabrication and machining.
- Homogenizing – improves chemical homogeneity in castings.
- Solution treating and age hardening (aging) – a process of heating into the beta or high in the alpha-beta phase region, quenching and then reheating again to the alpha-beta region. A wide range of strength levels is possible. Fatigue strength increases while ductility, fracture toughness and creep resistance are enhanced.
- Stress relief – used to reduce residual stresses during fabrication or following severe forming or welding to avoid cracking or distortion and to improve fatigue resistance. Strength and ductility will not be adversely affected and cooling rate is not critical.

Fig. 2. Load of titanium sheet for vacuum age hardening (Photograph Courtesy of Solar Atmospheres of Western Pennsylvania)

- Tempering – When titanium is quenched from an elevated temperature, reheated to a temperature below the beta-transus, held for a length of time and again quenched, it is said to have been tempered. Three variables exist in tempering: the phases present, the time held and the tempering temperature.

Fig. 3. Load of titanium ingots for vacuum degassing (Photograph Courtesy of Solar Atmospheres of Western Pennsylvania)

Custom heat treatments include:

- Beta vacuum annealing and vacuum aging (Fig. 2) – improves fatigue and yield strength as well as elongation in alloys such as Ti-5553 (Ti-5Al-5V-5Mo-3Cr).
- Brazing – induction, resistance and furnace brazing in an argon atmosphere or in vacuum. Cleanliness is important to avoid contamination.
- Creep forming – takes advantage of the fact that titanium moves and takes a set well at temperature.
- Degassing – involves removing of entrapped gases such as hydrogen (to under 50 ppm) to avoid embrittlement (Fig. 3).
- Diffusion bonding – powder metallurgy where individual particles fuse from intimate surface contact.
- Hydriding – the deliberate addition of hydrogen to embrittle the material.
- Isothermal transformation – involves quenching an alloy from the all-beta region into the alpha-beta field, holding and then continuing to quench to room temperature. This causes precipitation of the alpha phase from the beta.
- Sintering – typically involving hot isostatic pressing and laser sintering of powder particles to form near-net-shape components.

Practical Considerations – What's Important?

Heat-treat-furnace capacity is an important consideration since many titanium parts are volume-limited rather than weight-limited. Load support is a critical issue in many applications to prevent creep or other dimensional changes, especially for intricate or complex part geometry.

Temperature measurement and control must be exact – usually ±10°F (5.5°C) or better throughout the entire working zone of the furnace. Work thermocouples are required because part temperature must be known. Caution: when heating parts over 1730°F (943°C), titanium cannot be in contact with a nickel alloy or stainless steels since eutectic melting will occur.

Vacuum pumping systems must be capable of reaching high vacuum levels, 1 x 10^{-5} torr or lower before starting to heat. This vacuum level must be maintained while heating (requiring very slow ramp rates) and at temperature. Diffusion pumping systems must be properly maintained for maximum efficiency and to avoid backstreaming.

Since titanium is a strong "getter" material, vacuum-furnace interiors must be pristine. Ideally, all-metal hot zones and dedicated furnaces are desired, but graphite-lined furnaces, also used for other processes, are typical throughout the industry as a practical necessity. Thus fixtures and furnaces must be "baked out" (cleaned) before use typically at 2400°F (1315°C).

Finally, the reason why vacuum is used to process titanium is:

- Superior buy-to-fly ratio (no wasted material)
- Minimization of alpha case – avoidance of an oxygen contamination layer that is very brittle and must be removed by mechanical or chemical means (allows closer near-net-shape parts)

References

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3. Wood, J. R. and P. A. Russo, "Heat Treatment of Titanium Alloys," Industrial Heating, December 2004.

Understanding Copper & Copper Alloy Heat Treatment

Different combinations of properties can be produced by varying the heat treatment of copper and its alloys—influencing strength, hardness, ductility, conductivity, impact resistance, and inelasticity. Let's learn more.

Types of Heat Treatment

Common heat treatments applied to copper and its alloys are:

- Homogenizing to reduce chemical segregation and coring of cast structures, and create a more uniform structure in hot worked materials
- Annealing to soften work hardened (strain-hardened) materials (Fig.1)
- Stress relief to stabilize properties and improve strength and dimensions particularly for cold worked parts, and to reduce residual stress
- Solution treating and precipitation (age) hardening to provide increased strength by precipitation of constituents from solid solution
- Quenching hardening by a martensitic-like transformation followed by tempering

Copper and copper alloys are supplied in the solution treated condition, in the solution treated and cold worked condition, and in the age-hardened condition. Their heat treatment falls into two general categories: hardening either by low temperature precipitation treatments or hardening by quenching from elevated temperature.

Hardening Mechanisms—Low Temperatures

Precipitation Hardening

Solution treating followed by precipitation hardening is applied to strengthen special types of copper alloys above the levels ordinarily obtained by cold working. All precipitation hardened copper alloys have similar metallurgical characteristics: they can be solution treated to a soft condition by high temperature heating and quenching and then precipitation hardened at a moderate temperature for a relatively short time (around 3 hours) since hardness reaches a peak then decreases with time. Copper alloys cannot be hardened by natural aging as aluminum alloys can.

The main advantages of these alloys are:

- Ease of fabrication in the soft solution annealed

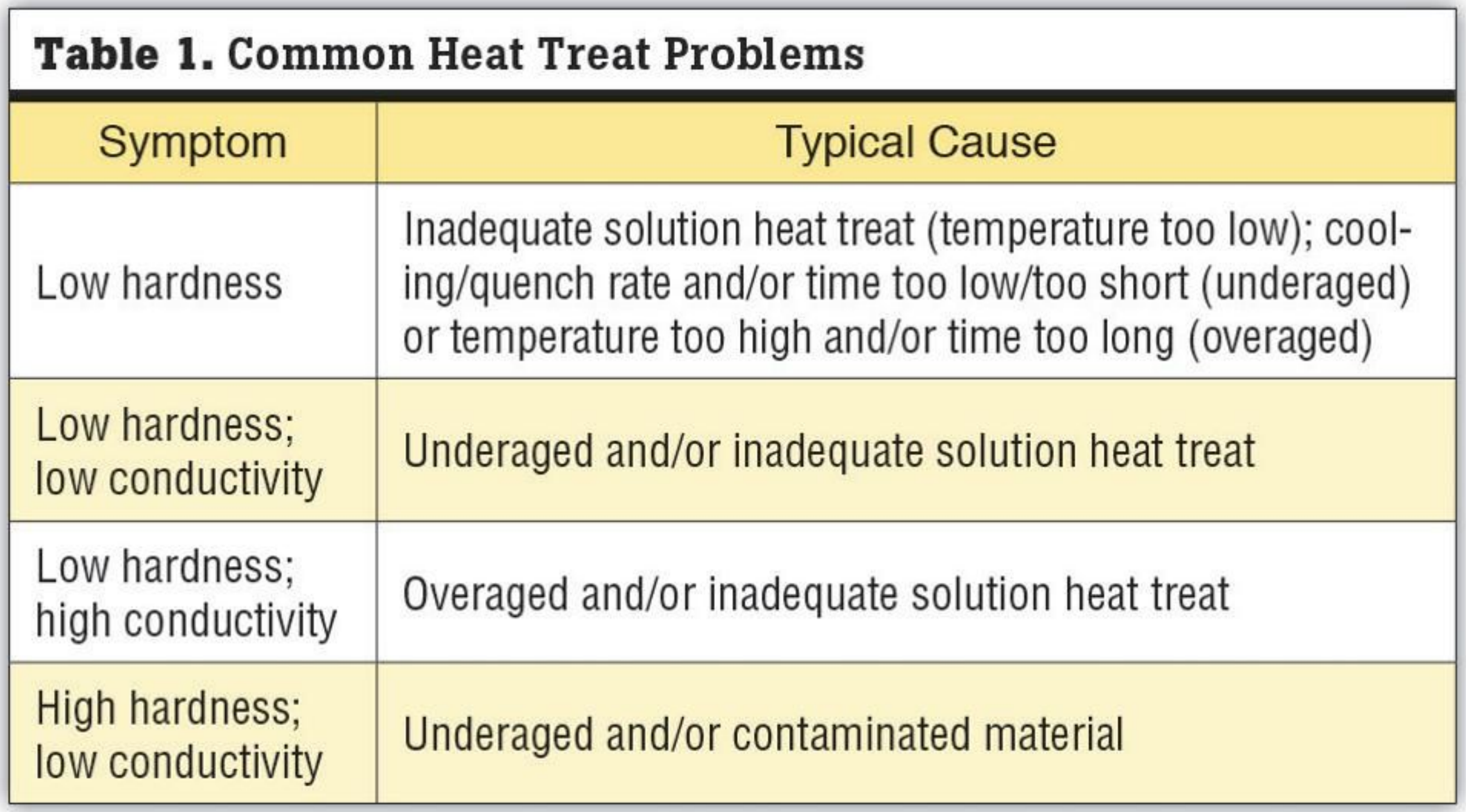

Table 1. Common Heat Treat Problems

Symptom	Typical Cause
Low hardness	Inadequate solution heat treat (temperature too low); cooling/quench rate and/or time too low/too short (underaged) or temperature too high and/or time too long (overaged)
Low hardness; low conductivity	Underaged and/or inadequate solution heat treat
Low hardness; high conductivity	Overaged and/or inadequate solution heat treat
High hardness; low conductivity	Underaged and/or contaminated material

condition
- Precipitation hardening (usually) in air
- Wide variety of mechanical properties achievable;

Example of precipitation hardening copper alloys include beryllium copper (some of which contain nickel, cobalt or chromium), copper chromium, copper zirconium, copper-nickel-silicon and copper-nickel-phosphorous alloys.

Spinodal Hardening

What differentiates these alloys is that their hardening mechanism does not result in

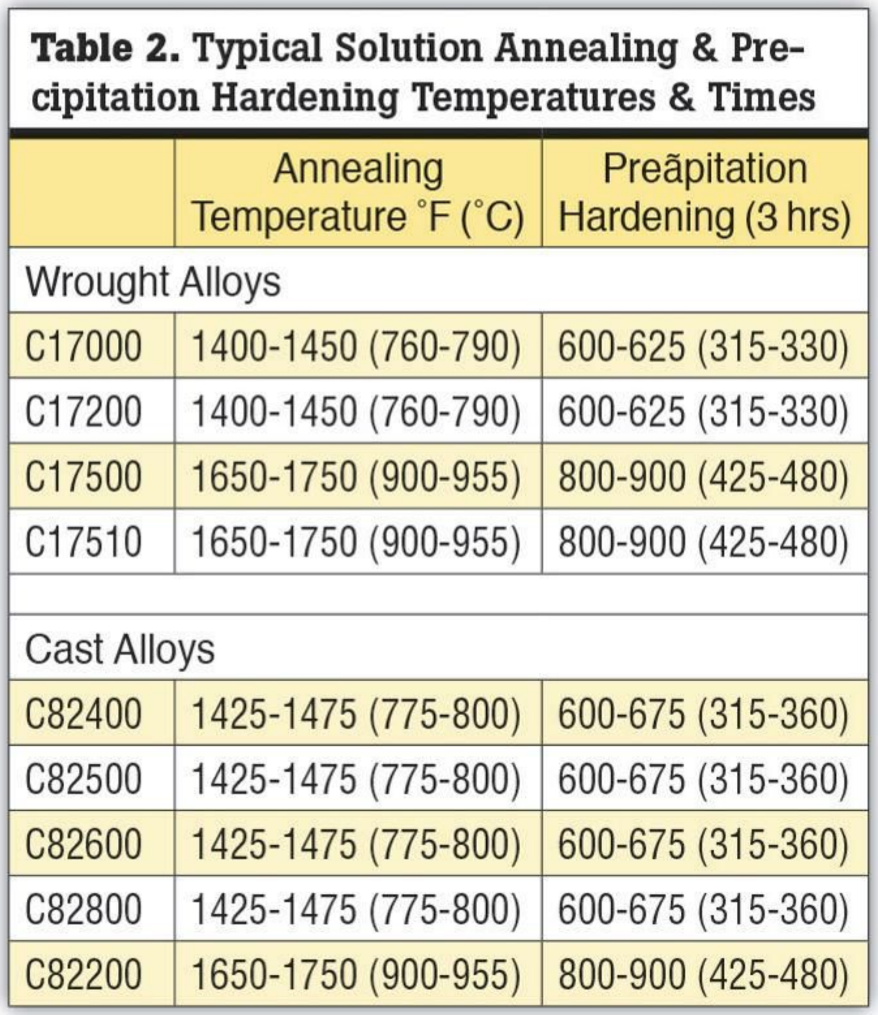

Table 2. Typical Solution Annealing & Precipitation Hardening Temperatures & Times

	Annealing Temperature ˚F (˚C)	Preãpitation Hardening (3 hrs)
Wrought Alloys		
C17000	1400-1450 (760-790)	600-625 (315-330)
C17200	1400-1450 (760-790)	600-625 (315-330)
C17500	1650-1750 (900-955)	800-900 (425-480)
C17510	1650-1750 (900-955)	800-900 (425-480)
Cast Alloys		
C82400	1425-1475 (775-800)	600-675 (315-360)
C82500	1425-1475 (775-800)	600-675 (315-360)
C82600	1425-1475 (775-800)	600-675 (315-360)
C82800	1425-1475 (775-800)	600-675 (315-360)
C82200	1650-1750 (900-955)	800-900 (425-480)

precipitation but results from a miscibility difference in the solid solution resulting in ultra fine chemical segregation of the alpha crystalline phase. Thus spinodal alloys exhibit excellent dimensional stability after hardening. Common spinodal hardening alloys include copper-nickel alloys with additions of chromium or tin.

Order Hardening

When short range ordering of dissolved alloying elements occurs within the copper matrix, the alloys are called order hardening. These alloys nearly saturated with an alloying element dissolved in the alpha phase after significant cold working under respond by ordering when annealed at relatively low temperatures. Order hardening alloys typically exhibit improved stress relaxation, and the process is usually performed after the final fabrication step. Examples of order hardening alloys include some silicon and aluminum bronzes with silicon and copper-zinc-aluminum-cobalt alloys.

Hardening Mechanisms—High Temperatures

Transformation Hardening

Transformation hardening mechanisms resulting from quenching at high temperatures induces internal changes producing harder, stronger phases. Tempering such structures improves toughness and reduces hardness in a similar manner to that of alloy steels. Quench hardening alloys include aluminum bronzes, nickel-aluminum bronzes, and a few copper zinc alloys.

Fig. 1 Vacuum annealing of copper heat sink blanks

Beryllium Copper Alloys

Heat treating is the key to the versatility in the beryllium copper alloy system, which varies considerably with form, alloy, and desired temper and is applicable to all beryllium copper cast and wrought alloys. These alloys are used when good electrical and thermal conductivities and high strength is required and are classified either as high conductivity or high strength alloys.

Heat treating of beryllium copper is a two-step process, which consists of solution annealing and age hardening (Table 2). Solution annealing is often done by the mill and involves a high temperature soak at 1450˚F (790˚C) for the high strength alloys and 1650˚F (900˚C) for the high conductivity alloys. Excess time or temperatures may cause undesirable grain growth. Solution annealing is followed by water quenching.

The age or precipitation hardening results from the precipitation of a beryllium containing phase from a supersaturated solid solution of mostly pure copper. The precipitation occurs during the slow cooling of the alloys because the solubility of beryllium in alpha copper decreases with decreasing temperature. Typically the alloys are rapidly cooled from the annealing treatment, so the beryllium remains in solid solution with the copper. Then the alloy is given a precipitation or age hardening treatment for an hour or more at a temperature between 400˚F (200˚C) and 860˚F (460˚C). Upon tempering, the beryllium containing phases, called beryllides, precipitate out of solution.

Common aging conditions include:

- Underaging—producing strength and hardness levels less than optimally attainable by using lower temperatures, less time, or a combination of the two, but can tailor a specific property or

combination of properties. Underaging is used when better formability is needed rather than maximum strength.
- Peak Aging—producing the highest strength, hardness, and the highest electrical and thermal conductivity by using the optimal temperature and time combination.
- Overaging—producing strength and hardness properties less than optimally attainable by using a higher temperature, longer time at temperature, or both, but in different combinations. Overaging is used where greater toughness and/or impact strength is more important than absolute strength.

Precipitation hardening is cumulative. Multiple thermal treatments may be applied except for overaged material whose properties are beyond peak.

Unique Application—Shape Memory Alloys

A shape memory alloy (SMA) has the ability to return to some previously defined shape or size. These materials can be plastically deformed at relatively low temperature and upon exposure to some higher temperature, return to their original shape. Copper-based SMA's with commercial interest include CuZnAl and CuAlNi alloys.

Copper SMA's are metastable and require solution heat treatment in the parent (beta) phase region and subsequent controlled cooling to retain beta phase for shape memory effects. Prolonged solution heat treatment results in undesirable zinc evaporation and grain growth. Water quenching is widely used, but air cooling also may be sufficient for high aluminum content alloys. Post quench aging is needed to establish stable transformation temperatures. The thermal stability of these SMA's requires avoidance of prolonged exposure above 300˚F (150˚C) for CuZnAl and 390˚F (200˚C) for CuAlNi alloys.

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Understanding Magnesium Heat Treatment

The world is becoming lighter, faster and more energy efficient. For all these reasons, and more, the spotlight is turning toward the use of magnesium and magnesium alloys, especially in the aerospace and automotive industry. We need to better understand these alloys and how to heat treat them. Let's learn more.

Alloying

Magnesium is rarely used as an engineered material in its unalloyed form. Its hexagonal close-packed (HCP) lattice structure promotes alloying with many elements including aluminum, zinc, lithium, cerium, silver, zirconium and thorium. Magnesium alloys are available as cast and wrought products.

Techniques for producing wrought alloys include rolling (sheet, plate), extrusion and forging. They are designed with properties such as low-to-medium-to-high strength, weldability, corrosion and creep resistance, and ultra lightweight. Wrought magnesium alloys that can be strengthened by heat treatment are grouped according to composition with examples in parentheses.

- Aluminum-manganese (LA141)
- Aluminum-manganese-zinc (AZ31, AZ61, AZ80)
- Manganese (M1)
- Manganese-zinc (ZM21)
- Thorium-zirconium (HK31)
- Thorium-manganese (HM21)
- Zinc-zirconium (ZK31, ZK61)
- Zinc-zirconium-thorium (HZ11)

Various casting techniques (sand cast, chill cast) and temper conditions (e.g. T4, T5, T6) produce castings of various compositions having characteristics such as good room-temperature strength and ductility, good creep resistance and proof stress, and they are easily weldable. Magnesium-alloy castings that benefit from heat treatment are grouped based on the alloying elements they contain:

- Aluminum-manganese
- Aluminum-zinc
- Aluminum-zinc-manganese (AZ63, AZ81, AZ91, AZ92)
- Rare earth metal-zinc-zirconium (ZE41, ZE 63, EZ33)
- Rare earth metal-silver-zirconium, with or without

Fig. 1. Typical heat-treat processing oven (Photograph courtesy of Wisconsin Oven Corporation)

Fig. 2. Magnesium casting after solution treating

thorium (OE22, EQ 21, QH21)
- Rare earth metal-yttrium-zirconium (WE43, WE54)
- Thorium-zirconium, with or without zinc (HK31, HZ32)
- Zinc-zirconium (ZK51, ZK61)

Types of Heat Treatment

The types of heat treatments applied to magnesium and its alloys are:

- Annealing of wrought magnesium alloys is done to negate the effects of strain hardening or tempering.
- Solution heat treatment yields proper mechanical properties such as increased strength and ductility.
- Stress relief, as applied to castings, helps avoid warping and distortion in subsequent heat treatments and reduces the risk of stress-corrosion cracking (SCC) in welded components.
- Stress relief of wrought alloys, though not common, is used to counteract stress induced by cold and hot working, shaping, forming, straightening and welding.

Specifications such as ASTM B661 (Standard Practice for the Heat Treatment of Magnesium Alloys) and AMS 2768 (Heat Treatment of Magnesium Alloy Castings) are often referenced for specific treatment details.

Solution Heat Treating

Solution heat treatment of magnesium alloys results in high tensile strength and maximum ductility. Magnesium alloys tend to reach temperature quickly due in part to their high thermal conductivity and low specific heat. In normal practice, the soak time begins when the furnace reaches set point.

The time at temperature is a function of the section thickness of the material and may vary from several minutes to many hours. A good "rule of thumb" is to double the time at solution heat-treatment temperature for thick-sectioned castings. For example, AZ63A castings normally require 12 hours at 725°F (385°C) but require 24+ hours when section thickness exceeds 2 inches (50 mm). Similarly, the suggested solution treating time for AZ92A castings is about 6 hours at 760°F (405°C) followed by 2 hours at around 660°F (350°C) and 10 hours again at 760°F (405°C) to prevent excessive grain growth. But for castings with sections more than 2 inches (50 mm) thick, it is recommended that the last soak be extended from 10 to 20 hours. Checking the part microstructure is by far the best way to determine whether or not additional solution treating time is required.

Normal oven requirements (Fig. 1) for temperature uniformity are ±10°F (±5.5°C), but certain alloys require tighter tolerances. The time required to heat a load to the treatment temperature also increases with section thickness and loading arrangement. Thus, the total cycle time must take into consideration these factors.

Certain alloys like magnesium-aluminum-zinc require precautions such as loading into the oven, which is at a preheat temperature typically around 500°F (260°C), and then slowly ramping to solution-treating temperature to avoid fusion of eutectic compounds and formation of voids. The time required to bring the load from 500°F (260°C) to the solution-treating temperature is determined by the size of the load and by the composition, size, weight and section thickness of the parts.

Other special cases apply. Alloys containing large percentages of thorium, rare-earth metals (yttrium, hafnium, etc.) and zirconium, used in the T5 or T6 temper tend to shrink rather than grow at solution heat-treatment temperatures. Still, other castings of aluminum-manganese or aluminum-manganese-zinc exhibit permanent growth if subjected to long exposure to temperature.

Protective Atmospheres

Above 750°F (400°C), protective atmospheres are used in solution heat treatment so as to prevent surface oxidation and as a safety measure due to the unpredictable combustive nature of magnesium, especially if the processing temperature is exceeded. Ovens and furnaces used for solution heat treatment must therefore have gas-tight construction.

Sulfur dioxide (SO_2) and carbon dioxide (CO_2) are the most common gases used. Inert gas such as argon or nitrogen may be used if one can ensure that oxygen is not allowed to enter the furnace atmosphere. Sulfur dioxide has a pungent odor (rotten-egg smell) and can be corrosive to certain materials (e.g. nylon). A typical concentration of 0.7% (0.5% minimum) is used to prevent the material from self-igniting up to a temperature of 1050°F (565°C), provided melting has not occurred. Carbon dioxide in a concentration of 3% will prevent the pyrophoric reaction to 950°F (510°C), and a carbon dioxide concentration of 5% will provide protection to about 1000°F (540°C).

Safety

In large sections, magnesium's high thermal conductivity makes it difficult to ignite and under normal heat-treat conditions prevents it from burning. Magnesium will not burn until it reaches its melting point of 1204°F (651°C), but when it does, the result is extremely high temperatures and, quite literally, blinding white light. Magnesium in the form of fine chips or dust, however, is easily ignited. Should a fire occur, it can be extinguished with special powders such as soapstone or graphite. Water or any standard liquid or foam fire extinguishers cause magnesium to burn more rapidly and may cause an explosion.

Quenching

Unlike aluminum alloys, most magnesium alloys (Fig. 2) are quenched in air following solution heat treatment. For dense loads or heavy castings, fans are used to accelerate the cooling process.

Aging (Age Hardening)

The response to age hardening by magnesium alloys is significantly less than with aluminum alloys. When aging, parts should be loaded into the furnace at the treatment temperature, held for at least one hour per inch of cross-sectional thickness and then cooled in still air.

Stress Relief (Wrought Alloys or Castings)

Magnesium castings do not normally contain a high level of residual stresses and do not generally require stress relief. However, precision machining of castings to close dimensional limits and the low modulus of elasticity of magnesium alloys means that comparatively low stresses can produce appreciable deformation. Stress relief is also used to prevent stress-corrosion cracking in, for example, welded magnesium-aluminum casting alloys. Residual stresses may arise from contraction due to mold restraint during solidification, from no uniform cooling after heat treatment or from quenching. Machining operations can also produce residual stress and require intermediate stress relieving prior to final machining.

Annealing

Annealing can be used for both heat-treatable and non-heat-treatable alloys to increase ductility with a slight reduction in strength. A typical annealing cycle for wrought magnesium alloys is to heat them to 550-850°F (290-455°C) for at least one hour. Since most forming operations are done at elevated temperature, most wrought material is already fully annealed.

Other Thermal Treatments

Additional thermal treatments are seldom necessary. If the heat-treated microstructure is not optimum, or if a casting has not been properly cooled after solution treating, for example, a reheat process is necessary.

Preparation of Samples for Analysis of Microstructures

If you are going to look at the microstructure of these alloys, a few tips are in order. Pure magnesium is difficult to polish because it is very soft and is attacked by many dilute organic acids, slowly attacked by water (many magnesium alloys are rapidly attacked) and mechanical twinning may be produced during cutting and (abrasive) grinding. Fine magnesium dust is also a fire hazard.

Macroetchants reveal flow lines, grain structure and internal defects. Other etchants (Ref. 3) are used to reveal microstructure, grain contrast, grain boundaries and compositional variation within grains.

Chemical and electrolytic polishing and attack etching are methods used to prepare magnesium alloys. Attack etching (etch polishing or etch attack) involves adding a dilute chemical etchant to the polishing medium to increase polishing rate and change to dissolution-predominate polishing mechanisms. It is also performed when removal of passivating films is necessar.

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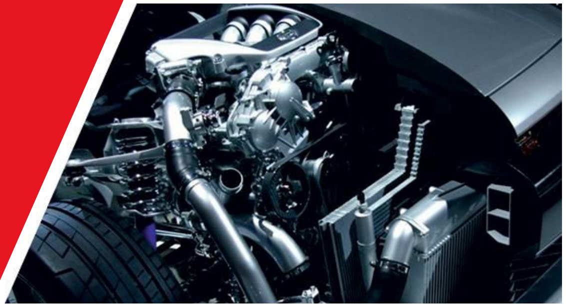
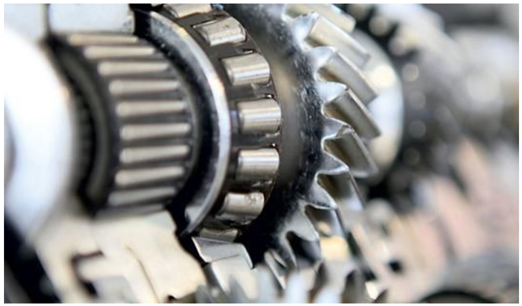
UniCase Master





Considerations in Heat Treatment Part One: Furnace Atmospheres

A critical consideration in heat treatment is the type, consistency and control of the furnace atmosphere. The purpose of a furnace atmosphere varies with the desired end result of the heat-treating process. The atmospheres used in the heat-treat industry have one of two common purposes:

- To protect the components being processed from harmful chemical reactions that could occur on their surfaces (such as oxidation or carburization) – that is, to be passive (chemically inert) to the metal surface.
- To allow the surface of the parts to be changed (by adding carbon, nitrogen or both) – that is, to be reactive (chemically active) to the metal surface.

Types of Furnace Atmospheres

Many types of furnace atmospheres are available for use in heat treating (Table 1). In most instances, hardening and case hardening operations use endothermic gas or nitrogen/methanol systems. Most tempering operations are performed in air atmosphere as long as the presence of a tightly adherent oxide surface ("skin") will not affect the part's performance.

Table 1. Common types of furnace atmospheres

Type	Symbol	Remarks
Air		Typically used in tempering operations
Argon	Ar	An inert gas
Carbon Dioxide	CO_2	A common constituent in generated atmospheres
Carbon Monoxide	CO	A common constituent in generated atmospheres
Custom Blends		Examples include alcohols and combinations of nitrogen and hydrocarbon gases
Generated atmospheres		Endothermic, exothermic, dissociated ammonia
Helium	He	An inert gas
Hydrocarbon Gases		Typically used as additions or enriching gases to furnace atmospheres. Common types include methane (CH_4), propane (C_3H_8) and butane (C_4H_{10}).
Hydrogen	H_2	A constituent of many furnace atmospheres used to aid in heat transfer and react with oxygen present.
Nitrogen	N_2	A blanketing gas that is not truly inert
Oxygen	O_2	Oxidizing to a hot steel surface
Products of combustion		Produced from a mixture of a hydrocarbon fuel gas and air, the atmosphere typically consists of high amounts of carbon dioxide and water vapor.
Steam	H_2O	Water vapor is most often used to impart a protective oxide layer.
Sulfur Dioxide	SO_2	Used in the heat treatment of magnesium alloys.
Synthetic atmospheres		Nitrogen and methanol (methyl alcohol)
Vacuum		The absence of an atmosphere

Otherwise, an inert gas (vacuum) is selected.

Endothermic Gas Atmospheres

Endothermic gas generators are common equipment

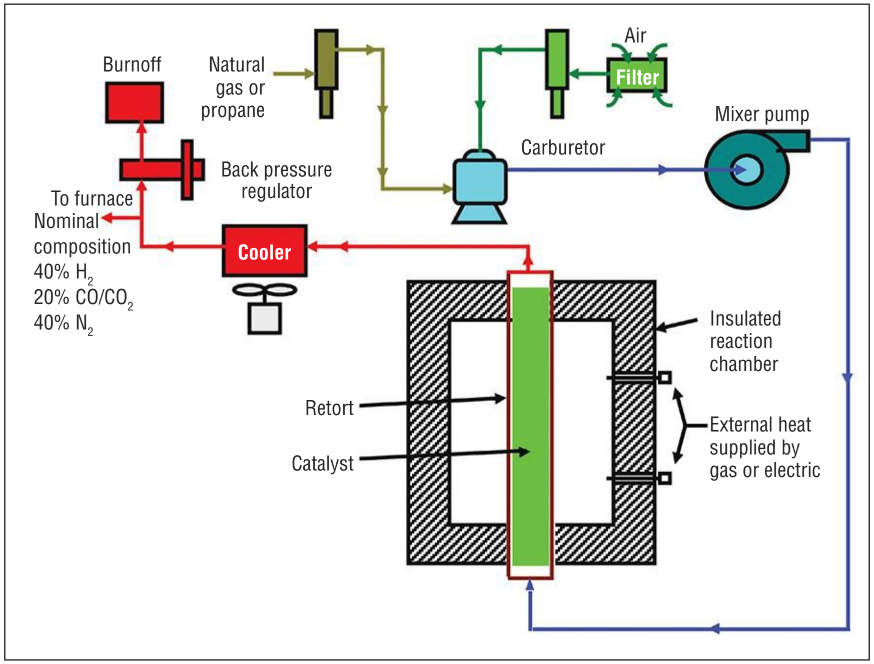


Fig. 1. Endothermic gas generator schematic piping arrangement

in the heat-treat shop. The main components of an endothermic generator (Fig. 1) consist of:

- Heated reaction retort with catalyst
- Air-gas proportioning control components
- Pump to pass the air-gas mixture through the retort
- Cooler to "freeze" the reaction and prevent soot formation

Endothermic gas – also called endo or Rx™ gas – is produced when a mixture of air and fuel is introduced into an externally heated retort at such a low air-to-gas ratio that it will normally not burn. The retort contains an active catalyst, which is needed for cracking the mixture. Leaving the retort, the gas is cooled rapidly to avoid carbon reformation (in the form of soot) before it is sent into the furnace. The endothermic-gas composition (Tables 2 & 3), by volume, varies depending on the type of hydrocarbon-gas feed stock.

Endothermic gas is used for neutral hardening and as a carrier gas for gas carburizing and carbonitriding. It is generally produced so that its composition is chemically inert to the surface of the steel and can be made chemically active by the addition of enrichment (hydrocarbon) gas, which is usually done at the furnace.

Nitrogen/Methanol or Nitrogen/Hydrogen Atmospheres

An endothermic equivalent gas atmosphere can be obtained by cracking liquid methanol (methyl alcohol) and combining it with nitrogen (Eq. 1), using a blend of 40% nitrogen and 60% methanol (dissociated).

$$(1) \qquad CH_3OH + N_2 \rightarrow CO + H_2 + N_2$$

This chemical reaction typically takes place inside the furnace as the liquid methanol and gaseous

Table 2. Compositional ranges for endothermic gas

Gas constituent	Percentage (based on natural gas)	Percentage (based on propane)
N_2	40.9 %	40.9%
CO	19.6 %	23.3%
CO_2	0.4 %	0.1%
H_2	38.9 %	35.5%
CH_4	0.2 %	0.2%
Dew point	+20/+50°F	-10/-15°F
(Air/Gas) Ratio	2.6:1	7.8:1

Table 3. Nitrogen/methanol atmosphere field data[1]

Flow data[2]	% N_2	% H_2	% CO_2	% CO	% CH_4	Dew Point, °F (°C)
Nitrogen/methanol with natural gas and/or air enrichment	37-46	38-42	0.4–1.1	11.8–14.1	6-11	+30 to +65 (0 to +17)

Notes: A 2,000 lb/hour (900 kg/hour), 48-inch-wide (1.2-m) electrically heated mesh-belt conveyor furnace operating at carbon potential settings between 0.20-0.45%C. Approximate gas flows: 600-800 cfh (17-23 m^3/hour) nitrogen, 190 cfh methanol (3 l/hour), 200-300 cfh natural gas (6-9 m^3/hour), 40-50 cfh (1.0-1.5 m^3/hour) air.

Table 4. Comparison of synthetic furnace atmospheres

Atmosphere	Type	%H_2	%N_2	%CO	Dew Point, °F (°C)
Hydrogen	Pure	100	0	0	-95 to -120 (-70 to -85)
Dissociated Ammonia (DA)	Generated	75	25	0	- 40 to -50 (-40 to -75)
Nitrogen-DA	Blended	90	10	0	> -50
Endothermic	Generated	40	40	20	+40 to -10 (3 to -23)
Nitrogen-Endo	Blended	12	82	6	< 0
Nitrogen-Hydrogen	Blended	3–75	97–25	0	-60 (-51)

nitrogen are metered in through a special injector called a sparger, which atomizes the liquid and sprays it into the chamber, usually onto a hot target such as the furnace fan. The equivalent of 4 KW of heat is required per gallon to crack the methanol. One gallon per hour (3,785 ml/hour) of methanol liquid produces 241 cfh (6.8 m^3/hour) of dissociated methanol.

For some neutral-hardening applications, a gas is produced with a lower carbon monoxide value than an endothermic equivalent atmosphere (Table 3).

The most common problems with nitrogen/methanol systems have to do with the failure to properly atomize. Large droplets do not properly decompose, resulting in difficulties in furnace control. Also, methanol is corrosive to nickel alloys used for the internal furnace components (e.g., fans, radiant tubes, belts, etc.).

Other types of blended atmospheres (Table 4) produced with nitrogen and/or hydrogen are less common but have been used in some applications. The resultant atmosphere may not contain carbon dioxide (CO_2) or carbon monoxide (CO).

Gas Reactions

The gas reactions involved can be classified into four general categories:

- Oxidation reactions
- Reduction reactions
- Carburizing reactions
- Decarburizing reactions

Reactions Involving Oxygen

In the presence of oxygen, steel will oxidize. This tendency increases in severity as the temperature is raised. In addition, oxygen will decarburize steel. If steel is to be kept bright during heat treatment and free of decarburization, free oxygen (O_2) in the furnace atmosphere must be eliminated.

Table 5. Volume changes required for safe purging of furnaces

Number of volume changes	Percentage (%) of air remaining
0.1	90.48
0.2	81.87
0.3	74.08
0.5	60.65
1.0	36.79
2.0	13.53
3.0	4.98
4.0	1.83
5.0	0.67

Reactions Involving Water Vapor

The water-gas reaction (Eq. 2) is the most important furnace-atmosphere chemical reaction. This equation involves the major constituents of the gas atmosphere as it controls the reactants formed on each side of the equation. The equal sign indicates chemical equilibrium – that is, the reaction can go either way, to form CO and water vapor (H_2O) or to form CO_2 and hydrogen (H_2) depending on the relative percentages of each in the furnace atmosphere.

(2) $CO_2 + H_2 = CO + H_2O$
(Water-Gas Reaction)

Water vapor and CO_2 both appear in this equation, and we can use this fact to control the carbon potential of a furnace atmosphere. In simplest terms, dew-point analyzers look at the H_2O/H_2 ratio in the water-gas reaction. Infrared analyzers and oxygen-probe devices look at the CO/CO_2 ratio in the water-gas reaction.

Water vapor is a strongly decarburizing gas. Any constituent such as CO_2 will have a tendency to form water vapor, therefore, CO_2 must also be closely controlled. In addition, to prevent decarburization by water vapor, the CO and H_2 must be present in amounts to satisfy the equilibrium condition at each temperature.

Water vapor and CO_2 oxidize and decarburize steel. Hydrogen is formed when water vapor oxidizes iron. Therefore, to prevent oxidation and to keep iron bright, a definite excess of H_2 over H_2O vapor is required for each temperature.

Reactions Involving Carbon Dioxide

CO_2 is one of the reaction products when a hydrocarbon fuel is burned in air. CO_2 oxidizes iron at elevated temperatures. To prevent oxidation, it is necessary to have an excess of CO. Therefore, to prevent oxidation, CO is a desirable constituent. CO_2 is not only oxidizing to steel but it is extremely decarburizing. To prevent decarburization, CO_2 must be controlled very closely. The actual amount depends upon the CO content, temperature and the carbon content of the steel.

Reactions Involving Carbon Monoxide

CO is a strong carburizing agent. The reversible reaction of CO to form carbon (C) and CO_2 is of particular interest in a furnace atmosphere. CO has a high carbon potential and becomes increasingly more stable at elevated temperatures. It is only at lower temperatures 480 - 730ºC (900 - 1350ºF) that CO will supply carbon (Eq. 3) in the form of soot in the so-called carbon-reversal reaction. Soot causes most of the maintenance-related issues with gas generators and heat-treating furnaces.

$$2CO = C + CO_2 \quad (3)$$

Reactions Involving Nitrogen

Below about 1010ºC (1850ºF), molecular nitrogen (N_2) will not react with the surface of steel or stainless steel. However, atomic nitrogen (N), which does not normally occur in a furnace atmosphere unless it is purposely introduced by the addition of ammonia (NH_3), will react by being absorbed into the steel surface.

Reactions Involving Hydrocarbons

Methane and other hydrocarbons (propane and/or butane) are carburizing agents. At elevated furnace temperatures, methane (CH_4) breaks down into carbon (C) and H_2. The higher the furnace temperature, the greater the tendency for CH_4 to break down. Because of this tendency, CH_4 and other hydrocarbon gases are introduced into the furnace to help change the atmosphere from neutral to one with a high carbon potential (the driving force of carbon into the surface of the steel).

Atmosphere Volume Requirements

During operation, the volume of protective atmosphere required for safe use in a particular heat-treating furnace and the ability to properly control that atmosphere depends to a great extent on the:

- Type and size of furnace
- Presence or absence of doors and/or curtains
- Environment (especially drafts)
- Size, loading, orientation and nature of the work

being processed
- Metallurgical process involved

In all cases, the manufacturer's recommendations should be followed for gas introduction, purging and removal since the original equipment manufacturer has taken these factors into account during the design of the equipment.

National Fire Protection Association (NFPA) Standard 86, "Standard for Industrial Furnaces Using a Special Processing Atmosphere," applies to all furnaces, and the procedures listed within this standard must be followed.

A "rule of thumb" to remember is that to purge air out of a furnace prior to introduction of a combustible furnace atmosphere requires a minimum of five volume changes of the chamber (Table 5). This is to ensure that the oxygen content of the chamber is below 1% prior to the introduction of the atmosphere.

Important Cautions

In order to interpret furnace-atmosphere data correctly it is important to understand the whole picture, including knowing how the data was collected as well as understanding the exact furnace operating conditions at the time the data was collected (e.g., zone temperatures and gas flows, furnace pressure, exhauster settings, fan rotation and speed, etc.). Part two of this article will discuss atmosphere-control techniques.

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PROTON' PEM

Exothermic Gas Generators: Forgotten Technology?

Do you think there is nothing new in the field of gas generators? Think again. An old friend, the exothermic gas generator, is making a comeback by combining smart controls and innovative design features. Let's learn more.

Once the dominant leader in supplying a source of nitrogen for purging/blanketing gas and for producing a furnace atmosphere with up to 12% hydrogen, in recent years, exothermic gas generators have taken a back seat to non-generated gases and gas mixtures. However, with some new wrinkles and with rising costs, it may be time once again to investigate the use of exothermic gas.

Applications, sizes and types

Exothermic gas generators produce either a lean or rich atmosphere composition (Table 1) depending on the chosen air-to-gas ratio. Today, generator capacities vary from 500 to 50,000 cfh and larger. Depending on the application, the gas produced is cooled either by direct water spray into the gas stream or indirectly through a shell and tube heat exchanger. Outlet pressures of approximately 0.5 to 2.0 psig are common, but output gas pressure of up to 150 psig (and higher) can be designed. Although hydrocarbon gases such as natural gas, propane and butane are the most common, virtually any combustible fuel can be used including coke-oven gas, MAP gas, and liquid fuels (for lean atmosphere only), such as kerosene, alcohols, and fuel oil. Conversions between gases require only changing a burner and flowmeters.Exothermic gas can be used in a variety of industries including heat treating, melting, petrochemical and food. Table 1 shows typical use of exothermic gas in heat treatment and other thermal processes.

The use of a catalyst in the combustion chamber of rich exothermic gas generators is common, especially in refractory lined units, to retain temperature and ensure complete cracking of the reaction products. Upon completion of the combustion process, the gas (now depleted of oxygen) is cooled to about 100°F (38°C) is sent to its intended use.

Normally, the final dew point of the process gas is approximately 10°F (6°C) above the cooling water temperature. However, with the addition of gas dryers and refrigerated coolers, dew points as

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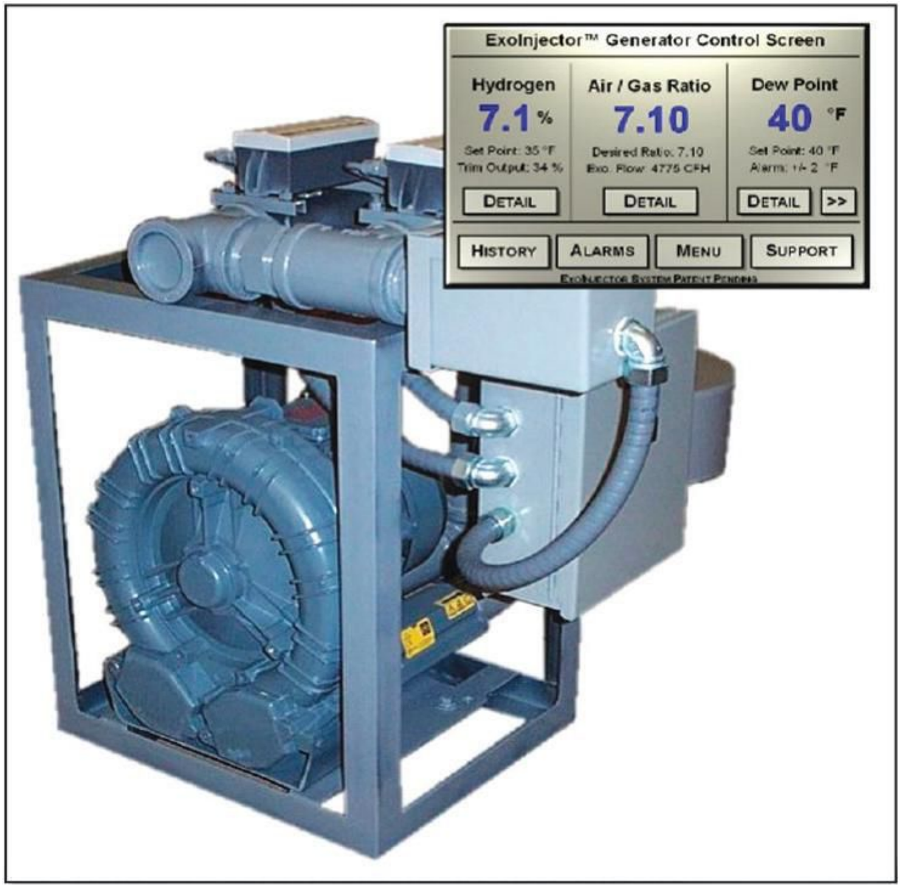

ExoInjector™ Generator Control Screen

Hydrogen 7.1 %
Set Point: 35 °F
Trim Output: 34 %

Air / Gas Ratio 7.10
Desired Ratio: 7.10
Exo. Flow: 4775 CFH

Dew Point 40 °F
Set Point: 40 °F
Alarm: +/- 2 °F

Fig. 1 Exoinjector fuel-injection gas mixing system. Courtesy of Atmosphere Engineering Co.

low as -40°F (-40°C) are possible. Purifiers are available to reduce O_2 or CO_2 to less than 10ppm, so lean exothermic gas can be used in some cases as a source of nitrogen.

Fig. 2 Exothermic gas generator with cogeneration capability. Courtesy of Modern Equipment Co.

What's new?

Mixer system technology

New systems (Fig. 1) using advanced software packages combined with regenerative blowers are now capable of delivering flow on demand throughout the working range of the generator. Ratio deviation alarms can be provided to confirm that the desired gas mixture is being produced. These types of systems typically replace existing carburetor/pumps with a gas-injection system. These systems can be adapted for low pressure gas supplies or very high turndowns.

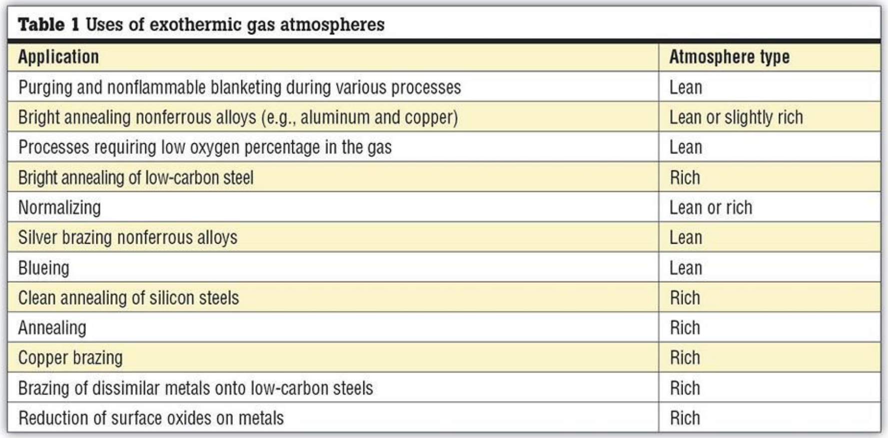

Table 1 Uses of exothermic gas atmospheres

Application	Atmosphere type
Purging and nonflammable blanketing during various processes	Lean
Bright annealing nonferrous alloys (e.g., aluminum and copper)	Lean or slightly rich
Processes requiring low oxygen percentage in the gas	Lean
Bright annealing of low-carbon steel	Rich
Normalizing	Lean or rich
Silver brazing nonferrous alloys	Lean
Blueing	Lean
Clean annealing of silicon steels	Rich
Annealing	Rich
Copper brazing	Rich
Brazing of dissimilar metals onto low-carbon steels	Rich
Reduction of surface oxides on metals	Rich

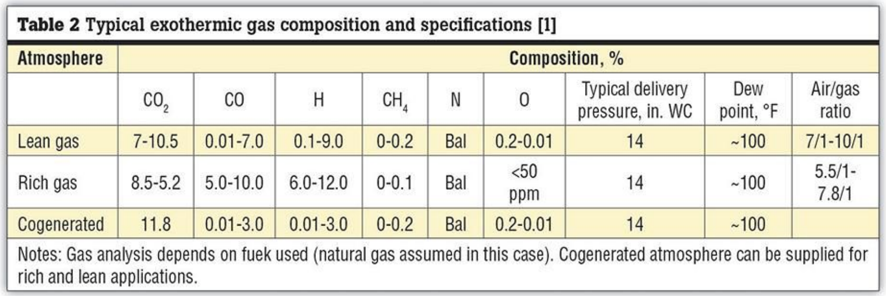

Table 2 Typical exothermic gas composition and specifications [1]

Atmosphere	Composition, %								
	CO_2	CO	H	CH_4	N	O	Typical delivery pressure, in. WC	Dew point, °F	Air/gas ratio
Lean gas	7-10.5	0.01-7.0	0.1-9.0	0-0.2	Bal	0.2-0.01	14	~100	7/1-10/1
Rich gas	8.5-5.2	5.0-10.0	6.0-12.0	0-0.1	Bal	<50 ppm	14	~100	5.5/1-7.8/1
Cogenerated	11.8	0.01-3.0	0.01-3.0	0-0.2	Bal	0.2-0.01	14	~100	

Notes: Gas analysis depends on fuek used (natural gas assumed in this case). Cogenerated atmosphere can be supplied for rich and lean applications.

Cogeneration technology

Some generators combine the ability to produce exothermic gas with that of generating steam or hot water (Fig. 2). This cogeneration technology can be adapted to either lean or rich generators and can be operated independently or integrated into an existing system, which adds flexibility, saves energy and reduces cost. To do this, the standard water-cooled combustion chamber is replaced with a commercially available fire tube boiler. The efficient use of heat recovery is reported to lower operating costs by as much as 50%.

Combustible analyzers

Today's gas analyzers measure a number of variables including O_2, CO and CO_2, total combustibles, temperature and even net combustion efficiency. Many of these systems are equipped with alarms and 4 to 20-mA signals, and can be easily integrated into a control scheme. Outputs connected to motorized valves maintain a desired gas analysis based on analyzer set point. They provide a quick and easy way to fine tune generators and furnaces or other industrial combustion processes.

Heating-chamber insulation

Reliable ceramic fiber linings now available are capable of withstanding mixture temperatures to 2,800°F (1540°C). They reportedly offer a number of benefits including rapid heating, longer life, and in some designs, the elimination of catalyst for rich operation. Conventional lining of insulating firebrick or hard firebrick also have advantages.

CO monitors

There are a number of excellent panel mounted and portable CO sensors available for monitoring the generator area, which is recommended. There are also a variety of other sensors that look at a wide range of combustible and toxic gases, as well as oxygen deficiency.

Gas coolers and refrigerant dryers

Today, refrigerant dryers are charged with environmentally friendly refrigerants such as chlorine-free HFC-134a as an alternative to traditional CFC refrigerants such as R- 12. Most dryers have the capacity to cool and dry the rated gas flow from 105°F (40°C) down to 40°F (4°C) dew point. Hot gas-bypass systems in some designs allow the dryer to run from 100% of full rating down to approximately 60%, or the dryer can be bypassed completely. They are built in a variety of sizes from 500 to 10,000 cfh and larger capacities. Gas dryers can then reduce the +40°F gas to -40°F or lower.

System maintenance

Most of the maintenance problems can be traced to poor water quality (i.e., water high in dissolved minerals and hardness values; >7 grains/gal). The water treatment options available today have made great strides in eliminating problems with water jackets and heat exchangers. Shell-and-tube heat exchanger should provide effective cooling to within 15°F (8°C) of the cooling water temperature. Symptoms often include an uncontrollably high dew point. Other causes for high dew point can be traced to faulty flowmeters and air infiltration into the combustion chamber. Sooting of the combustion chamber and catalyst bed (especially at low air to gas ratios) and soot collecting on the tubes in the heat exchanger tube bundle are other maintenance issues. Gas-analysis systems can help to prevent sooting by controlling the combustibles content in the produced gas.

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Endothermic Gas Generators – Operation and Maintenance

The father of the endothermic gas generator was a gentlemen by the name of Norbert K. Koebel, who was fond of saying to young engineers such as The Doctor, "Treat 'em right and they'll treat you right." He knew that the endothermic gas generator was the heart of any atmosphere heat-treat operation. True then, true now. Let's learn more.

Endothermic gas (aka Rx® or endo gas) is primarily used for neutral hardening and as a carrier gas for gas carburizing and carbonitriding. Today, endo gas is typically supplied to the furnace so that the furnace atmosphere is essentially neutral to the surface of many steels and can be made chemically active by the addition of enrichment (hydrocarbon) gas, ammonia or air at the furnace proper.

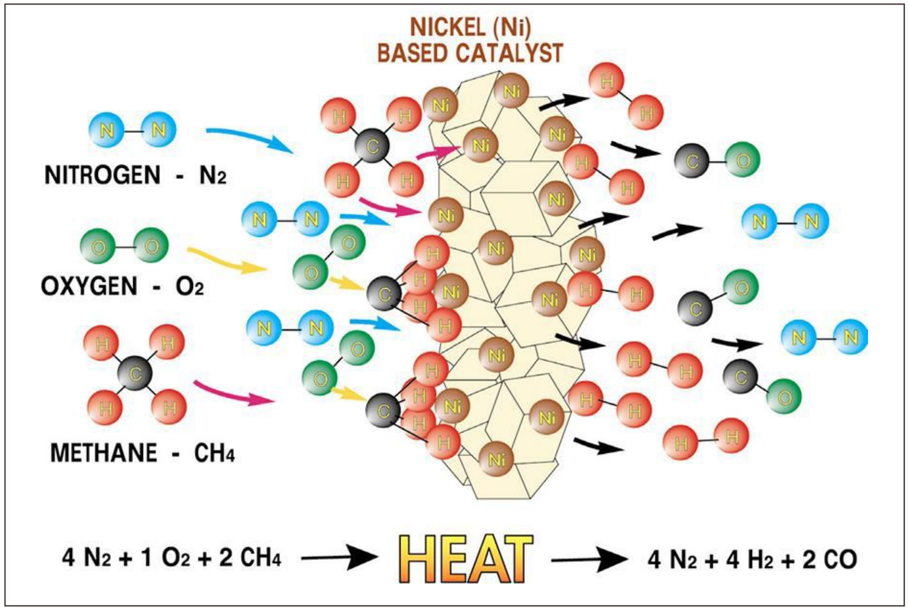

Fig. 1. Endothermic gas generator chemistry
(courtesy of Surface Combustion, Inc.)

Gas Chemistry

Endothermic gas is produced when a mixture of air and fuel is introduced into an externally heated retort at such a low air-to-gas ratio that it will normally not burn. The retort contains an active catalyst, which is needed for cracking the mixture. Leaving the retort, the gas must be cooled rapidly enough to avoid the so-called carbon reversal or carbon reformation reaction, where carbon monoxide breaks down into carbon dioxide and carbon (in the form of soot) before it reaches the furnace. The gas needs to be rapidly cooled in the temperature range of approximately 705°C (1300°F) to 480°C (900°F) or below to avoid this reaction.

The endothermic gas reaction (Equations 1-2) occurs in two steps and produces an atmosphere of nitrogen, hydrogen and carbon monoxide with varying percentages of carbon dioxide, water vapor and residual hydrocarbon as methane if natural gas is the feedstock.

$$(1)\ CH_4 + \text{air}\ (2O_2 + 8N_2) \rightarrow CO_2 + 2H_2O + 8N_2$$

$$(2)\ 2C_3H_8 + \text{air}\ (3O_2 + 11.4N_2) \rightarrow 6CO + 8H_2 + 11.4N_2$$

The endothermic gas composition (Table 1), by volume, varies depending on the type of hydrocarbon gas feedstock. The use of a nickel-based catalyst (Fig. 1) accelerates the reaction. The nickel attracts the hydrogen atoms of the methane, which attaches to the catalyst. The oxygen molecules approach and are attracted to the carbon atoms. The carbon atoms combine with the oxygen atoms to form carbon monoxide (CO). The hydrogen atoms combine to form H_2 and are released from the nickel attraction. The now-available nickel attracts new methane to continue the reaction (cracking) process. After the passage of the air-gas mixture over the catalyst, the reaction is "frozen" by chilling the gas rapidly to around 315°C (600°F) in either an air-cooled or water-cooled heat exchanger.

Table 1. Typical compositional ranges for endothermic gas

Gas constituent	**Percentage (based on natural gas)**	**Percentage (based on propane)**
N_2	40.9%	40.9%
CO	19.6%	23.3%
CO_2	0.4%	0.1%
H_2	38.9%	35.5%
CH_4	0.2%	0.2%
Dew point	+20/+50ºF	-10/-15ºF
(Air/Gas) Ratio	2.6:1	7.8:1

Equipment Overview

Endothermic gas generators consist of several basic components: a gas mixer, burner, combustion chamber and heat exchanger. They are available in single-retort (Fig. 2) and multiple-retort (Fig. 3) designs. The products of combustion of a fuel (e.g., natural gas) and air are combined at air/gas ratios typically between 2.5:1 and 3.5:1 to create the atmosphere. The reaction requires heat to proceed (hence the name endothermic), and, as such, these generators typically have heated combustion chambers.

Table 2. Dew point vs. percent carbon for an atmosphere of 20% CO, 40% H_2

%C	1500F	1550F	1600F	1650F	1700F	1750F	1800F	1900F	2000F
0.30	73	66	60	54	48	43	38	30	22
0.35	68	61	55	49	44	93	34	26	18
0.40	64	57	51	45	40	35	31	22	15
0.45	60	54	48	42	37	32	27	19	11
0.50	57	50	44	39	34	29	24	16	9
0.55	54	47	42	36	3	26	22	14	6
0.60	51	45	39	34	28	24	19	11	4
0.65	48	42	37	31	26	21	17	9	2
0.70	46	40	34	29	24	19	15	7	0
0.75	44	38	32	27	22	17	13	5	-2
0.80	42	36	30	25	20	15	11	3	-4
0.85	40	34	28	23	18	14	9	2	-5
0.90	38	32	26	21	16	12	8	0	-7
0.95	36	30	25	20	15	10	6	-2	-8
1.00	34	28	23	18	13	9	5	-3	-10

Table 3. Applications for endothermic gas

Steel type	Low carbon (to 0.20%)	Medium carbon (0.20-0.60%)	High carbon (above 0.60%)	Special steels and irons
Process	Carburizing	Clean hardening	Bright annealing	Bright annealing
	Carbonitriding	Carburizing	Clean hardening	Clean hardening
	Brazing	Carbonitriding		
	Sintering	Bright annealing		
		Carbon restoration		
		Brazing		

Fig. 2. Single-retort endothermic gas generator *(courtesy of AFC-Holcroft)*

Features

Endothermic gas generators are common equipment in the heat-treat shop. The main components of an endothermic generator (Fig. 4) are relatively simple and consist of:

- Heated reaction retort with catalyst
- Air-gas proportioning control components
- Pump to pass the air-gas mixture through the retort
- Cooler to "freeze" the reaction and prevent soot formation
- Firecheck valve to prevent backfire in the fuel supply line
- Burnoff vent to combust excess gas produced
- Thermocouples (control, over-temperature, recording) and control instrumentation

Retort

The retort for an endothermic gas generator is typically a cast alloy – HU (38% Ni, 18% Cr) and HK (20% Ni, 25% Cr) are common. In some instances, retorts are fabricated from Inconel® 600 (preferred alloy choice) or made of silicon carbide.

Retorts in most industrial generators are either thin and tall or thick and short. They vary in diameter from about 150 mm (6 inches) to 300 mm (12 inches). In larger-diameter designs, either the inlet pipe runs down through the center of the retort (to preheat the gas) or the space is occupied by a closed-ended pipe, typically 50-75 mm (2-3 inches) in diameter to avoid issues with a cold center in the catalyst bed.

Catalyst

For economic reasons only, manufacturers have gone away from supplying pure nickel shot as a catalyst and today utilize insulating firebrick catalyst cubes typically 25 mm (1 inch) in size coated with 3-7% nickel sulfate ($NiSO_4$). Smaller-sized cubes, 17.5 mm (11/16 inch) and spheres of 19 mm (3/4 inch) diameter, have also been used, but the pressure drop through the catalyst bed must be monitored due to increased packing density. The use of a refractory catalyst often suggests a smaller-diameter retort to assure both proper heat distribution throughout the catalyst bed and adequate dwell time at temperature for complete dissociation.

Mixer

A mixing pump and (optional) carburetor control the air/gas ratio of an endothermic gas generator in the range of 2.5:1 to 3.5:1 for natural gas (Fig. 5a) and 7.25:1 and 9.25:1 for propane (Fig. 5b). In some generator designs air/gas ratios have been known to run as low as 2.0:1 for natural gas.

Fig. 3. Modular multi-retort endothermic gas (Rx®) generator *(courtesy of Surface Combustion, Inc.)*

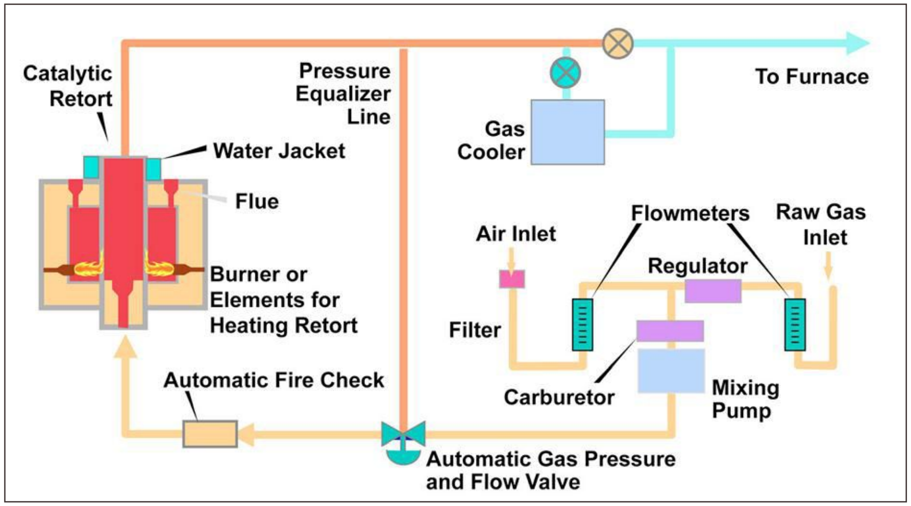

Fig. 4. Endothermic gas generator schematic piping arrangement[3]

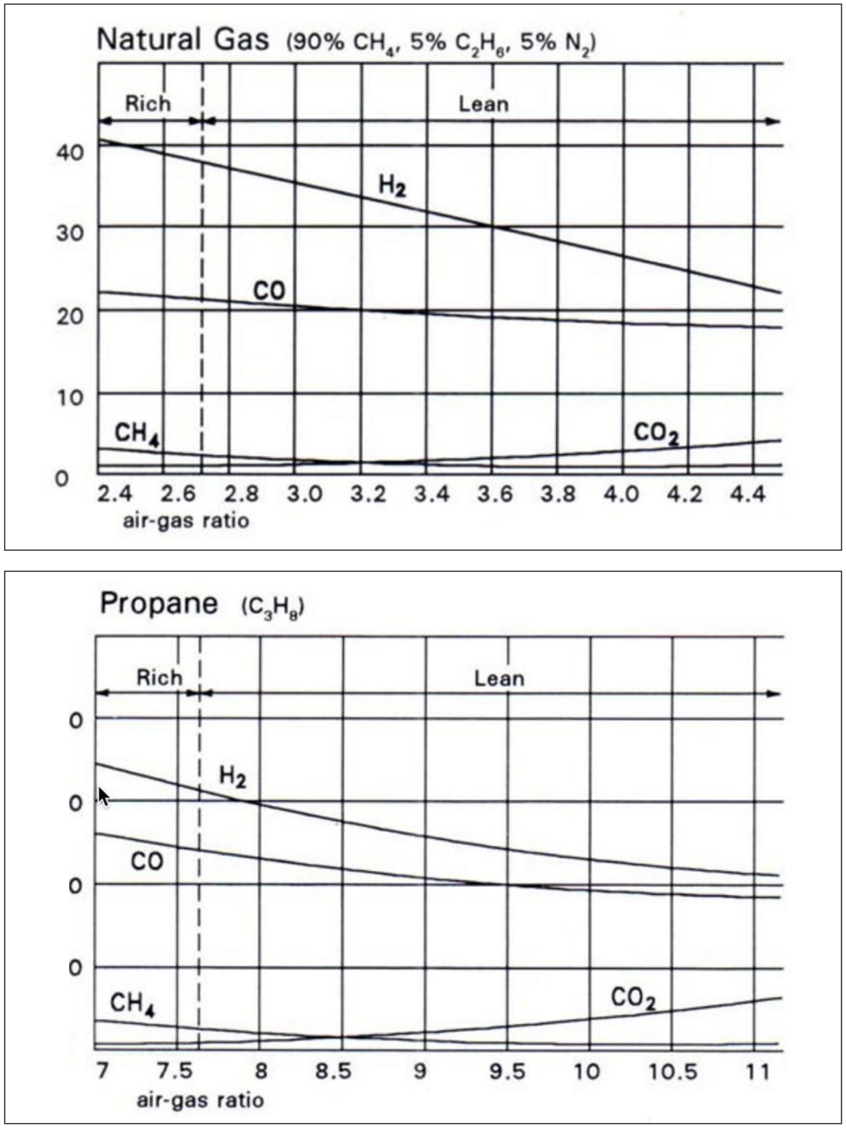

Fig. 5a. & 5b. Air-gas ratio values and gas compositions for endothermic gas generators operating with a natural gas or propane feedstock[5]

Today, most generators run a dew point (Table 2) in the range of +4.5 ± 0.2ºC (+40 ± 2ºF) to minimize maintenance concerns (e.g., sooting). For carburizing applications, some heat treaters prefer to run generators in the range of -1.1 ± 0.2ºC (+30 ± 1ºF). Final adjustments to the atmosphere are typically made at the furnace.

Similar to their exothermic gas generator counterparts, endothermic generators can be equipped with advanced software packages combined with regenerative blowers capable of delivering "flow on demand" throughout the working range of the generator (Fig. 6).

Insulation

The heating chamber of an endothermic gas generator can either be refractory-lined or lined with ceramic-fiber insulation. Modular designs and clam-shell-style chambers (to facilitate retort removal) housing single retorts are more popular today than the past practice of using large heating chambers housing multiple retorts. Cracking of the refractory and shrinkage of the ceramic fiber result in hot spots on the shell that can be avoided by proper design and heat-up practices of the unit from ambient temperature.

Heating Source

Endothermic gas generators are either gas-fired or electrically heated. If gas-fired, ring burners or combustion burners are commonplace. Electrically heated units either use nickel-chromium or silicon-carbide heating elements. If metallic, they often include the addition of rare-earth elements (e.g., Hf, Y) to extend operating life since the elements run in air. Power is regulated by on/off or proportional control (zero-fired or phase-angle-fired SCRs). The typical operation temperature of an endothermic gas generator is 1010-1095°C (1850-2000°F), depending on design. Most units run in the 1065°C (1950°F) range.

Thermocouples

Most generators use either type K or type S thermocouples. Type N thermocouples are often used as a check (SAT) thermocouple.

Firecheck

The firecheck (Fig. 7) is a safety device designed to prevent backfire into the incoming gas supply line. The functionality of the firecheck should be checked every six months or more frequently if recommended by the manufacturer. The sad reality is that most heat treaters and plant maintenance personnel don't understand its function and never check that it is operating properly!

Fig. 6. Endoinjector® fuel-injection gas mixing system mounted on an endothermic gas generator *(courtesy of Atmosphere Engineering Company)*

Applications

The uses of endothermic gas (Table 3) include the following heat-treatment processes:

- Carburizing
- Bright hardening
- Sintering
- Brazing
- Carbonitriding
- Carbon restoration
- Neutral hardening

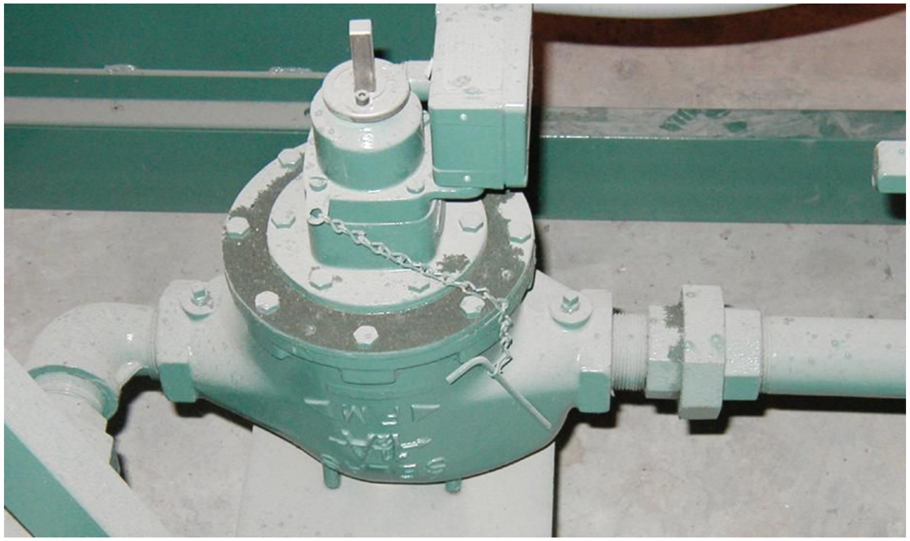

Fig. 7. Firecheck on an endothermic gas generator

Maintenance

One of the tasks The Doctor was assigned that first week on the job was to change the catalyst in an endothermic generator – a task that left an indelible impression on a heat-treat rookie. It was fun, it was informative and it was hard work! Maintenance on endothermic gas generators today is even more critical than it was back then and includes the following general tasks (depending on the generator involved):

Test to determine if the catalyst bed is sooted. Indicators are:

- Small ratio adjustments do not result in a change of dew point.
- Very high dew-point readings
- Methane (CH_4) higher than 0.5% after a proper air burnout operation
- When operating between 30-40°F, the CO_2 should be approximately the equivalent to the dew point divided by 100 (0.30-0.40% CO_2).

Over the years, checklists[6] have been developed (and customized) for most endothermic gas generators. Here's a general list of maintenance activities by suggested frequency:

Daily checks

☐ Check the temperature control instrumentation for proper operating temperatures.
☐ Check for proper flow and pressure of the generated atmosphere.
☐ Check for proper inlet air-gas ratios.
☐ Check either the gas analysis or the dew point of the unit. Make sure that manual and automatic readings coincide. Recalibrate automatic gas analyzers.
☐ Check that the floats in the gas flow tubes are free and operating.
☐ Check that the compressor is operating and functional.
☐ Check that the gas cooler is operating. If installed, check the temperature of the exiting gas to confirm

that the carbon reversal reaction is not occurring (and that soot is not being formed) on gas discharge from the generator to the furnace.
  – If the system is water-cooled, check sight drains or temperature gauges (or both) to confirm proper water flow, pressure and temperature.

- ☐ Check that there are no leaks from any of the joints on the process retort, particularly at the point of entry of the process gas from the compressor.
- ☐ Check the heating chamber and visually confirm it is incandescent.
  - – If gas-fired, check the combustion equipment including pilots, spark igniters and flame rods for proper operation. Check burners for proper ignition and combustion characteristics.
  - – If electrically heated, check the current draw on the heating elements.
- ☐ Make sure atmospheric burners or pilots (or both) are protected from drafts.
- ☐ Check the burnoff stack to confirm ignition of flammable atmosphere gases.
- ☐ Monitor the carbon monoxide (CO) level in the immediate area of the generator (confirm it is <0.01%).
- ☐ Check for proper operation of the exhaust hoods and stacks.
- ☐ Check for excessive temperature in all areas of the generator.
- ☐ Check hand valves, manual dampers, secondary air openings or adjustable bypasses, valve motors, and control valves for smooth action, proper position and adjustment.
- ☐ Check all pressure switches for proper pressure settings.
- ☐ Check blowers, compressors and pumps for unusual noise or vibration.
- ☐ Check belt tension.
- ☐ Check for evidence of any damage, from any cause.

Weekly Checks

- ☐ Burnout/regenerate the catalyst as per the recommended manufacturer's instructions and at the frequency recommended by the manufacturer.
- ☐ Remove the air filter from the compressor, clean and/or replace.
- ☐ Once the burnout/regeneration is complete, start the gas-making procedure. Check either the gas analysis or the gas dew point.
- ☐ Make sure the flame-sensing equipment is in good condition, properly located and free of foreign debris.
  - – Clean the burner flame rod.
  - – Check ignition spark electrodes for proper operation and gap.

☐ Test thermocouples and leadwire for shorts and loose connections. Check protection tubes for sagging, cracks and proper insertion depth.
☐ Test visible and audible alarm systems for proper functionality.
☐ Remove the air filter from the compressor, clean and or replace.
☐ Check ignition spark electrodes for proper operation and gap.
☐ Remove the floats from the flow-meter glass tube and clean the internal and external surfaces of the flow meter and re-assemble.
☐ Check the thermocouples for calibration.
☐ Check the gas pressure of the gas at the compressor.
☐ Check the instrumentation for calibration. This means temperature as well as gas analysis or dew point.
☐ Test interlock sequences of all safety equipment. Manually make each interlock fail, noting that related equipment closes or stops as specified by the manufacturer.

Monthly Checks

☐ Test pressure-switch settings by checking switch movements against pressure settings and comparing with actual impulse pressure.
☐ Inspect all electrical devices for proper current and voltage and be sure that all electrical contacts and switches are functioning properly.
☐ Clean or replace the air blower filter.
☐ Clean any filters or strainers.
☐ Inspect burners and pilots.
☐ Check ignition cables and transformers.
☐ Test automatic and manual turndown equipment.
☐ Test pressure-relief valves; clean as necessary.
☐ Check backpressure regulators; inspect and clean/replace diaphragms.

Quarterly Checks

☐ Inspect the catalyst and fill (if necessary) to the recommended mark, or replace if necessary.
☐ Inspect and clean the burners. Check the gas train for functionality.
☐ Remove the gas delivery line from the generator to the furnace and clean. There may be soot present if there have been any problems with the gas cooler.
☐ Check all safety solenoids and safety controls.

Semiannual or Annual Checks

☐ Inspect the retort, refractory, heat exchangers, refrigerators, dryers and other accessories; repair or replace as necessary.
☐ Lubricate the instrumentation, valve motors,

valves, blowers, compressors, pumps and other components.

- ☐ Test instrumentation; clean slidewires and electrical components.
- ☐ Test flame safeguard units.
- ☐ Burn out carbon in the retort(s).
- ☐ Check for plugging of hot pipes, tube bundles and jacketed pipes.

Problems

The most common problems experienced with endothermic gas generators involve:

- Temperature – Efficiency of the endothermic reaction is thermally dependent. The entire atmosphere must reach a minimum temperature in order for the gas to be completely reacted. If the temperature is not reached no reaction will occur.
- Gas coolers – If the gas coolers are not operating efficiently, sooting will occur due to the carbon-reversal reaction. This typically takes place outside the retort and causes a restriction in the outlet piping and the piping to the furnace, which causes backpressure to occur and a loss of flow. Soot can accumulate in a gas cooler in a matter of minutes, which is why ratio control is so important.
- Catalyst – The catalyst is often a nickel-impregnated refractory chosen for its capability to support itself at the required high operating temperature to withstand catalyst regeneration cycles and to maintain physical stability in the presence of the reaction products (mostly carbon monoxide). If the temperature is not high enough and the gas is not completely reacted, then sooting in the catalyst bed will result. Once the catalyst starts to soot, it becomes ineffective, and the gas composition will drift and produce higher percentages of methane, carbon dioxide and water vapor.

Safety

Like any other piece of atmosphere equipment that uses a combustible gas, great care must be taken when starting up, producing gas and operating endothermic gas generators. Returning the unit to service after a shutdown, the generator temperature should be raised slowly to reduce the risk of thermal shock to the refractory and creating stress on the process retort. Under no circumstances should you consider putting gas or any other combustible gas mixture into the retort (or into the furnace) when the temperature is below 760ºC (1400ºF). Otherwise, a serious explosion will most likely occur and can result in serious injury or death and significant damage to the equipment. All NFPA 86 standards should be followed.

Summary

Endothermic gas generators have a long and proven track record of success. The gas produced is relatively stable and adequate for a broad spectrum of process applications. Maintenance is relatively simple, and problems with the equipment and technology are well understood and solvable on the shop floor.

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Peak Shaving: What Impact Does It Have On The Heat Treater?

Heat treating requires energy and lots of it. We have all heard that the cost of natural gas is going up this winter, possibly to record levels. Availability is always questionable. What we haven't heard that much about is the type of gas being delivered to our doorstep. As heat treaters, we have learned to respect and perhaps dread the words "peak shaving." Once only an occasional concern, it is becoming a fact of life, one that we must deal with on almost a daily basis. It can play havoc with our generated atmospheres, change the way in which our heating systems perform and worst of all adds variability to our heat treating processes. This means we must look carefully at our process control devices to assure that we have the correct ones and that they are operating properly. Let's learn more.

In a perfect world…

Demand for clean-burning natural gas is growing around the world. And in North America, the gas-energy marketplace now offers an array of competitive choices–and risks–from wellhead to burner tip.

Natural gas is a clean-burning, highly efficient fuel used in many heat treating furnaces as a source of heating and in gas generators to create a furnace atmosphere most typically endothermic or exothermic gas. Natural gas is typically 90-95% methane, depending on location. The issues and problems associated with controlling equipment being heated with or generating atmospheres from natural gas are well understood. Manual or automatic dew point analyzers, infrared controllers (single- and three-gas analyzers), and oxygen probes are common control devices. In the new gas-energy marketplace, there is a reliance on propane peak shaving and standby systems to provide supply security and flexibility for most gas utilities and consumers. Our challenge as heat treaters is to manage this change.

Peak shaving explained

Peak shaving involves the use of fuels and equipment to generate additional gas to supplement the normal supply of pipeline gas during periods of extremely high demand. Dilution of the incoming natural gas supply is usually done with another hydrocarbon gas (typically aerated propane) to achieve a heat content (Btu value) close to that of natural gas. The objective of peak shaving (from the supplier's point of view) is to save natural gas and to ensure that a constant fuel supply is available. The challenge for the heat treater is to detect and control the variability induced by a change to the gas supply.

Aerated propane

Propane is one of several liquefied petroleum gases derived during both natural gas production and crude oil refining. The gas we call propane actually consists of a mixture of different gases, which depending on source, will vary in composition, typically between these limits: propane (87.5-100%); ethane (0-7%); propylene (0-5%) and butanes such as n-butane and iso-butane (0-2.5%). An odorant, ethyl mercaptan (0-50 ppm) is often added. In the case of propane, the percentage of heavy hydrocarbons is a significant concern since they tend to form soot and tar.

Commercial propane, under the trade names LPG, LP-gas and HD-5 Propane, is widely used as peak shaving fuel. Stored as a liquid, a gallon of propane contains the energy equivalent of over 90 ft^3 of natural gas. Eleven gallons of propane have about the same energy as 1-million ft^3 (Mcf) of natural gas, or about 1 million million Btu (MMBtu). Therefore, the use of "propane-air" for direct replacement of natural gas makes sense.

Propane-air is a highly versatile and scalable commodity. The argument for its use is that it offsets costly ongoing demand charges with a fixed asset supply. The reality is that it offers a way to expand system capacity without adding the costs associated with off-site development, extension of gas mains, and adding more pipelines. The anticipated benefit to customers is lower cost gas energy.

Most peak shaving systems operate as follows: liquid is drawn from the tank(s) and the pressure raised via motor-driven pumps. The liquid

LDC (local distribution company)
- LDCs operate natural gas-distribution systems linking consumers and pipelines.
- LDCs may also provide gas storage and peaking services.

LNG (liquefied natural gas)
- Methane becomes a liquid for storage or transport when cooled to about -260ºF (-160ºC) in insulated tank cars, trucks, and ships.

LPG (liquefied petroleum gas)
- LPG, or LP, gas refers to several liquids, including propane and butane.

NG (natural gas)
- NG is a mixture of methane and various other hydrocarbons and inert gases.

NGL (natural gas liquids)
- NGLs are hydrocarbon components of natural gas other than methane, including ethane, propane and butane.

Table 1 Typical Propane-Air Data(a)

Btu/ft^3 of propane air mixture	Vol % propane	Vol% air	Vol% oxygen	Specific gravity of propane-air mixture
1450	57.54	42.46	8.874	1.299
1440	57.14	42.86	8.957	1.297
1430	56.75	43.25	9.040	1.295
1420	56.35	43.65	9.123	1.293
1410	55.95	44.05	9.206	1.291
1400	55.56	44.44	9.289	1.289
1390	55.16	44.84	9.372	1.287
1380	54.76	45.24	9.455	1.285
1370	54.37	45.63	9.538	1.283
1360	53.97	46.03	9.621	1.281
1350	53.57	46.43	9.704	1.279
1340	53.17	46.83	9.787	1.277
1330	52.78	47.22	9.869	1.274
1320	52.38	47.62	9.952	1.272
1310	51.98	48.02	10.035	1.270
1300	51.59	48.41	10.118	1.268
1290	51.19	48.81	10.201	1.266
1280	50.79	49.21	10.284	1.264
1270	50.40	49.60	10.367	1.262
1260	50.00	50.00	10.450	1.260
1250	49.60	50.40	10.533	1.258

(a)Source: Ruffcon, S. D., Hoch, J.P., Energy Gases Data Extensions (1994), Standby Systems Inc.

is heated in the vaporizer and converted to a superheated vapor. A gallon of liquid propane will produce about 36 standard ft^3 of vapor. The energy content of 1 scf of propane vapor is about 2,520 Btu with a specific gravity of 1.52. Propane vapor is mixed or blended with air, producing a propane-air mix. Propane-air mixtures can vary in their heat content (Table 1). For example, a propane-air mixture containing 1,383 Btu/ft^3 has burning characteristics similar to natural gas containing 1,000 Btu/ft^3 with a specific gravity of 0.60. The mix is then injected into the natural-gas distribution system.

The volume of propane-air is normally limited to less than about 50% of the combined natural gas/propane-air stream, keeping the specific gravity of the combined stream at less than 1.00 (air = 1.00). A common mixture is 45-55% propane-air mix in natural gas with 95% methane.

Control issues

As energy prices spiral to new heights, and supply interruptions become a more frequent occurrence, the use of energy and active management of energy demand are more important than ever since electric and gas utilities are under constant pressure to reduce peak demand, and today this affects all industrial customers, large and small. The frustration for the heat treater is that often we are not given the choice of accepting or controlling when these alternative energy sources are provided to us. Peak shaving is one such instance. One of the ways in which we must adapt is to understand the importance of the analysis of the incoming supply fuel. The use of specific gravity analyzers and gas chemistry analysis using gas chromatography or other gas analysis techniques is mandatory.

Propane-air additions to natural gas can play havoc with generator ratios, and unsaturated hydrocarbons, such as ethylene and propylene, break down quickly into oily soot and coke, which can be carried along with the gas stream into the furnace. It is essential, therefore, that the gas supply be kept under close observation, and that all critical components of the system be inspected and cleaned on a regularly scheduled basis. It is always advisable to inform your gas/utility supplier of your special needs or concerns, whether they can act on them or not.

What control devices are necessary?

Oxygen probe control has been shown to minimize the effect of peak shaving. However, the use of oxygen probes on furnaces and generators has been so successful that routine dew point or infrared analysis has become less and less frequent and in some cases, abandoned altogether. This means that we are only seeing a portion of what we need for complete process control. Rapid response, on-line multigas analyzers are now available, and when used in conjunction with temperature and pressure monitoring, can provide real-time understanding of the furnace atmosphere. When setup with the proper feedback loops, this allows for complete process control, reduced variability and improved product quality.

Where do we go from here?

The secret to detecting and controlling the effects of peak shaving on the end product quality after heat treatment begins and ends with the constant monitoring of the changes that are taking place; in the incoming gas stream, in the generated atmosphere and in the heating system performance. Benchmarking results obtained with the data gathered from multiple devices is the best way to keep in control. Peak shaving is here to stay, and to combat its negative effects, the heat treater will need to spend both time and money on effective process control devices. It is well worth the investment.

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Considerations in Heat Treatment Part Two: Furnace Atmosphere Control

The composition of the furnace atmosphere is constantly changing, so we must use measurement and control devices to ensure good quality control. In particular, for atmospheres designed to run "neutral," avoiding decarburization and/or carburization is critical to the proper functionality of the component part. This is accomplished by making sure that one or several of the following control methods is monitoring and/or controlling the process:

- Dew point analysis
- Infrared analyzer (single- or multiple-gas analyzers)
- Oxygen (carbon) probe

The trend today is to use multiple measurement tools to obtain the most accurate snapshot of the atmosphere in real time.
Whether neutral or case hardening, a number of variables determine how well a furnace does its job. Throughout the entire cycle it is critical to the process that we control the percentage of carbon dioxide, oxygen and water vapor and the ratio of

Table 1. Typical dew point levels

Dew Point, ˚F (˚C)	Water Vapor (ppm)
+46 (+8)	10,590
+25 (-4)	4,320
0 (-18)	1,240
-40 (-40)	127
-90 (-68)	3.4

Note: 1.04% moisture = 10,590 ppm water vapor = +46°F dew point

Table 2. Dew point vs. surface carbon (%)

DewPoint,˚F(˚C)	1500˚F	1600˚F	1700˚F
+30 (-1.1)	1.10	0.80	0.55
+40 (4.4)	0.85	0.60	0.40
+50 (10)	0.60	0.40	0.27

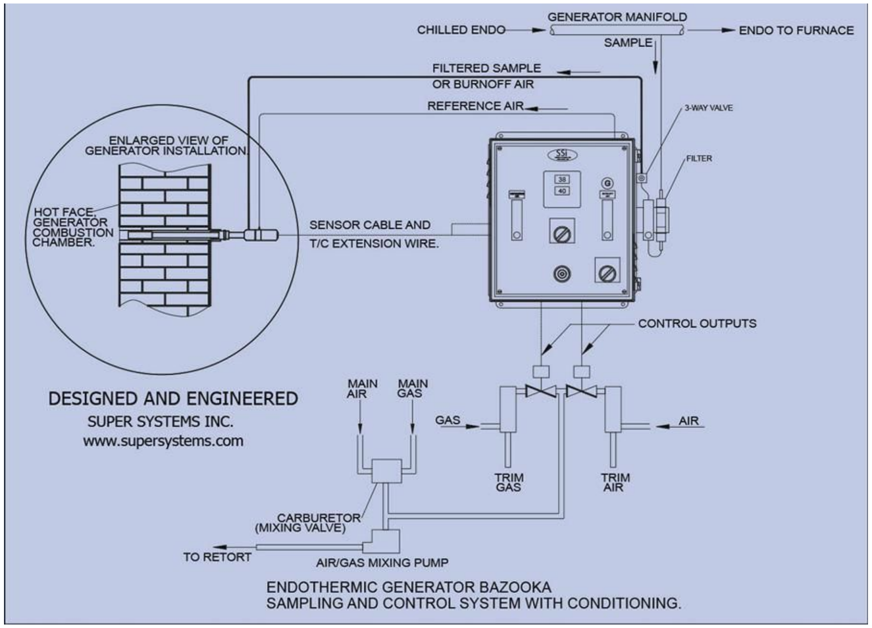

Fig. 1. Endothermic generator dew point control scheme via oxygen probe (Courtesy of Super Systems, Inc.)

enriching gas (or air) to carrier gas. For example, surface carbon can be controlled within ±0.10% by measuring one or more of these constituents. Natural gas is a clean-burning, highly efficient fuel used in many heat treating furnaces as a source of heating and in gas generators to create a furnace atmosphere most typically endothermic or exothermic gas. Natural gas is typically 90-95% methane, depending on location. The issues and problems associated with controlling equipment being heated with or generating atmospheres from natural gas are well understood. Manual or automatic dew point analyzers, infrared controllers (single- and three-gas analyzers), and oxygen probes are common control devices. In the new gas-energy marketplace, there is a reliance on propane peak shaving and standby systems to provide supply security and flexibility for most gas utilities and consumers. Our challenge as heat treaters is to manage this change.

Table 3. Typical field data for an operating endothermic gas generator[1,2]

Constituent	1ST Quarter	2nd Quarter	3rd Quarter	4th Quarter
% CO	19.02	19.66	19.32	19.21
% CO_2	0.260	0.252	0.254	0.257
% CH_4	0.07	0.08	0.09	0.09
Generator Dew Point, °F	+39	+39	+40	+39
Dew Point at Furnace Inlet, °F	+37	+38	+38	+38
Zonal Dew Point (Z1–Z4), °F[3]	+40 to +42	+40 to +42	+40 to +42	+40 to +42

Notes: [1] 3,000 CFH (85 m^3/hour) output; [2] Natural gas feedstock; [3] Neutral hardening

Dew Point Control

Dew point is defined as the temperature at which water vapor starts to condense. In simplest terms, a dew point analyzer measures the amount of water vapor present in the furnace atmosphere (Table 1). This information can then be used to determine the carbon potential of the atmosphere (Table 2). It is considered an indirect measurement technique if it

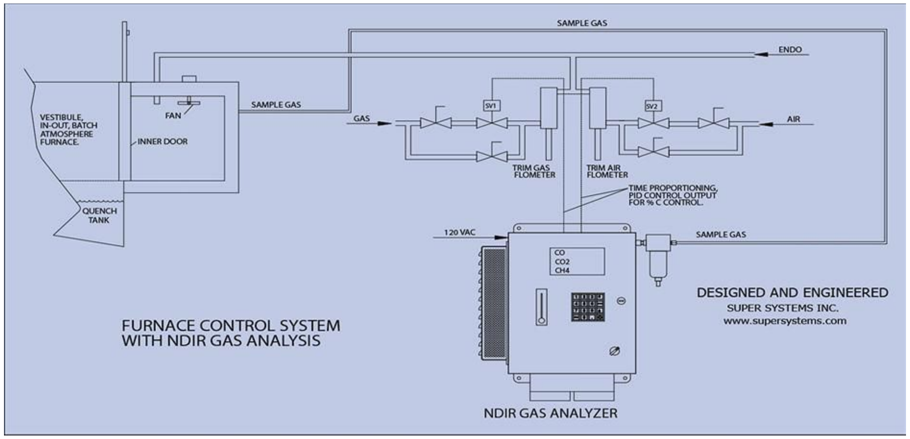

Fig. 2. Furnace infrared (three-gas) control scheme (Courtesy of Super Systems, Inc.)

involves pulling a gas sample from the furnace into the instrument.

If performed properly, dew point is a simple and accurate atmosphere measurement technique and indicates the condition inside the atmosphere generator or heat-treat furnace. It will tell you if the reaction is stable or unstable (constant dew point or changing dew point over time). It can tell you when the catalyst bed in your endothermic gas generator is starting to soot and if there is a water leak, an air leak or non-uniformity ("breathing") of the atmosphere inside your furnace.

The types of dew point analyzers available include capacitance sensors, chilled mirrors and fog chambers (Alnor®, dew cup). Condensation is a problem for all dew point devices if the sample temperature is less than the dew point of the gas. One solution is to heat trace the sample lines. If the ambient temperature exceeds 105ºF (40ºC) as it often does in heat-treat shops, the instruments will not give accurate readings unless special precautions are taken. Dew point control can also be measured using an oxygen-probe arrangement (Fig. 1).

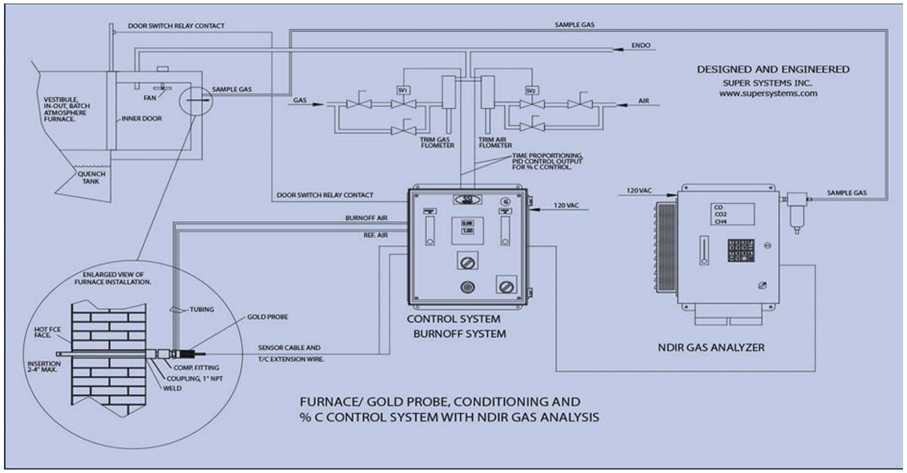

Fig. 3. Combination furnace oxygen-probe and infrared control (Courtesy of Super Systems, Inc.)

Infrared Control

Infrared analysis uses the infrared spectrum (light, for example, is in the middle of the spectrum in the "visible" range) to analyze a gas sample and determine the percentage of that constituent in the

Table 4. Control settings – mesh-belt conveyor furnace

Steel	Process	Setpoint Values (Oxygen Probe), %C	Actual Values (Oxygen Probe), %C
10B21	Carbonitriding	1.05	0.69–0.83
1038	Neutral hardening	0.38	0.19–0.34

furnace atmosphere. Single-gas (carbon monoxide) or multiple-gas (carbon dioxide, carbon monoxide, methane) analyzers detect the presence of these gases in the furnace atmosphere.

The amount of carbon dioxide in the furnace is another indirect way of measuring the carbon potential of the atmosphere.

Today, three-gas infrared analyzers are used to monitor the carbon monoxide, carbon dioxide and methane contents of generators (Table 3) and furnaces (Fig. 2). Individual gases absorb infrared radiation of very specific wavelengths. The amount of absorption increases with gas concentration. The unit operates under the principle that a gas sample passes through a cell where a heated wire emits infrared energy of known wavelength. The sensor converts measured infrared energy into an electrical signal. These values are usually compared to the values obtained with a reference gas. Infrared analyzers are known for their fast response and are easily calibrated.

Oxygen-Probe Control

The oxygen (or carbon) probe is an in-situ device that looks similar to a thermocouple for measuring temperature and typically sits inside the furnace, inside the generator above the catalyst bed or in a separate heated "well" into which the furnace atmosphere is pumped. In whatever location, the oxygen probe measures changes in the furnace atmosphere. A difference in partial pressure of oxygen in the furnace atmosphere and the partial pressure of oxygen in the room air induces a voltage – electromotive force, or EMF – across the electrodes in the probe. At any given temperature, there is a known relationship between the voltage output and the oxygen potential of the atmosphere. The oxygen potential can be directly related to the carbon potential. Therefore, the carbon potential of the furnace atmosphere can be controlled by monitoring the furnace temperature and the probe output.

The oxygen probe uses a conductive ceramic sensor manufactured from zirconium oxide (Zr_2O_3) that can be mounted in-situ inside the furnace (Fig. 3). Operating range is between 1200°F (650°C) and 1800°F (980°C). Oxygen probes can be used for a variety of gases, but they need to be calibrated for the specific atmosphere used. They are fast-response devices and subject to contamination by carbon or zinc. When used in carbonitriding applications, the presence of ammonia will shorten the life of the probe.

Typical oxygen-probe data for a mesh-belt conveyor furnace (Table 4) running at the rate of 4,000 pounds/hour (1,815 kg/hour) shows deviations from setpoint values due to nonequilibrium conditions present within the furnace.

Important Cautions

In more than one instance, data was being analyzed and adjustments made based on improper sample-port locations, instrumentation that was not properly calibrated and/or sample ports not extending fully into the furnace chamber. Mistakes such as these can be devastating to both equipment and part quality.

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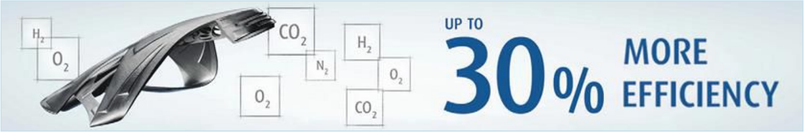

Furnace Atmosphere Control Methods (Part One)

It seems ironic that the one constant in atmosphere heat treating is that the composition of the furnace atmosphere is always changing. As such, we must use measurement and control devices to ensure the proper atmosphere composition necessary to achieve the desired metallurgical quality and mechanical/physical properties throughout the workload. Let's learn more.

The trend today is to use multiple measurement tools to obtain the most accurate snapshot of the atmosphere in real time. In addition, the industry is rapidly moving toward automated control systems.

Manual or Automatic Control?

In one form or another, atmosphere control has been around for decades, and the control method may be categorized as either manual or automatic. There are benefits and limitations to both (Table 1).

In a manual control scheme, success depends on the vigilance of the people operating the equipment, relying on their experience coupled with either trial and error or historical records/information. A typical example of this type of control would be as follows.

A furnace operator sets up a load, charges it into the furnace and manually adjusts temperature and atmosphere parameters. As process values approach setpoint, the operator begins periodically checking the atmosphere with sensor devices (e.g., dew-point instruments) and logging values on a heat-treat form of some design.

If process deviation occurs, the operator intervenes and attempts to make adjustments "on the fly" based on experience. At the end of the process, the heat

Fig. 1(a). Typical oxygen (carbon) probes *(courtesy of Super Systems Inc.)*

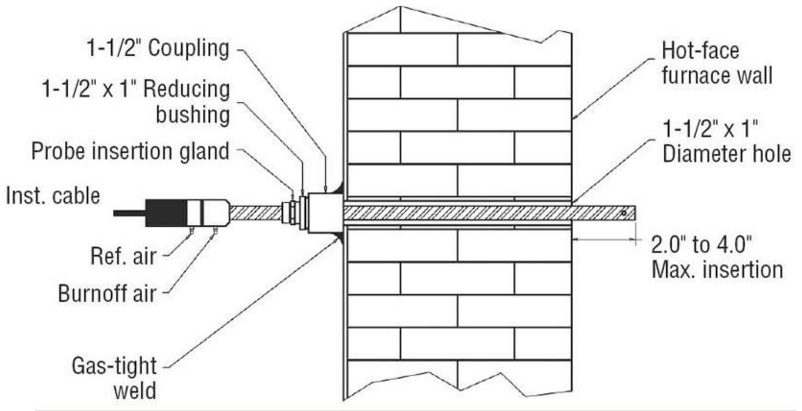

Fig. 1(b). Recommended mounting arrangement *(courtesy of Super Systems Inc.)*

treater checks the load to confirm it does not need to be reworked or scrapped. Essentially, operators learn what works and what does not on a case-by-case basis over time, adding to their knowledge base. For key parameters or data records, a form, a recorder or a printer would provide a paper copy to be filed.

Automated control differs from manual control mainly in that sensors, computers and specially designed software are used during the heat-treatment process to monitor, control and record data about the furnace atmosphere. Load-entry software can even be used to control the timing of a charge and electronically record all load parameters set up by the operator. Output is set and changed by a process controller, such as a loop controller, designed specifically for heat-treatment processes or a PLC.

If the controller detects a process deviation or a condition likely to result in one, it can make adjustments and/or generate a process alarm if operator intervention is needed. Information is delivered through mobile devices to operators, supervisors or even maintenance based on an escalation and profile of the alarm and recipient. Data logging is electronic. Logged data points can be accessed at any point in the future.

Measurement Devices and Sensors

Monitoring and/or controlling the heat-treatment process is accomplished by use of sensors and measurement devices. The most common of these are:

- Oxygen (carbon) probes
- Non-dispersive infrared analyzers (NDIR) – single- or multiple-gas
- Dew-point analyzers
- Oxygen analyzers
- Combustion analyzers

Whether neutral or case hardening, annealing or normalizing, a number of variables determine how well a furnace does its job. Throughout the entire cycle it is critical to the process that we control the percentage of carbon dioxide, oxygen and water vapor as well as the ratio of enriching gas (or air) to carrier gas.

For example, surface carbon can be controlled within ±0.05% C during carburizing by measuring one or more of the gases mentioned above and adjusting the addition gases (hydrocarbon and/or air) accordingly.

Oxygen (Carbon) Probes

The oxygen (aka carbon) probe (Fig. 1a - b) is an in-situ device that looks similar to a thermocouple for measuring temperature and typically sits inside the furnace, inside the generator (typically above the catalyst bed or in a separate heated "well" into which the furnace atmosphere is pumped). In whatever location, the oxygen probe measures minute changes in oxygen concentration of the furnace atmosphere.

A difference in partial pressure of oxygen in the furnace atmosphere and the partial pressure of oxygen in the room air induces a voltage across the electrodes in the probe. At any given temperature, there is a known relationship between the probe millivolt output and the oxygen potential of the atmosphere. The oxygen potential can be directly related to the carbon potential. Hence, monitoring the furnace temperature and the probe output can control the carbon potential of the furnace atmosphere.

The oxygen probe uses a conductive ceramic sensor, most often manufactured from zirconium oxide (Zr_2O_3). Operating range of the probe is normally 650-980˚C (1200-1800˚F). Oxygen probes can be used for a variety of atmosphere compositions, but they need to be calibrated for the specific one in use. They are fast-response devices and subject to contamination by carbon, zinc and certain stop-off paint vapors. When used in carbonitriding applications, the presence of ammonia will shorten the life of the probe.

An oxygen probe in a carburizing atmosphere must incorporate periodic air burnouts (Table 2). The carburizing process in use will determine the burnout frequency.

A burnout consists of at least 0.28 m^3/hour (10 cfh) of air piped to the burn-off fitting on the head of the probe. Room air or filtered combustion air are most commonly used. It is important not to use compressed air due to water and oil contamination that can damage the probe. The carbon controller should either control the frequency and duration of the burnout or shut off the gas additions in order to prevent excessive gas from compensating for the flow of air to the probe. Burnout flow and duration recommendations vary by manufacturer based on sheath diameter and tip design.

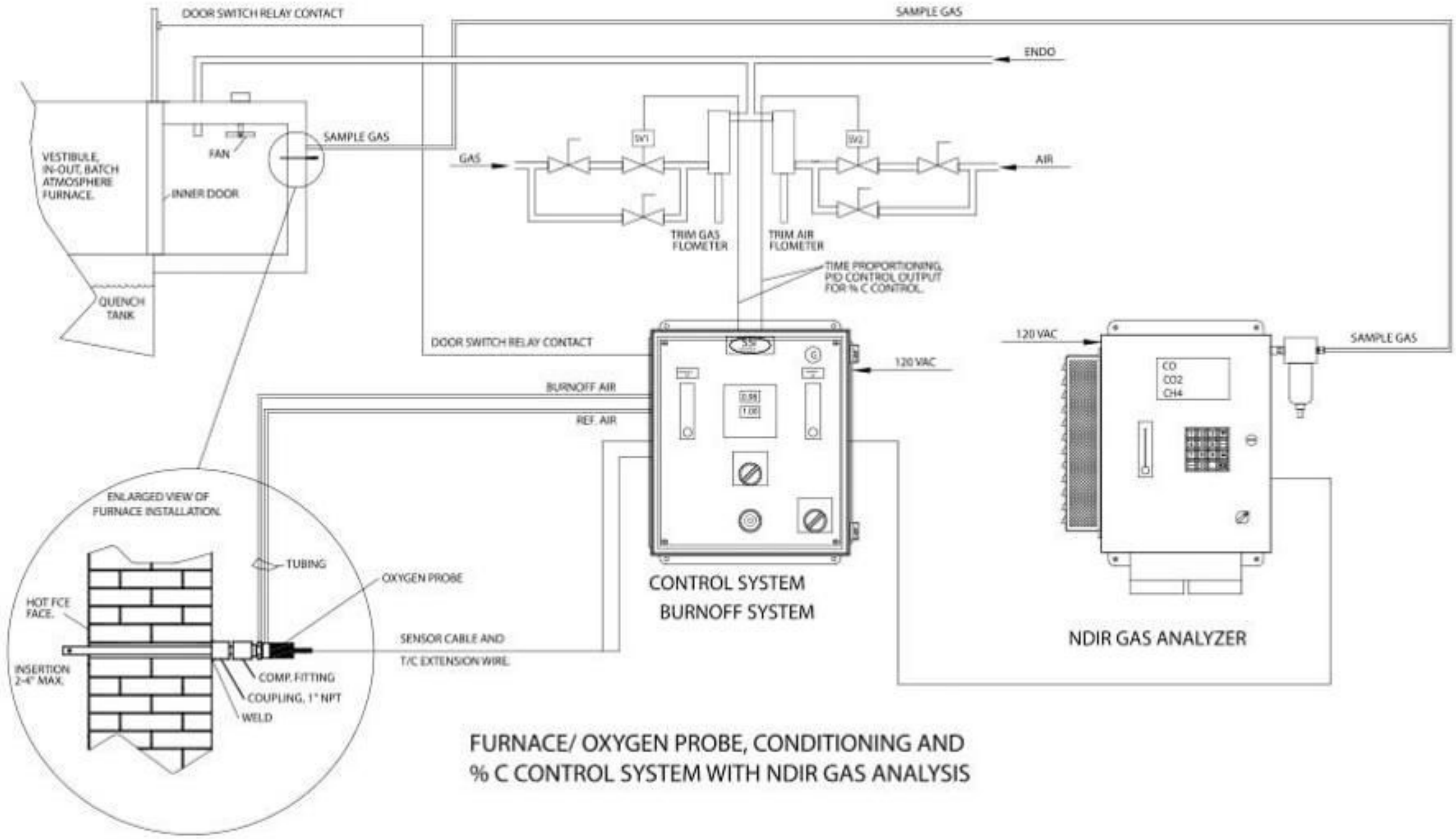

Fig. 2. Combination furnace oxygen probe and infrared control
(courtesy of Super Systems Inc.)

Table 1. Benefits and limitations of manual and automated control[4]

	Manual Control Scheme	Automated Control Scheme
Benefits	• Lower up-front cost • Initial training is easier • Benefits of "manufacturing wisdom" of experienced line operators	• Significantly reduced need for operator intervention • Greater control precision • Ability to customize recipes and alarms • Ability to automate maintenance alerts • Significant cost savings possible over time due to reduced scrap and reduced rework • Reduced possibility of operator error • Opportunity to replace obsolete older equipment for which replacement parts may not be available or which may not be in compliance with industry requirements
Limitations	• Reduced ability to quickly respond to possible deviation conditions • Higher amount of scrap and rework • Much higher possibility of operator error • Limits on alarm complexity	• Higher up-front cost • Reduced ability to apply "manufacturing wisdom" that comes with operator experience on the line • Additional knowledge/training requirements for operation

Table 2. Recommended burnout frequency[5]

Atmosphere Type	Frequency of Sensor Burnout (hours)	Duration of Sensor Burnout[a] (seconds)	Flow of Burnout Air, m^3/h (cfh)
Neutral	24	90	0.28 (10)[b]
High carbon	8 to 12	90	0.28 (10)[b]

Notes: [a]Recommended burnout time. Maximum time should not exceed 120 seconds before allowing the system to re-stabilize. [b]Minimum recommended flowrate.

It is good practice never to exceed 90 seconds of air addition at any one time to avoid overheating the tip of the probe. A consistent way to verify a correct burnout is to monitor the millivolts of the carbon controller during the burnout phase. If a proper burnout is taking place, the output will drop below 200 millivolts. This can also vary based upon the circulation in the furnace and the probe placement.

A possible side effect of extended burnout duration is oxidation of the tip of the sensor. This problem can manifest itself in higher-than-normal millivolt values over the remaining life of the sensor, which will require a lower CO-factor setting for the same calculation of carbon potential. Consideration should be made for the duration of the burnout based upon the carbon level in the furnace.

The current trend is to use oxygen probes in combination with three-gas analysis (CO, CO_2, CH_4) equipment (Fig. 2) to calibrate the probe to a known CO value and to monitor the amount of free hydrocarbon gas in the furnace atmosphere.

Measuring an Endothermic Atmosphere in a Furnace[5]

Carbon (oxygen) probes, shim stock or carbon analyzers and dew point are typically used to measure the carbon potential of the endothermic atmosphere in a heat-treatment furnace. An infrared analyzer can also be used. The CO_2 value – similar to water vapor and oxygen – varies based on furnace temperature and carbon potential but will typically lie between 0.25 and 0.50%. Multiplying the CO_2 value by 100 can roughly approximate the dew point (in ˚F). Charts and tables can also be used and are available from most manufacturers.

If a radiant tube is leaky in a gas-fired furnace, the leak (oxygen and moisture) will dilute the atmosphere carbon potential in the heat chamber when the furnace goes to high fire. As a result, the carbon controller will compensate by calling for more enriching gas to increase the carbon potential coincidental with high fire. A “saw-tooth” trend appears on the recorder. Setting the control to low fire will eliminate the saw tooth if the tube is cracked and leaking.

Oxygen probes and infrared analyzers are often used in conjunction when monitoring and controlling a nitrogen-methanol atmosphere. Using NDIR, values between 14-28% CO have been reported (target 20%). CO_2 values typically range from 0.10-0.80%, and free methane can be as high as 8% depending on the carbon setpoint. All testing should occur without hydrocarbon (e.g., natural or propane) gas additions. For nitrogen/methanol systems, the CO percentage is largely determined by furnace temperature and the relative flows of nitrogen and methanol.

Measuring an Endothermic Atmosphere in a Generator[5]

Another popular trend, especially in the control of endothermic gas generators, is to use an oxygen probe in combination with a dew-point analyzer.

Using an oxygen sensor to measure and control the dew point in an endothermic generator is done either in-situ, in a thermal well or by use of an oxygen probe with an integral thermal well/sheath arrangement. The millivolt signal can be converted to a dew-point value or, in some of the more advanced dew-point control systems, by calculation. This calculation uses the millivolts generated by the probe, the hydrogen factor of the controlling instrument and the temperature of the oxygen sensor. The temperature is required for the calculation, but the dew point of the gas is not temperature-dependent.

Oxygen sensors are often run at 1040˚C (1900˚F), although they will provide the same reading when operating at 815˚C (1500˚F). The generator gas exiting the retort is sent through a heat exchanger to freeze the composition. As long as the sensor is accurately measuring the millivolts of the gas, the temperature of the sensor can be as low as 593˚C (1100˚F).

Changes in sensor location have occurred over the years. Initially, a sensor would be mounted on the top of a retort in an air-cooled, fabricated fixture to measure the oxygen. Then a ceramic reheat well mounted through the sidewall of the generator was used. Now a modified sheath with an integral reheat well makes the installation much easier. The sheath and the integral well are aluminized prior to assembly so that the nickel in the sheath material does not react with the endothermic gas. This is especially important between 705˚C and 480˚C (1300˚F and 900˚F) where, over time, the endothermic reaction will reverse if nickel is present.

Lambda-style probes like those used in an automobile engine are also used in generator applications. While less expensive, they often lack the long-term stability of the zirconia technology and must be re-oxidized to avoid drift.

Next Time: In Part Two we learn more about measuring and controlling furnace atmospheres.

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Furnace Atmosphere Control Methods (Part 2)

How can one ever forget the smell of burning shoe leather? In the early 1970s, The Doctor, with the soles of his shoes literally melting away, was working atop a pusher carburizer to change one of those newfangled oxygen-probe sensors.

It was summer. It was hot. It was humid. It was Iowa. The job had to be done every few hours or so – such was the life expectancy of an oxygen probe in those days. The salesman working next to me had it tougher, though. He was wearing a three-piece suit!

Throughout his career, The Doctor has been in the right place at the right time in history more times than not. Such was the case here – to be present at the dawn of the introduction and commercialization of the oxygen probe into the heat-treatment industry. As the phrase goes, we've come a long way, baby. Let's learn more.

Table 1. Typical field data for an operating endothermic gas generator[1]

Constituent	1ST Quarter	2nd Quarter	3rd Quarter	4th Quarter
% CO	19.02	19.66	19.32	19.21
% CO_2	0.260	0.252	0.254	0.257
% CH_4	0.07	0.08	0.09	0.09
Generator dew point, °C (°F)	4°C (+39°F)	4°C (+39°F)	4°C (+40°F)	4°C (+39°F)
Dew point at furnace inlet, °C (°F)	3°C (+37°F)	3°C (+37°F)	3°C (+37°F)	3°C (+37°F)
Zonal dew point (Z1 - Z4),°C (°F)[3]	4.5°C to 5.5°C (+40°F to +42°F)	4.5°C to 5.5°C (+40°F to +42°F)	4.5°C to 5.5°C (+40°F to +42°F)	4.5°C to 5.5°C (+40°F to +42°F)

Notes: [1]85 m^3/hour (3,000 cfh) output; [2]Natural gas feedstock; [3]Neutral hardening

Maintenance Tips for Sampling Systems

Certain other types of furnace-atmosphere measurement and control devices require pulling a gas sample from the furnace. Preventive maintenance programs are common to all heat-treatment operations, but nowhere is it more important than for gas-sampling systems.

By using existing inputs and outputs on controllers and PLCs, operators can set specific timers and counters, which are extremely useful when determining when certain maintenance tasks should be planned or considered. With an

Table 2. Typical dew-point levels

Dew point, ˚C (˚F)	Water vapor (ppm)
+8 (+46)	10,590
-4 (+25)	4,320
-18 (0)	1,240
-40 (-40)	127
-68 (-90)	3.4

Note: 1% moisture = 10,000 ppm water vapor = +4.4°C (+40°F) dew point

established baseline, quick evaluation of real-time versus historical information can identify problem situations. Taken a step further, notification software can be used to automate e-mails and text messages to key personnel.

Here are several items and a few tips to keep sampling systems running trouble-free.

Sampling Lines

- Install, inspect and replace the line and analyzer filters as necessary.
- Verify sample flow (many analyzers are flow-dependent).
- Use flowmeters at the furnace sample port or at the analyzer (or both).
- Provide a way to clean out the sample port (i.e., rod out if carbon buildup occurs).
- Do not over-pressurize/backpressure the analyzer (many cells are flow- and pressure-dependent). Install metering valves if necessary.
- Purge the sample lines and analyzer with nitrogen when not in use.

Analyzers

- Calibrate (i.e., zero or span) the infrared and thermal-conductivity analyzers with nitrogen and a span gas (typically from a cylinder filled with a gas of known composition).
- Span with outside air or a moisture generator.
- Span with lithium-chloride or potassium-chloride salt solutions.
- Some units (Alnor® dew pointers) require factory calibration only.

Oxygen Probes

- Check surface-carbon values against shim stock.
- Measure temperature near the probe.
- Measure and correct for CO content of the furnace atmosphere.
- Confirm reference air and burnout air are being supplied unimpeded.
- Confirm proper probe location and insert depth.
- If hot insertion or removal is required, follow manufacturer's recommendations with respect

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Fig. 1. (a) Portable three-gas analyzer, (b) Portable dew point analyzer (courtesy of Super Systems, Inc.)

to prescribed speed of movement of the probe. Slower is always better.

Infrared (NDIR) Analyzers

Infrared analysis uses light in the infrared spectrum to analyze a gas sample and determine the percentage of each constituent in the furnace atmosphere. Single gas (carbon monoxide) or multiple (3-8) gas analyzers are used to detect these percentages. The amount of carbon dioxide in the furnace is an indirect way of measuring the carbon potential of the atmosphere, and charts/tables can be used to correlate CO_2 readings with dew-point or millivolt signals from oxygen probes. For example, the CO_2 value – similar to water vapor and oxygen – varies based on furnace temperature and carbon potential but will typically lie between 0.25 and 0.50%. Multiplying the CO_2 value by 100 can roughly approximate the dew point (in Fahrenheit).

Table 3. Dew point vs. surface carbon at selected temperatures

Dew point, °C (°F)	815°C (1500°F)	870°C (1600°F)	925°C (1700°F)
-1.1 (+30)	1.10	0.80	0.55
+4.4 (+40)	0.85	0.60	0.40
+10 (+50)	0.60	0.40	0.27

Today, three-gas infrared analyzers (Fig. 1a) are popular and used to monitor the atmosphere produced by generators (Table 1) and furnaces. The analyzers work on the principle that individual gases absorb infrared radiation of very specific wavelengths. The amount of absorption increases with gas concentration. The unit operates under the principle that a gas sample passes through a cell where a light emits infrared energy of known wavelength. The sensor converts measured infrared energy into an electrical signal. These values are usually compared to the values obtained with a reference gas. Infrared analyzers are known for their fast response and are easily calibrated.

Fig. 2. Typical sample-port configuration on a continuous mesh-belt furnace

Dew Point

Dew point is defined as the temperature that water vapor starts to condense. In simplest terms then, a dew-point analyzer (Fig. 1b) measures the amount of water vapor present in the furnace atmosphere (Table 2). This information can then be used to determine the carbon potential of the atmosphere (Table 3). It is considered an indirect-measurement technique if it involves pulling a gas sample from the furnace into the instrument.

If performed properly, dew point is a simple and accurate atmosphere measurement technique and indicates the condition inside the atmosphere generator or heat-treat furnace. The sample port (Fig. 2) should keep the sample line free of carbon and include a way to open and close the gas flow (soot). It should also include the capability to filter and cool the gas sample. The port should be designed in such a way as to keep the port open (i.e., the ability to “rod out” the line).

Dew point will help tell you if the reaction is stable or unstable (constant dew point or changing dew point over time). It can tell you when the catalyst bed in your endothermic gas generator is starting to soot and if there is a water leak, an air leak or non-uniformity (“breathing”) of the atmosphere inside your furnace.

The types of dew-point analyzers available include capacitance sensors, chilled mirrors and older-model fog chambers (Alnor®, Dew Cup). Condensation is a problem for all dew-point devices if the sample temperature is less than the dew point of the gas.

One solution is to heat trace the sample lines. Also, if the ambient temperature exceeds 40°C

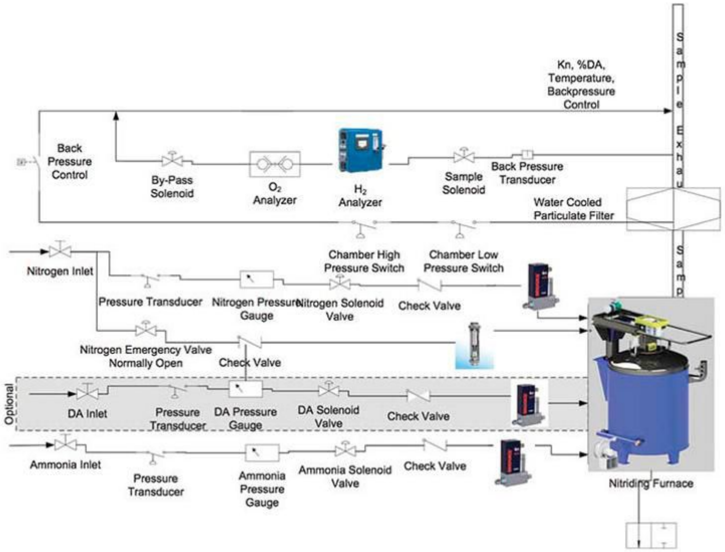

Fig. 3. Nitriding control scheme (courtesy of Super Systems, Inc.)

(105°F), as it often does in heat-treat shops, many dew-point analyzers will not give consistently accurate readings unless special precautions are taken (such as keeping the unit in the office and bringing it out on the shop floor for only a short period of time).

Interpretation of Data

In order to interpret furnace-atmosphere data correctly, it is important to collect data in real-time. However, it is of critical importance to understand the exact furnace operating conditions at the time the data was collected (e.g., zone temperatures and gas flows, furnace pressure, exhauster settings, fan rotation and speed, etc.).

In more than one instance, data was being analyzed and adjustments made based on improper sample port locations, instrumentation that was not properly calibrated, filters that were dirty and/or sample ports not extending fully into the furnace chamber. Mistakes such as these can be devastating to productivity, quality and equipment.

Process Control

In its basic form, process control applied to heat treatment is controlling temperature to maintain a desired setpoint. Process control can then be extended to other variables and in its most complex form to a lights-out operation where all mechanical, electrical and control functions are being handled by instrumentation. Here are some simple examples.

Generator Control

For gas generators, maintaining the quality of the gas (i.e., temperature, gas composition, dew point) using microprocessor controls and sensors to regulate the addition of enriching gas or dilution air is key. Control systems can incorporate fuel injection, which performs the mixing and control

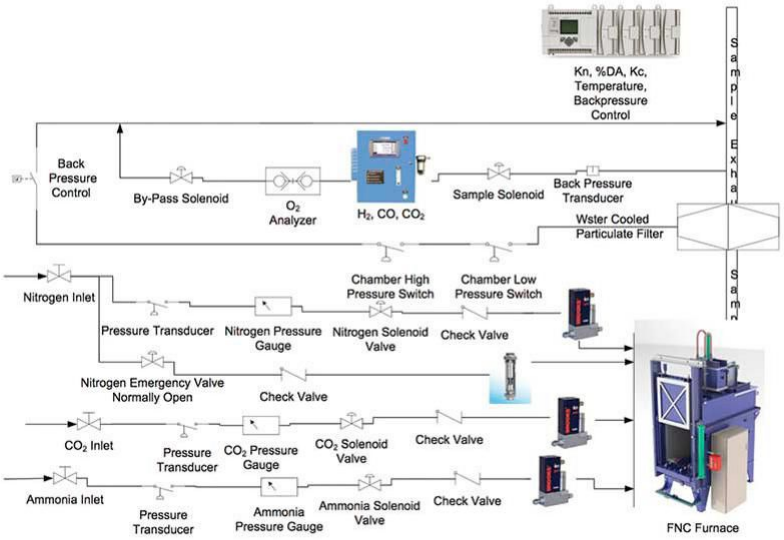

Fig. 4. Nitrocarburizing control scheme (courtesy of Super Systems, Inc.)

in one package with very little adjustment or maintenance required.

Carburizing Control

Carburizing requires precise management of the atmosphere composition to achieve the desired metallurgical results in the parts being processed. This includes such items as heat-up and soak times, temperature, carbon potential (single or boost /diffuse), cool down and quench.

The carburizing atmosphere is typically controlled by use of an oxygen probe. The carbon algorithm (using sensor millivolts and furnace temperature) used when determining carbon potential available in the furnace atmosphere assumes a consistent gas composition of the natural gas coming into the building. The equation also assumes a known CO value. If the composition of the natural gas or air changes, it not only influences the quality of the endothermic gas but also changes the amount of additive or enriching gas. The calculation of surface carbon from the millivolts and temperature monitored by the probe could be off. The automation component comes in when the calculation in the controller is automatically adjusted based on the three-gas calculation of carbon potential, thus providing a more accurate reading of carbon. This is commonly referred to as IR compensation.

Periodic verification of the process is typically performed using dew point, shim stock, shim coils or portable gas analyzers. If possible, shim stock (the only "true" test of carbon available in the furnace atmosphere) should always be used for verification.

Nitriding Control

Nitriding is another process that requires precise management of its variables. To achieve this, multivariable control systems (Fig. 3) monitor

Table 4. Relationship between dissociation and nitriding potential (K_N)

Percentage dissociated ammonia	Nitriding potential, K_N
1.33	986.67
2.67	344.13
6.67	83.48
13.33	27.41
20.00	13.77
26.67	8.20
33.33	5.33
40.00	3.65
46.67	2.58
50.00	2.18
53.33	1.84
60.00	1.33
66.67	0.94
73.33	0.65
80.00	0.43
86.67	0.25
93.00	0.11
100.00	0.00

temperature, atmosphere, nitriding potential, flow rate and pressure to provide both safe and precise control of compound (i.e., white) layer and diffusion-zone variations in material. Load and furnace types are controlled automatically as well.

Automated control for both nitriding and nitrocarburizing is based on temperature, time, flow and measurement of hydrogen, oxygen, carbon monoxide (CO) and carbon dioxide (CO_2). In nitriding, hydrogen is the process variable that must be measured as a result of the reaction of NH3 during dissociation.

It is possible to define the material, group and class. Determination of the desired K_N and K_C (Equation 1, Tables 4-5) based on the required epsilon layer and allowable porosity can be found in AMS 2759/12 (latest revision).

$$K_N = \frac{p(NH_3)}{p(H_2)}$$

where: p = gas partial pressure

Ferritic Nitrocarburizing (FNC) Control

FNC control (Fig. 4) is similar to nitriding but with the addition of a carbon-bearing gas. When controlling FNC, hydrogen is one of a number of variables that must be considered. With the use of CO_2 (common in pit-type furnaces), there is a reaction with hydrogen that produces CO to create carbon activity, K_C (Equation 2).

$$K_C = \frac{p(CO_2)p(H_2)}{p(CO)p(H_2O)}$$

where: p = gas partial pressure

CO and CO_2 are both reliably measured with infrared technology. It should be noted that the infrared-cell technology must be compatible with NH_3 and water for the FNC process. Oxygen is measured using zirconia- or lambda-probe technology. As with nitriding, it is important to meter the gas flows when using nitrogen and any other diluents. When using endothermic gas with 40% hydrogen present, it is important to meter and compensate for the gas flow and hydrogen content. With these variables being considered, K_N and K_C are calculated to allow for more precise control of the nitrogen and carbon compound layers.

Sample lines and filters to the H_2 analyzer and oxygen cell must be maintained on a regular basis. Calibration of the instrumentation and analyzers must be completed at manufacturer-required intervals. In addition, the mechanical assembly of the system must take into account the need for filtration and ease of maintenance. For example, sample systems must be supplied with drip legs in order to remove any moisture that might be generated during the pre-oxidation process.

When nitriding stainless steel and using a pre-activation process (typically involving polyvinylchloride), filtration and heat tracing must be included in the exhaust piping. Lines and filters must be cleaned for each run. Filtration systems should be designed with pressure-indication and automatic-bypass modes. FNC processes, in particular, are more challenging due to the formation of ammonium carbonate in the exhaust.

Table 5. K_N and K_C values

Material	Process Temperature[a]		Process Time (hours)	Class 1				Class 2			
				Not exceeding 15% of thickness of white layer				Above 10% but not exceeding 50% of thickness of white layer			
				K_N		K_C		K_N		K_C	
	°F	°C		min	max	min	max	min	max	min	max
Group 1[b]	1040 1075	560 579	3 - 6 2 - 5	2.13 1.50	2.41 1.60	0.57 1.10	0.69 1.22	2.48 1.68	2.68 1.78	0.49 0.86	0.54 0.94
Group 2[c]	980	527	6 - 30	4.51	5.55	0.16	0.24	6.03	7.10	0.09	0.13
Group 3[d]	1060	571	3 - 10	1.82	2.10	0.76	0.99	2.22	2.64	0.48	0.68

Notes: [a]Temperatures shown are not firm requirements. Once a temperature is selected, however, both potential values shall be within specified limits for the given temperatures. [b]Group 1: HLSA, carbon steels; [c]Group 2: 4140, 4340, Nitralloy 135M; [d]Group 3: Cast iron

Summary

While manual control methods are highly effective when properly employed and continue to be used throughout the heat-treatment industry, the future lies in the precision and customizability of automated atmosphere-control methods. By utilizing specialized process controllers and software, these systems help enhance productivity through real-time monitoring and control, leading to reduced scrap and freeing up operators for other tasks.

Automated systems also form the basis for electronic data acquisition that improves process traceability and makes it much easier to analyze data in making decisions for improving processes. Preventive maintenance programs often grow from automated methods, reducing unplanned downtime. Instrumentation upgrades associated with automation bring many additional benefits, including:

- Increased focus on operation-specific features, such as carburizing, vacuum heat treating and nitriding.
- Recipe control and management, leading to fewer entry points and operator requirements.
- Potential for greater operating ranges due to the ability to manage multiple parameters at the same time.

What will the future hold with respect to atmosphere control? Artificial intelligence and real-time load-monitoring devices seem to be only a step away. As technology improves, processes improve. While the initial cost of control upgrades may be seen as significant, quality demands and assurance of absolute process repeatability make them a necessity.

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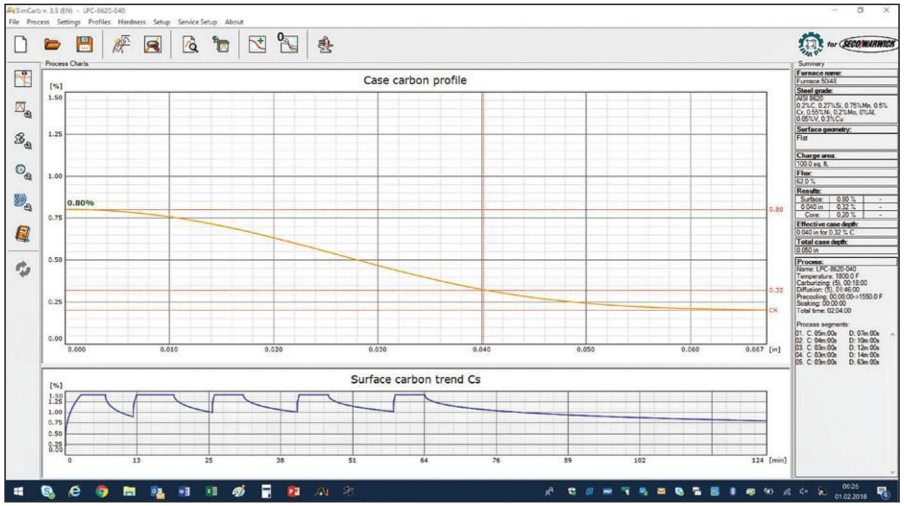
Simulation Software (Part Two: Low-Pressure Carburizing)

The acceptance of low-pressure (vacuum) carburizing (LPC) technology in manufacturing is due in large part to the ease of recipe development and modification made possible by the use of simulators designed specifically for this purpose. Let's learn more.

Simulator Basics

Today's LPC simulators are highly accurate, often being able to develop the proper recipe without test loads for most common steels (this is especially useful for the commercial heat-treatment industry). Custom steels or chemistry modifications often require only one or two test loads prior to starting production runs. Inputs for simulator calculations usually include material type, part geometry, carburizing temperature, hardening temperature, target effective case depth, surface area of the load and targeted final surface carbon content.

Based on this input data, a carburizing recipe is established complete with the number and duration of hydrocarbon gas injection steps during the boost stage and the number and duration of evacuation or gas dilution steps during the diffusion stage. Gas flowrates are precisely determined and controlled via mass flowmeters. The program output is displayed graphically, illustrating the carbon profile as a function of depth as well as the surface carbon concentration at various steps in the process (Fig. 1).



Furnace name:
Furnace 50/48
Steel grade:
AISI 8620
0.2%C, 0.27%Si, 0.75%Mn, 0.5% Cr, 0.55%Ni, 0.2%Mo, 0%Al, 0.05%V, 0.3%Cu
Surface geometry:
Flat
Charge area:
100.0 sq. ft.
Flux:
62.0 %
Results:

Surface:	0.80 %	-
0.040 in	0.32 %	-
Core:	0.20 %	-

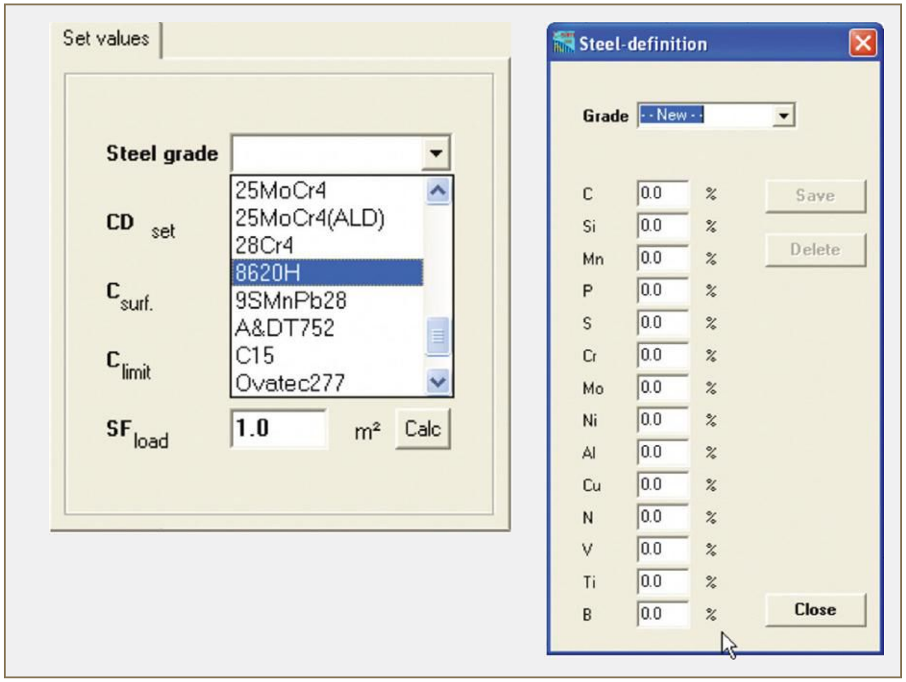
Effective case depth:
0.040 in for 0.32 % C
Total case depth:
0.050 in
Process:
Name: LPC-8620-040
Temperature: 1800.0 F
Carburizing: (5), 00:18:00
Diffusion: (5), 01:46:00
Precooling: 00:00:00->1550.0 F
Soaking: 00:00:00
Total time: 02:04:00

Process segments:

01.	C: 05m:00s	D: 07m:00s
02.	C: 04m:00s	D: 10m:00s
03.	C: 03m:00s	D: 12m:00s
04.	C: 03m:00s	D: 14m:00s
05.	C: 03m:00s	D: 63m:00s

Fig. 1. Typical output screen from an LPC simulator (courtesy of SECO/Vacuum Technologies)

Material selection is typically done either from a lookup table of common steels (U.S. and/or European grades) or through the input of a custom chemistry. This chemical composition data (often in conjunction with Jominy data) is also used by some simulator packages to help determine proper quench response.



CD set

C surf.

C limit

SF load 1.0 m²

Grade - - New - -

C 0.0 %
Si 0.0 %
Mn 0.0 %
P 0.0 %
S 0.0 %
Cr 0.0 %
Mo 0.0 %
Ni 0.0 %
Al 0.0 %
Cu 0.0 %
N 0.0 %
V 0.0 %
Ti 0.0 %
B 0.0 %

Fig. 2. Recipe input – material selection (courtesy of ALD Vacuum Technologies, GmbH)

These process simulators can also provide anticipated hardness profiles as a function of depth from the surface. It should be noted that when a carbon profile is converted into a hardness profile, part geometry and quenching variables may skew the final result.

One of the simplest ways in which to determine the effectiveness of a particular manufacturer's simulator is to review various material choices available and ask if the output recipe has been confirmed by extensive empirical trials. In other words, have loads been run and results analyzed via testing or just through scientific calculation?

Part geometry is usually limited to simple shapes (cylindrical, spherical, rectangular, etc.) rather than asking the user to input sophisticated geometries. On the other hand, calculation of load surface area must be more accurate (within about ±10%) so that the proper amount of hydrocarbon gas is added. Surface carbon values are lower than their atmosphere counterparts, 0.70-0.75% C being typical.

Outputs for simulator calculations usually include recipe/program, carbon profile, carbon flux/mass flow and surface carbon content.

While the final surface carbon content always corresponds to the setpoint value, it is important to recognize that most simulators estimate the carburizing case depth within about ±5% of this value. For this reason, an initial trial run is always recommended. It should also be noted that these simulators have now become more and more precise and should be able to produce specification requirements with little testing. Once a process has been successfully simulated, repeatability of results is almost a foregone conclusion (provided one has control of the equipment-induced variability).

Most simulator packages are run remotely on an office computer, and process recipes are uploaded to the furnace with a few keystrokes. They can also be manually input through a setup Wizard following instructions and dropdown menus (Fig. 2).

Tweaking the Cycle

In some instances, the data obtained by running test parts indicates that the recipe needs to be modified to fine-tune the results. Common reasons for this include issues with:

- Carbide formation and/or carbide necklacing. This indicates that either the surface carbon content is too high during the boost portions of the cycle, the diffusion time was insufficient, the soak time at temperature was too short or the final hardening temperature was too low. Possible recipe changes include either shortening the boost times or reducing the number of boost steps. Additionally, adding more diffusion steps or increasing soak times and/or temperature will eliminate the problem.
- Retained austenite. One possible reason for this is that the surface carbon content of the steel in question is too high, so lengthening the last diffusion step will help reduce the final surface carbon value. Another cause is direct quenching from carburizing temperature, so reducing the temperature before quenching is recommended.
- Carbon content too high/low. The surface area of the load should be carefully calculated to see if the gas flowrate value is too high/low. Some manufacturers offer a limited number of flowrates (e.g., high, normal, low) that are not ideally matched to the actual surface area present, resulting in a higher/lower surface carbon content than anticipated.
- Surface pitting. This indicates that the surface carbon was too low during processing and should be increased. Pitting on gear teeth is a common example.
- Over-carburizing. In areas where the geometry influences carbon absorption (such as the tips of gear teeth), reducing the flowrate during the boost stage or adding more but shorter-duration boost steps helps minimize this effect.
- Part distortion. There are numerous factors that influence part distortion, including the type and hardenability of the steel, the type of quench medium (oil or high-pressure gas) and the final hardening temperature, to name a few. One wants to select a material with the lowest hardenability that will achieve the required mechanical properties, including surface and core hardness. Quenching from

a lower temperature may also help, and the recipe should be adjusted so that there is enough time for the core/surface temperature of the parts (load) to equalize before quenching.

Universal LPC Simulation Software

Until recently, LPC simulators were captive to the type of equipment being purchased. The principal limitation was that (as designed) the software would work only on a particular manufacturer's furnaces running one type of hydrocarbon gas in a defined pressure range. In essence, the OEMs had proprietary software designed to work only with their systems. Today, at least one manufacturer has developed LPC simulation software that will produce recipes for all types of carburizing furnaces running all of the common hydrocarbon gas mixtures in use (e.g., acetylene, acetylene + hydrogen, actelyene + nitrogen and acetylene + ethylene + hydrogen).

Summary

The inherent advantage of low-pressure carburizing is its consistency, repeatability and flexibility. As more and more LPC furnaces find their way into the heat-treat shop, the need for and convenience of a universal simulator will become paramount. One should expect to find, as a minimum, the following features on any LPC simulator:

- An extensive materials database
- The capability of creating and exporting a heat-treatment recipe (i.e., a sequence of cycle steps) showing the carbon profile (including surface, intermediate and limit carbon) as well as deviation from set values
- Save and load recipe functions
- The ability to edit recipes, add or delete alloys, add custom chemistries and change individual elements in the standard materials list (such as carbon content)
- Graphic displays for carbon and/or hardness profiles, carbon percentage at various stages in the process, and carbon flux and/or mass flow in addition to the process recipe inputs. For ease of use, a zoom function, scalable axes, historical record retrieval and the ability to save data to external files should be included.

If you are or will be using LPC in the future, be sure the equipment has or comes with one of these state-of-the-art devices.

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Simulation Software (Part Three: Nitriding, Nitrocarburizing)

Last but certainly not least, as the saying goes, is our discussion of gas nitriding and nitrocarburizing process simulators and how they are used to predict and control these case-hardening processes. Let's learn more.

Today, these simulators are available from several original equipment manufacturers and academic institutions for the purpose of determining the nitriding process parameters (i.e., cycle recipes) required to obtain a given case depth, to predict the compound-layer composition, and to anticipate both the final surface and core hardness as well as the hardness distribution throughout the case.

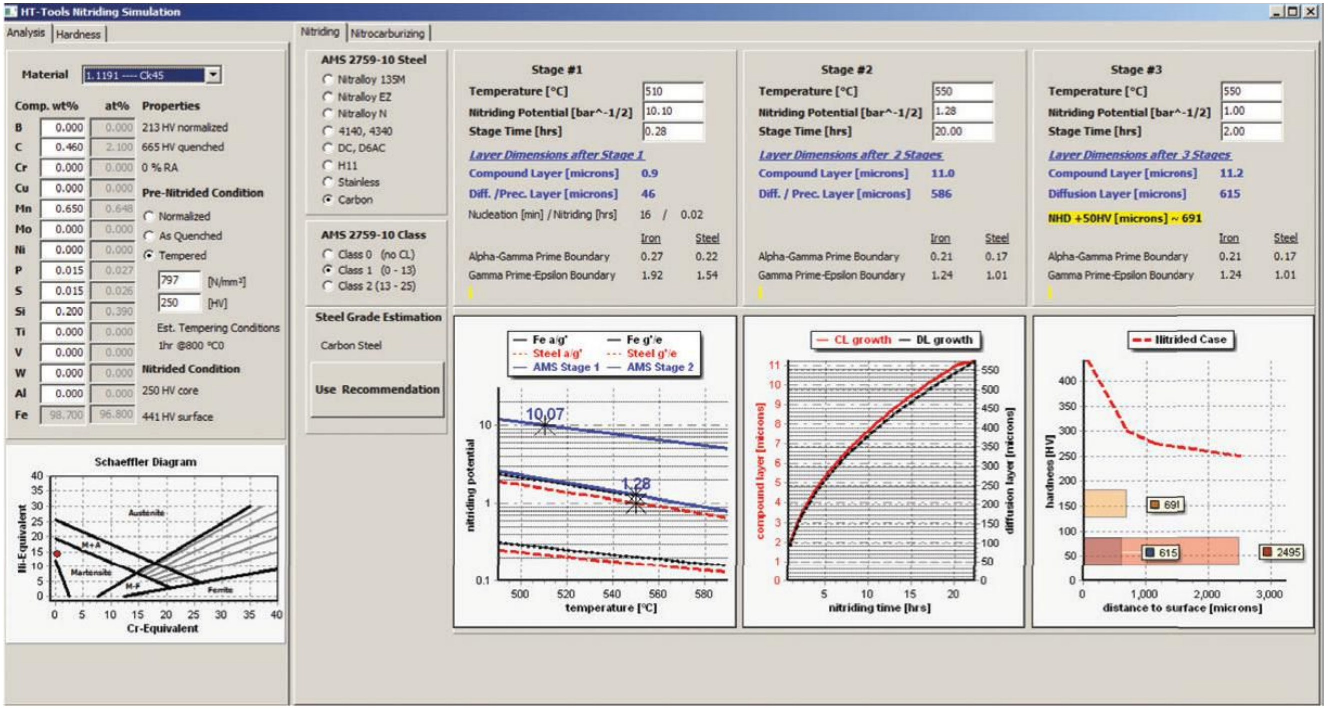
Key Challenges[2]

Nitriding simulations are strongly influenced by two preconditions: material composition (i.e., the effect of alloying elements on nitriding activity, solubility, phase boundaries and diffusion coefficients) and prior microstructure (i.e., core hardness and microstructure impacting the final surface-hardness increase, final core hardness and the final hardness distribution) produced by prior heat treatments such as annealing, normalizing, austenitizing and quenching, or quench-and-temper operations.

One of the reasons that the prior microstructure is so important is that simulators need to determine the amount of nitride formers not tied up as carbides. Nitriding processes are ideally performed on quenched-and-tempered steels. Tempering is performed at a temperature of at least 10˚C (50˚F) above nitriding temperature. Simulators (and most specifications) typically estimate hardness distribution and case depth. Case depth is defined as core hardness plus 50 HV.

Simulators also need to determine hardness changes during nitriding. This is done by assuming that the softening effects that occur with increasing temperature are similar to tempering effects. Other factors that influence the simulator models (and involve an in-depth understanding of kinetics and thermodynamics) are nucleation, compound-layer growth and composition, diffusion/precipitation-layer growth and final hardness distribution.

These factors are incorporated into the output of the various simulators (Fig. 1) based on work by such noted individuals as Sun and Bell (nucleation



HT-Tools Nitriding Simulation

Analysis | Hardness

Material: 1.1191 ---- Ck45

Comp.	wt%	at%
B	0.000	0.000
C	0.460	2.100
Cr	0.000	0.000
Cu	0.000	0.000
Mn	0.650	0.648
Mo	0.000	0.000
Ni	0.000	0.000
P	0.015	0.027
S	0.015	0.026
Si	0.200	0.390
Ti	0.000	0.000
V	0.000	0.000
W	0.000	0.000
Al	0.000	0.000
Fe	98.700	96.800

Properties
213 HV normalized
665 HV quenched
0 % RA

Pre-Nitrided Condition
○ Normalized
○ As Quenched
● Tempered
797 [N/mm²]
250 [HV]
Est. Tempering Conditions
1hr @800 °C0

Nitrided Condition
250 HV core
441 HV surface

Nitriding | Nitrocarburizing

AMS 2759-10 Steel
○ Nitralloy 135M
○ Nitralloy EZ
○ Nitralloy N
○ 4140, 4340
○ DC, D6AC
○ H11
○ Stainless
● Carbon

AMS 2759-10 Class
○ Class 0 (no CL)
● Class 1 (0 - 13)
○ Class 2 (13 - 25)

Steel Grade Estimation
Carbon Steel

Use Recommendation

Stage #1
Temperature [°C]: 510
Nitriding Potential [bar^-1/2]: 10.10
Stage Time [hrs]: 0.28
Layer Dimensions after Stage 1
Compound Layer [microns]: 0.9
Diff. /Prec. Layer [microns]: 46
Nucleation [min] / Nitriding [hrs]: 16 / 0.02

	Iron	Steel
Alpha-Gamma Prime Boundary	0.27	0.22
Gamma Prime-Epsilon Boundary	1.92	1.54

Stage #2
Temperature [°C]: 550
Nitriding Potential [bar^-1/2]: 1.28
Stage Time [hrs]: 20.00
Layer Dimensions after 2 Stages
Compound Layer [microns]: 11.0
Diff. / Prec. Layer [microns]: 586

	Iron	Steel
Alpha-Gamma Prime Boundary	0.21	0.17
Gamma Prime-Epsilon Boundary	1.24	1.01

Stage #3
Temperature [°C]: 550
Nitriding Potential [bar^-1/2]: 1.00
Stage Time [hrs]: 2.00
Layer Dimensions after 3 Stages
Compound Layer [microns]: 11.2
Diffusion Layer [microns]: 615
NHD +50HV [microns] ~ 691

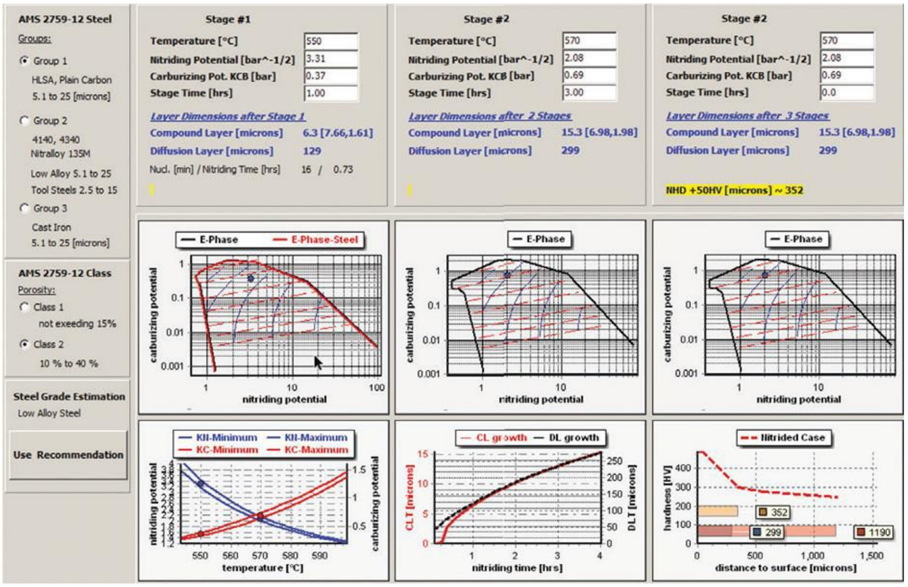
	Iron	Steel
Alpha-Gamma Prime Boundary	0.21	0.17
Gamma Prime-Epsilon Boundary	1.24	1.01

Fig. 1. Typical gas nitriding simulator output screen factoring in compound-layer composition and diffusion/precipitation-layer growth (courtesy of United Process Controls)

theory[4]), Hosseini, Ashrafizadeh and Kermanpur (compound-layer growth and composition[5]), Fick (diffusion modeling) and Kunze (precipitation layer growth[6]). While beyond the scope of this article, the reader is encouraged to review these papers to deepen their understanding of how these factors play a role in the inner workings of these simulators.

Ferritic Nitrocarburizing Simulators

Simulators for ferritic nitrocarburizing (Fig. 2) are typically adopted from their gas nitriding simulator cousins by looking at the iron-nitrogen-carbon diagram (Fig. 3) at a specific process temperature and adjusting the diagrams to match the steel in question by using appropriate activity coefficients.



AMS 2759-12 Steel
Groups:
● Group 1
HLSA, Plain Carbon
5.1 to 25 [microns]
○ Group 2
4140, 4340
Nitralloy 135M
Low Alloy 5.1 to 25
Tool Steels 2.5 to 15
○ Group 3
Cast Iron
5.1 to 25 [microns]

AMS 2759-12 Class
Porosity:
○ Class 1
not exeeding 15%
● Class 2
10 % to 40 %

Steel Grade Estimation
Low Alloy Steel

Use Recommendation

Stage #1
Temperature [°C] 550
Nitriding Potential [bar^-1/2] 3.31
Carburizing Pot. KCB [bar] 0.37
Stage Time [hrs] 1.00
Layer Dimensions after Stage 1
Compound Layer [microns] 6.3 [7.66,1.61]
Diffusion Layer [microns] 129
Nucl. [min] / Nitriding Time [hrs] 16 / 0.73

Stage #2
Temperature [°C] 570
Nitriding Potential [bar^-1/2] 2.08
Carburizing Pot. KCB [bar] 0.69
Stage Time [hrs] 3.00
Layer Dimensions after 2 Stages
Compound Layer [microns] 15.3 [6.98,1.98]
Diffusion Layer [microns] 299

Stage #2
Temperature [°C] 570
Nitriding Potential [bar^-1/2] 2.08
Carburizing Pot. KCB [bar] 0.69
Stage Time [hrs] 0.0
Layer Dimensions after 3 Stages
Compound Layer [microns] 15.3 [6.98,1.98]
Diffusion Layer [microns] 299
NHD +50HV [microns] ~ 352

Fig. 2. Typical ferritic nitrocarburizing simulator output screen based on considerations of the iron-nitrogen-carbon phase diagram (courtesy of United Process Controls)

Process Control

Control of a modern gas nitriding process (Eq. 1) is based on the modified Lehrer diagram[7] relating nitriding potential to the iron-nitrogen phase boundaries as a function of temperature.[11]

$$k_N = \frac{pNH_3}{p^{\frac{3}{2}}H_2} \quad (1)$$

Control of a modern nitrocarburizing process (Eq. 2) is based on the Fe-N-C control diagram, displaying $Fe_{2-3}NC$ epsilon (ε) phase depending on temperature, nitriding potential (K_N) and carburizing potential (K_C) as shown in Figure 3.

$$k_{CW} = \frac{pCO \cdot pH_2}{pH_2O} \qquad k_{CB} = \frac{pCO_2}{p^2CO} \quad (2)$$

Open Architecture – One Man's Opinion

The individual companies and research institutions that have invested considerable time, money and effort into developing these simulators should be complimented, but commercially available software packages running on desktop or laptop computers are needed for their wider adoption and use.

Summary

In this series of recent articles, we have introduced the idea to the heat-treat community that process simulators not only exist but can be used for highly accurate prediction of process recipes and to anticipate the metallurgical structure (and ultimately the performance characteristics) of the parts being run. Nitriding and nitrocarburizing simulators are no less capable than their carburizing counterparts.

Surveys of the heat-treatment industry conducted by ASM International in the late 1990s and early 2000s outlined a series of eight key industry goals, one of which was outcome prediction based on heat-treat modeling. The accomplishment of that goal is closer than ever. As

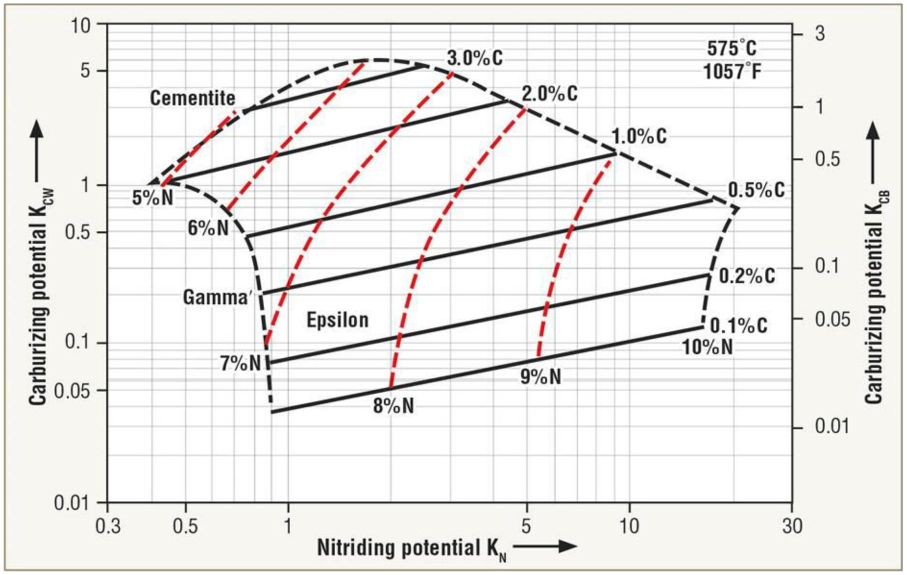


Fig. 3. Iron-nitrogen-carbon control diagram[12]

such, the heat treater needs to embrace and use these simulation tools in his/her daily work.

Acknowledgment

The writer – with great pride and humility – would like to acknowledge good friend Dr. Karl-Michael Winter for his contributions to this article; his unwavering commitment to advancing gas nitriding/nitrocarburizing technology throughout the heat-treatment industry; and his many lectures, papers and discussions over the years.

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Equipped with externally mounted 600 HP motor with Allen-Bradley Variable Frequency Drive (VFD) to provide controlled cooling with externally mounted 14,000,000 BTUs/hour primary heat exchanger and a 300,000 BTUs/Hr. post cooler to provide maximum quenching capabilities for various types of loads and large cross-sections. Furnace controls equipped with Honeywell HC900 and Wonderware Computer Control.

16" Cube of H-13 Steel (1160 Lbs.): Cool in 4-minutes from 1870°F to 740° F @ a rate of 282°F/Minute in 20 BAR of Nitrogen

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Technology Trends in Vacuum Heat Treating, Part One: Markets, Processes & Applications

Each of the common vacuum processes will be discussed in this and future articles in this series. The last decade has seen double-digit growth in the use of vacuum heat treating and increased vacuum market share throughtout the Americas (Fig. Nos. 1 - 5). Vacuum processing is growing more than any other technology, due in large part to the the demand for high-quality, precision and repeatability of part performance in ever-more sophisticated and demanding service applications. Let's learn more.

Annealing

Annealing treatments are undertaken primarily to soften a material, to relieve internal stresses and/or to modify the grain structure. These operations are carried out by heating to the required temperature and soaking at this temperature for sufficient time to allow the material to stabilize, usually followed by a slow cooling at a predetermined rate. The choice of vacuum annealing is primarily influenced by the cleanliness and high quality of surface finish (Fig. 6) that can be obtained relatively easily compared to controlled-atmosphere heat-treatment operations.

Copper and Copper Alloys

Annealing of copper alloys is normally performed to soften the material after work (strain) hardening and to retain bright surface finishes.

Stainless Steels

Processing of stainless steel components in vacuum furnaces is often specified not only because of the cleanliness of the finished product, but also because of the fast gas-quench capability in vacuum. Stainless steel grades are usually gas quenched in nitrogen for general commercial applications. However, austenitic steel grades stabilized with titanium or columbium (niobium) require argon or helium quenching to avoid sensitization (nitrogen pickup) that degrades the corrosion resistance, particularly for nuclear, medical and aerospace applications.

A number of different annealing methods – full, isothermal, subcritical – are commonly used for stainless steel. Austenitic stainless steels cannot be hardened by heat treatment, but they do harden by cold working. Annealing not only allows recrystallization of the work-hardened grains, it also places chromium carbides – precipitated at grain

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Fig. 1. Vacuum market in the Americas by industry

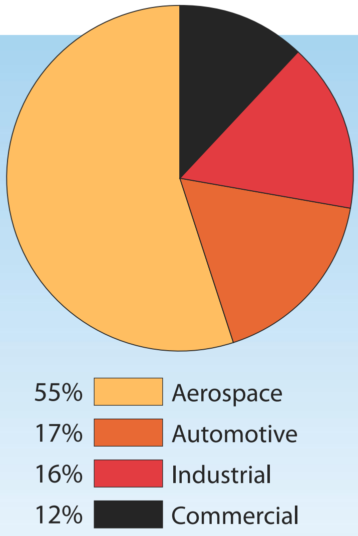

Fig. 2. Vacuum market in the Americas by process

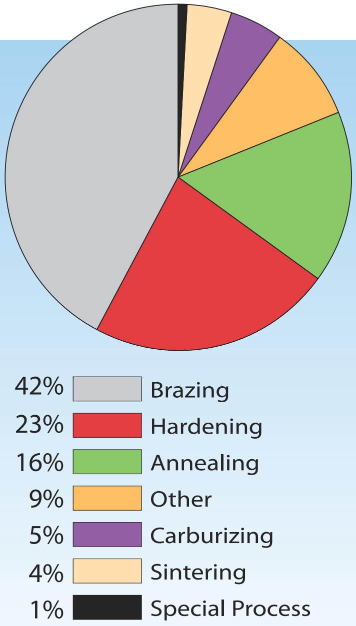

Fig. 3. Vacuum market in the Americas by segment

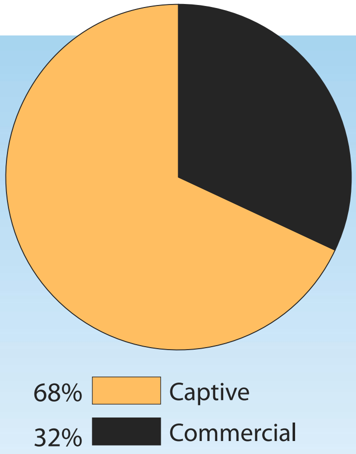

Fig. 4. Vacuum market in the Americas by equipment type

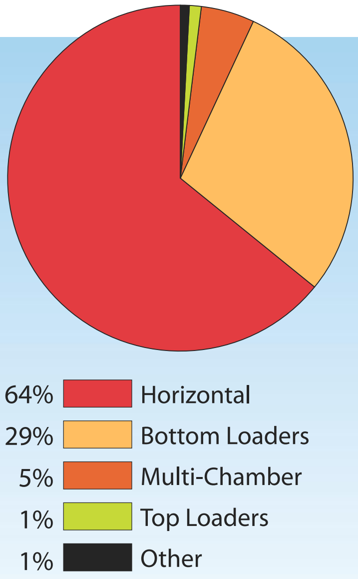

Fig. 5. Vacuum market in the Americas by technology

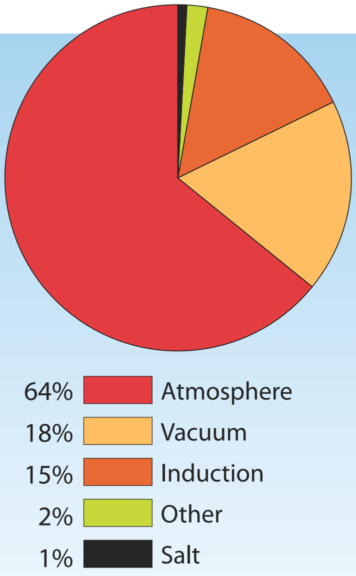

Fig. 6. Bright annealing of copper diodes for heat sinks

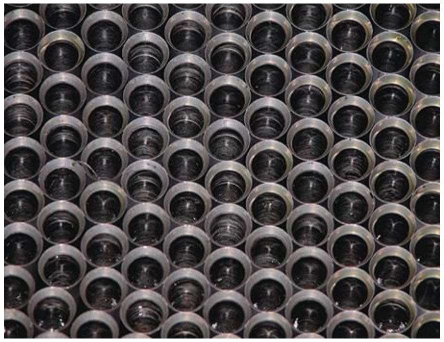

Fig. 7. Die-cutting punches after oil quenching in a horizontal batch vacuum furnace

boundaries – back into solution. Annealing can also be used for homogenization of castings or welds and to relieve stresses from cold working.

Time at temperature is often kept short to minimize surface oxidation and to control grain growth, which may lead to a surface phenomenon called "orange peel." Some chromium evaporation can take place during the annealing of stainless steels, but normally the amount lost is not significant because of the short time at heat and the slow diffusion rates of chromium in steel.

Annealing temperatures range from 630 – 900°C (1150 – 1650°F) for ferritic and martensitic stainless steels to above 1040°C (1900°F) for austenitic (stabilized and unstabilized) alloys. When fine grain size is desired the annealing temperature must be closely controlled.

Carbon and Low-Alloy Steels

These materials are only processed economically in applications where cleanliness of the products or the prevention of carburization or decarburization of the part surfaces is critical.

Tool Steels

Vacuum annealing is often used on tools that have been improperly hardened so that they can be reworked to meet required specifications on re-hardening. Annealing using other types of furnace equipment is impractical because all working surfaces of the tools might need to be reground due to intergranular oxidation or carburization/decarburization, thus losing the dimensional precision required.

Hardening by Oil Quenching

Oil quenching in horizontal (Fig. 7) or vertical (Fig. 8) vacuum furnaces is common using integral oil-quench designs. The design of the quench tank is similar to its atmosphere counterpart. Fixed- or variable-speed oil-circulation agitators or pumps are located on one or both sides of the tank, and internal baffles guide the respective oil flow around the load. The oil is commonly heated in the 50 – 65°C (120-150°F) range but with special oils can run at 135 – 175°C (275 – 350°F). Heaters control the temperature, and the oil is

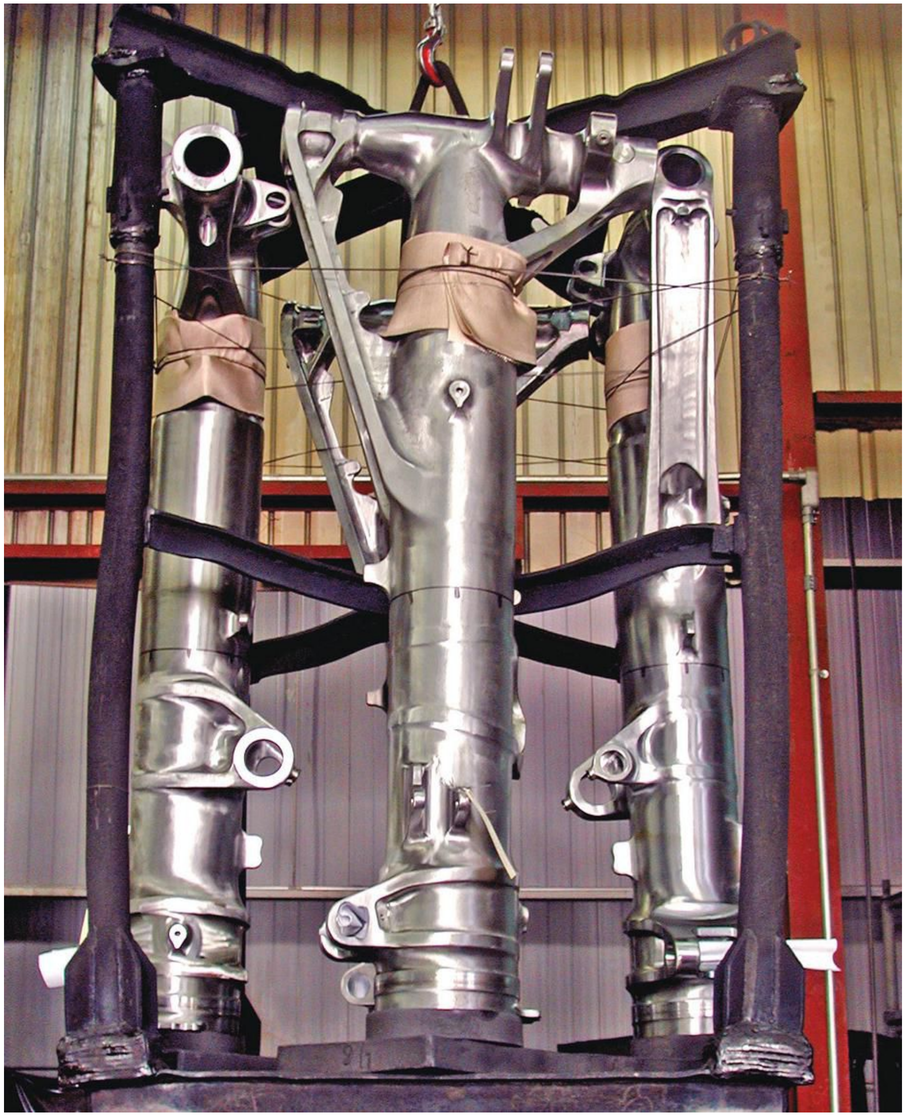

Fig. 8. Landing-gear load being assembled for hardening and oil quenching in a vertical vacuum furnace (Photograph courtesy of Vac-Aero International)

cooled via external oil cooler, usually employing air, for safety reasons.

A peculiarity of quenching in vacuum furnaces is that the low pressure above the oil causes standard quench oils to degas violently. The duration of this degassing process depends on the amount of air or nitrogen absorbed by the oil during the loading and unloading of the furnace. Vacuum oils have been created to minimize these problems. Oils that are not degassed properly have a worse quenching severity and produce discolored components.

Vacuum quench oils are distilled and fractionated to a higher purity than normal oils, which is important in producing the better surface appearance of quenched parts. In practice, the quenching in vacuum furnaces is frequently done with a partial pressure of nitrogen above the oil between 540 mbar (400 Torr) and 675 mbar (500 Torr).

It is well known, however, that a pressure increase just before initiating the quench also changes the oil-cooling characteristics. The pressure increase shortens the vapor-blanket phase, thus increasing the quench severity at high temperatures (in the pearlite-ferrite transformation range). On the other hand, it lowers the quench rate in the convective cooling phase (in the bainite or martensite transformation range). Thus, high partial pressures above the oil can be advantageous in producing full hardness on unalloyed or very low-alloy materials, whereas low pressures above the oil produce higher hardness and lower distortions on components made of alloyed steels.

Very low pressures (<50 mbar/<35 Torr) above the oil and very high quenching temperatures in the area of 1200°C (2200°F) can lead to carbon deposition and/or pick-up on the surface of parts. This is due to the thermal decomposition of the quench oil as has been experienced in hardening certain tool steels. The carbon originates from the fractionation of the oil vapor in contact with the hot surface of the load in the initial phase of the quench process. High nitrogen pressures (>150 Torr/>200 mbar) tend to reduce or eliminate this effect.

Hardening by Gas Quenching

Inert-gas pressure quenching in the range of 2–20 bar is the most popular form of quenching in vacuum furnaces. There is an interest in the use of hydrogen for cooling in the 25–40 bar range due to its extremely high heat-transfer rates. In gas quenching, part dimensional changes, although repeatable, are different than when quenching in oil. The trend today is to “dial in” the quench pressure. That is, use only the highest pressure required to properly transform the material. Recent changes in material chemistry and pressure quench design (e.g., alternating gas flows, directionally adjustable blades, variable-speed drives) has made this possible, and gas quenching is now used to produce full hardness in many traditional oil-hardening steels.

Flow rate and density of the cooling gas blown onto the surface of the load are important factors for achieving high heat transfer (high cooling) rates. In addition to high gas velocities, high gas pressures are needed to through-harden a wide variety of steel parts with appreciable dimensions. Calculations of the heat-transfer coefficient alpha (α) show that it is proportional to the product of gas velocity and gas pressure.

$$\alpha \approx (vp)^n \qquad (1)$$

where v is the gas velocity and p the pressure of the gas. The exponent n depends on the furnace design, the load and the properties of gas. It lies typically in the range of 0.6 – 0.8. The exponential behavior of the heat transfer makes it clear that the difference in the increase of heat transfer is considerable with the first few bars of pressure but decreases with increasing pressure. And the (negative) influence of equipment limitations can contribute to non-uniformity of cooling or the use of higher pressures or velocities that can contribute to increased distortion.

The critical transformation range for most steel is between 800°C (1475°F) and 500°C (930°F). Lambda

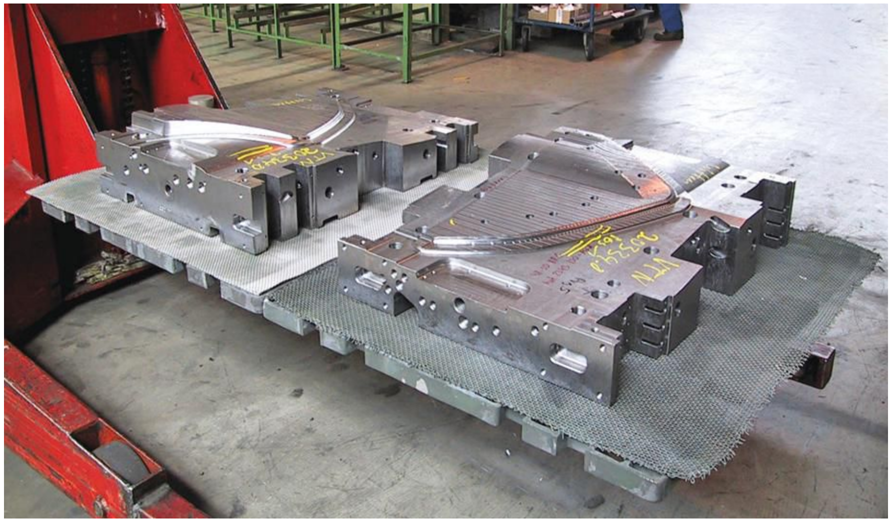

Fig. 9a. H-11 tool-steel die for hardening by high-pressure gas quenching (Photograph courtesy of Ipsen USA)

(λ) values – numbers that represent the time required to pass through this temperature range divided by 100 seconds – are now available for many types of materials as a relative measure of the required cooling rate.

Cooling in argon produces the slowest heat-transfer rates, followed by nitrogen, then helium and finally hydrogen. All these gas mixtures are popular, but nitrogen is the most attractive from a purely cost standpoint. Theoretically, there is no limit to the improvement in cooling rate that can be obtained by increasing gas velocity and pressure. Practically, however, very-high-pressure and very-high-velocity systems are complex and costly to construct. In particular, the power required for gas recirculation increases faster than benefits accrue.

There are pressure/gas combinations that achieve heat-transfer coefficients within the range of those produced by still and mildly agitated oil quenchants. Gas quenching has certain advantages over liquid (oil or salt) quenching. The cooling rate can be easily changed by altering gas velocity or pressure, allowing not only the heat treatment of a wide variety of materials but also complex shapes and components of large or variable cross section. The effect of load weight on the resultant cooling speed during gas quenching is more pronounced than, for example, in liquid quenching. Maximum section size (ruling section) is an important consideration as well.

Tool Steels

For most tool steels, equivalent end-product performance, surface hardness, and mechanical and microstructural properties (carbide size and distribution) can be achieved by vacuum hardening as compared to alternative technologies such as salt bath or atmosphere processing. A major advantage of using vacuum furnaces to process parts in this way is that surfaces are neither carburized nor decarburized and consequently exhibit superior performance.

In general, the air-hardening tool-steel parts are hardened in much the same way as in atmosphere furnaces. They are preheated, heated to a high austenitizing temperature and cooled at a moderate rate. The medium-alloy air-hardening steels in the A-series and the high-carbon, high-chromium steels in the D-series are regularly hardened in gas-quench furnaces using nitrogen up to 6 bar.

A rough vacuum in the range of 1.3 to 1.3 x 10^{-1} mbar (1 Torr – 1 x 10^{-1} Torr) is used in the heat treatment of tool steels. This level of vacuum is required mainly because of the relatively high vapor pressures of chromium, manganese and other easily vaporized elements.

Today, many grades of hot-work tool steels such as H11 (Fig. 9a) and H13 are high-pressure nitrogen-gas quenched at 10-bar pressure as opposed to oil quenched. In many cases, an isothermal hold (Fig. 9b) is introduced to minimize distortion. Gas quenching has been reported to achieve cooling rates in the 40 – 70°C/minute (100 – 160°F/minute) range, adequate for most service applications. Faster cooling rates extended life and enhanced performance. Oil quenching can achieve rates of up to 150°C/minute (300°F/minute).

Martensitic Stainless Steels

All grades of martensitic stainless steels have been processed in vacuum furnaces using the same austenitizing temperatures and considerations as those used in atmosphere furnaces. Since the austenitizing temperatures are usually below 1100°C (2000°F), vacuum levels in the range of 10^{-3} mbar (10^{-3} Torr) are very often used, which result in clean and bright part surfaces. To avoid evaporation of certain alloying elements, processing is also done at vacuum levels ranging from 10^{-1} to 10^{-3} mbar (10^{-1} – 10^{-3} Torr) with some sacrifice to brightness. Due to the differences in the hardenability of the various martensitic stainless alloys, there is a limitation on the section sizes that

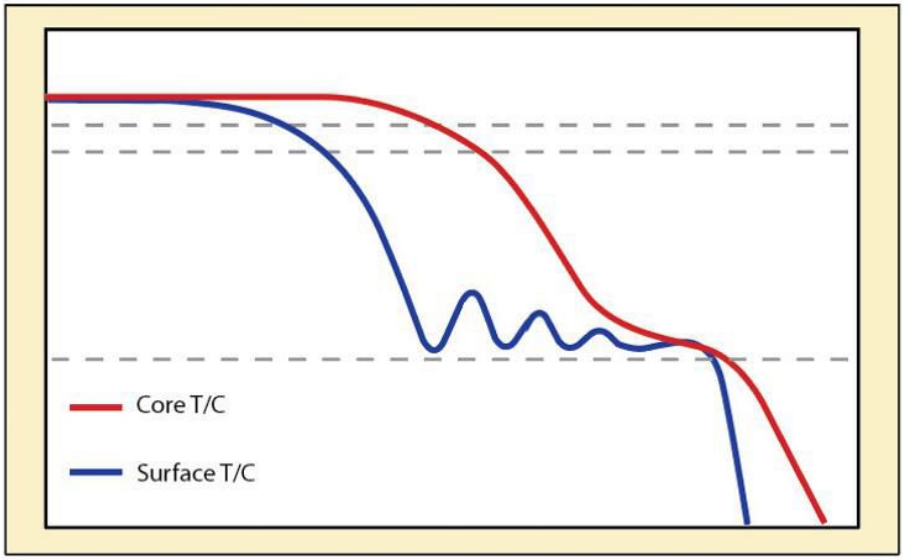

Fig. 9b. Isothermal quench cycle

can be fully hardened by recirculated nitrogen-gas quenching. Other types of cooling gas can be used, but the economic benefits must be carefully considered. The actual values of section-size limits depend on the type of cooling system and the capability of the specific furnace employed.

Alloy Steels

Higher gas pressures (up to 20 bar) and the proper choice of quench gas enable some low-to-medium alloy steels and most case-hardening steels to be hardened. The automotive and aerospace industries are taking advantage of this for their heat-treatable steels that are normally oil hardened.

Sintering

Vacuum sintering, compaction (CIP, HIP) and secondary heat-treatment operations are performed on both conventional powder metal (PM) as well as particulate (CIM, PIM, MIM) materials. In an industry dominated by atmosphere processing, increased interest in controlled-atmosphere sintering arises from factors such as:

- The purity of the vacuum environment and its effect on part microstructure
- The use of sub-atmospheric (partial) pressure to improve the efficiency of the sintering reactions, especially with highly alloyed materials that require elevated sintering temperatures
- The ability of the vacuum process to reduce pore size and improve pore-size distribution
- The higher furnace-temperature capabilities that permit faster sintering reactions carried out much closer to the melting point and with alloys of higher melting point interstitial elements

The limitation on the application of sintering in vacuum furnaces is the vapor pressure of the metals being processed at the chosen sintering temperature. If the vapor pressure is comparable with the working pressure in the vacuum furnace, there will be considerable loss of metal by vaporization unless a sufficiently high partial pressure of inert gas is used. In certain instances, the partial-pressure gas can react with the surface of the part, creating a surface layer that may need to be removed.

Stainless Steel

Vacuum sintering of stainless steel powder-metal parts is a common process, employed for 300 series (e.g. 304, 316) and 400 series (e.g. 410, 420) as well as precipitation-hardening grades – 17-4PH, 17-7PH. These products are very often superior to those sintered in hydrogen or dissociated-ammonia atmospheres with respect to their corrosion resistance and mechanical properties.

High-Speed Steel

Powder-metallurgy manufacturing methods have been developed for producing finished and full-density cutting tools of high-speed tool steel. Applications include such items as complex geometry hobs, pipe taps and reamers. Special isostatic compacting techniques have been developed that use neither lubricants nor binders for these types of components. The pressed compacts are sintered in vacuum furnaces under precise control of heating rate, sintering time, temperature and vacuum pressure in order to eliminate porosity. The result is predictable densification of the pressed compact with final size tolerances of ±0.5 – 1.0%. Full-density, sintered high-speed steel tools have been shown to be at least equivalent to conventional wrought material in cutting performance. Grindability is dramatically improved in particular for the high-alloy grades such as M4 and T15. This is attributed to a finer and more uniform carbide distribution.

Tempering and Stress Relief

Where surface finish is critical and clean parts are desired to avoid any post heat-treat processing, many heat treaters, especially commercial shops, now employ vacuum furnaces for tempering and stress relief. These units typically operate in the temperature range of 130 – 675°C (275 – 1250°F), below which radiant energy is an efficient method for heating. As such, heating by convection is utilized. The furnace is normally evacuated to below 0.10 mbar (0.075 Torr) then backfilled with an inert gas such as nitrogen, argon or even 97% nitrogen/3% hydrogen mixtures to a pressure slightly above atmospheric, typically in the range of 0.5 – 2 bar. A fan in the furnace recirculates this atmosphere, and parts are heated by both convection and conduction. Temperature uniformity in the range of ±5°C (±10°F) is common with tighter uniformities possible.

Certain horizontal single- and multiple-chamber furnaces have been designed to perform single or multiple tempering treatments after hardening or case hardening without having to remove the workload from the equipment. This process is in common use for tempering high-speed-steel components and a variety of other materials.

The series continues discussing aerospace and other applications in Part Two.

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1. Herring D. H., “A Perspective from the Americas: Advanced Technology Trends in Vacuum”, White Paper, May 2007.

Vacuum Tools for Heat Treat Productivity


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Technology Trends in Vacuum Heat Treating, Part Two: Processes & Applications

Part two of this series continues our discussion of vacuum heat-treating processes and applications. Vacuum thermal processing is key for Aerospace component manufacturers due to industry demands for the highest possible quality. Brazing and surface treatment are two process ares where vacuum technology by the Aerospace industry. Let's learn more.

Brazing

The volume of work produced by vacuum brazing far exceeds that of any other process in which vacuum furnaces are being utilized. The transportation (automotive and aerospace) industry has provided the impetus for the increasing use of vacuum furnaces for brazing, and the use of lightweight, high-strength materials have also contributed to the popularity of brazing.

As with all brazing, variables that need to be controlled to produce mechanically sound braze joints include base-metal selection and characteristics, filler-metal selection and characteristics, component design, joint design and clearance, surface preparation, filler-metal flow characteristics, temperature and time, and rate and source of heating.

Some factors affect the ability to produce a metallurgically sound braze joint by influencing the behavior of the brazed joint, while others affect the base-metal properties, while still others influence the interactions between the base metal and filler metal. Effects on the base metal include carbide precipitation, hydrogen embrittlement, the nature of the heat-affected zone, oxide stability and sulfur embrittlement. Filler-metal effects include vapor pressure, alloying, phosphorous embrittlement and stress cracking. Interaction effects include post-brazing thermal treatments, corrosion resistance and dissimilar-metal combinations.

Vacuum furnaces can be either horizontal or vertical in design (Figs. 1a, 1b) and have technical advantages including:

- The process permits brazing of complex, dense assemblies with blind passages that would be almost impossible to braze and adequately clean using atmospheric flux-brazing techniques.
- Vacuum furnaces operating at 10^{-5} to 10^{-4} mbar (10^{-5} to 10^{-4} Torr) remove essentially all gases that could inhibit the flow of brazing alloy, prevent the development of tenacious oxide films

and promote the wetting and flow of the braze alloy over the vacuum-conditioned surfaces.

- Properly processed parts are unloaded in a clean and bright condition, often avoiding additional processing.
- A wide variety of materials ranging from aluminum, cast irons, stainless steel, steels, titanium alloys, nickel alloys and cobalt-base superalloys are brazed successfully in vacuum furnaces without the use of any flux.

Many different types of nickel, nickel-base, copper, copper-base, gold-base, palladium-base, aluminum-base and some silver-base brazing alloys are used for the filler metal. Generally, alloys that contain easily vaporized elements for lowering the melting points are avoided. With respect to the heat treatment of steel, the copper and the nickel-base brazing alloys are the most widely used filler metals.

Aluminum and Aluminum Alloys

In the brazing of aluminum components, it is important that vacuum levels be maintained in the 10^{-5} mbar (10^{-5} Torr) range or better. Parts are heated to 575–590°C (1070–1100°F) depending on the alloy. Temperature uniformity is critical, typically ±5.5°C (±10°F) or better, and multiple-zone temperature-

Fig. 1a. Jet engine turbine blades brazed in a horizontal vacuum furnace

Fig. 1b. Various aerospace components brazed in a bottom-loading vacuum furnace

Fig. 2. Typical honeycomb seals

Fig. 3. Family of aerospace, automotive and industrial components illustrating the versatility of low-pressure vacuum carburizing

controlled furnaces are common. Cycle times are dependent on furnace type, part configuration and part fixturing. Longer cycles are required for large parts and very dense loads.

Copper and Copper Alloys

Copper filler metal applied to the base metal either as paste, foil, clad or solid copper can be vacuum brazed recognizing that the high vapor pressure of copper at its melting point causes some evaporation and undesirable contamination of the furnace internals. To prevent this action, the furnace is first evacuated to a low pressure 10^{-2} to 10^{-4} mbar (10^{-2} to 10^{-4} Torr) to remove residual air. The temperature is then raised to approximately 955°C (1750°F) to allow outgassing and to remove any surface contamination. Finally, the furnace is heated to brazing temperature normally 1100–1120°C (2000–2050°F) under a partial pressure of inert gas up to 1 mbar ($7.5\text{x}10^{-1}$ Torr) to inhibit evaporation of the copper. When brazing is completed, usually within minutes after the set-point temperature has been reached, the work is allowed to slow cool to approximately 980°C (1800°F) so that the filler metal will solidify. Parts can then be rapidly cooled by gas quenching, typically in the range of 2 bar.

Nickel-Base Alloys

Brazing with nickel-base alloys is usually done without any partial pressure at the vacuum levels in the range of 10^{-3} to 10^{-5} mbar (10^{-3} to 10^{-5} Torr). Normally, a preheat soak at 920–980°C (1700–1800°F) is used to ensure that large workloads are uniformly heated. After brazing, the furnace temperature can be lowered for additional solution or hardening heat treatments before gas cooling and unloading.

Aerospace Brazing Process Example

A honeycomb seal (Fig. 2) is a jet engine component designed to increase engine efficiency by surrounding the airfoil or turbine blade and prevent airflow around the blade tips. Honeycomb seals are made from a variety of nickel and cobalt superalloys (Table 1) designed to withstand the harsh service environments of jet engine applications.

Rolling and tacking are the two most important steps in the pre-braze assembly process. Firm tack is necessary to ensure intimate contact and achieve a sound joint during brazing. All dimensions are set during the tacking process. Braze tolerances of 0.25–0.50 mm (0.010–0.020 inch) are common. Proper cleaning is another critical pre-braze step. Every effort should be made to ensure the part is clean and free of all oxides, contaminants and oils prior to braze preparation.

Table 1. Honeycomb Materials

Component	AMS Specification			Major Constituents
Honeycomb	5536	5878		Ni, Cr, Co, Mo
Rings and Details	5662	5596	5706	Ni, Cr, Mo, Cb, Ti
Filler Metal	4777	4779	4782	Ni, Si, Cr, B

The furnace cycle is equally as critical as part preparation to the success of the brazing operation. Parts that ramp too fast are at risk for distortion and uneven temperature throughout the assembly. Parts that are not stabilized will not see proper braze flow. If the assembly quenches too rapidly, there is risk for distortion, quench cracking of the braze joint and splatter.

Brazing of these high-temperature nickel alloys is typically performed at 1040–1200°C (1900–2200°F) in a vacuum level of 10^{-4} to 10^{-5} mbar (10^{-4} to 10^{-5} Torr). Brazing is performed 40–65°C (100–150°F) above the braze-alloy melting point.

Common problems include splatter of the braze alloy, quench cracking and distortion. All of these problems can be prevented by controlling cleanliness of the part, using proper setup technique, designing a proper brazing recipe and operating the furnace properly. Multiple re-brazes can be performed using shorter brazing cycles at slightly higher temperatures. Stop-off paints such as alumina oxide (preferred) can be applied to reduce the risk of unwanted braze flow.

Case Hardening by Carburizing and Carbonitriding

Low-pressure vacuum carburizing (LPC) or low-pressure vacuum carbonitriding (LPCN) combined with either high-pressure gas quenching (HPGQ) or oil quenching (OQ) has become increasingly popular over the past decade, with industries such as aerospace, automotive and commercial heat treating spearheading the use of this technology (Figures 3 - 5).

It is generally agreed that low pressure can be defined as carburizing less than 27 mbar (20 Torr), typically at temperatures from 830–980°C (1525–1800°F) for carburizing and 800–900°C (1475–1650°F) for carbonitriding. In the past several years, higher carburizing temperatures – up to 1200°C (2200°F) in several instances – have been used for certain advanced materials.[1] There is also growing interest in Cr-Mn steels as alternatives to the more expensive conventional alloy grades.

Popular hydrocarbons include acetylene (C_2H_2) and acetylene mixtures such as [acetylene + hydrogen] and [acetylene + ethylene (C_2H_4) + hydrogen] and cyclohexane (C_6H_{12}), a liquid. The use of propane and methane, while still popular, is declining. Ammonia can be added to the selected hydrocarbon gas usually during the diffusion steps for carbonitriding.

Process control is achieved through the use of simulation programs applied to the respective kinetic and diffusion models to determine the boost and diffuse times for a given case depth. Carbon transfer rates are now well established for a given temperature, gas type, gas pressure and flow rate. Material chemistry and surface area must be taken into consideration in these programs, as well as initial and final surface-carbon levels. Prediction of case depth and hardness profiles are the most obvious output of these programs, with research continuing into prediction of microstructural results such as carbide size and distribution and retained austenite levels.


Fig. 4. Heavy-duty truck shafts low-pressure vacuum carburized and oil quenched

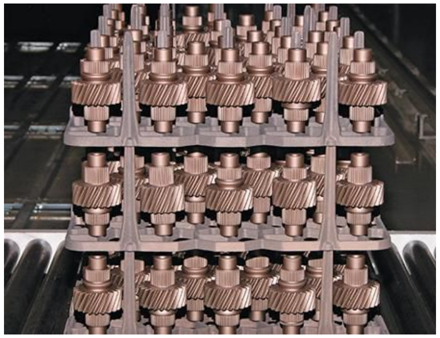
Fig. 5. Automotive transmission gears low-pressure vacuum carburized and high-pressure gas quenched

Case Hardening by Nitriding and Nitrocarburizing

Plasma (ion) nitriding (Fig. 6) using pulsed power generators is an option to the traditional gas nitriding

Fig. 6. Plasma nitriding of automotive crankshafts (Photograph Courtesy of Surface Combustion)

process. Nitriding is used in many applications for increased wear resistance and improved sliding friction as well as in components where increased load-bearing capacity, fatigue strength and corrosion resistance are important. Corrosion resistance can be especially enhanced by a plasma post-oxidation treatment. Dimensional changes are minimal, and the masking process for selective nitriding is simple and effective.

Plasma nitriding uses nitrogen gas at low pressures in the range of 1–10 mbar (0.75–7.5 Torr) as the source for nitrogen transfer. Above 1000°C (1832°F), nitrogen becomes reactive when an electric field in the range of 300–1200 V is applied. The electrical field is established in such a way that the workload is at the negative potential (cathode) and the furnace wall is at ground potential (anode). The nitrogen transfer is caused by the attraction of the positively charged nitrogen ions to the cathode (workpieces) with the ionization and excitation processes taking place in the glow discharge near the cathode's surface. The rate of nitrogen transfer can be adjusted by diluting the nitrogen gas with hydrogen (above 75%). The higher the nitrogen concentration, the thicker the compound layer.

The compound layer consists of iron and alloy nitrides that develop in the outer region of the diffusion layer after saturation with nitrogen. According to the iron-nitrogen phase diagram, basically two iron nitrides are possible – the nitrogen-poor gamma prime (γ') phase (Fe_4N) and the nitrogen-rich epsilon (ε) phase ($Fe_{2-3}N$).

The temperature of the workpiece is another important control variable. The depth of the diffusion layer also depends strongly on the nitriding temperature, part uniformity and time. For a given temperature, the case depth increases approximately as the square root of time. A third process variable is the plasma power or current density, which is a function of surface area and has an influence on the thickness of the compound layer.

Plasma nitrocarburizing is achieved by adding small amounts (1–3%) of methane or carbon dioxide gas to the nitrogen-hydrogen gas mixture to produce a carbon-containing epsilon (ε) compound layer ($Fe_{2-3}C_xN_y$). It is commonly used only for unalloyed steels and cast irons.

Part Three of this series will talk about future trends in vacuum heat treating.

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Technology Trends in Vacuum Heat Treating, Part Three: New Technologies & Future Developments

Many new technologies owe their success to vacuum processing. Let's learn more.

Examples include:

- Rotational compression brazing of aerospace rocket and military jet engines
- Titanium processing including beta annealing, slow cooling and age hardening (BASCA 160) of titanium alloys
- Vacuum carburizing/carbonitriding and high-pressure gas quenching of automotive powertrain components
- High-temperature sintering followed immediately by vacuum hardening for automotive transmission components

New Technologies

Let's look at each one of these technologies a little closer.

Rotational Compression Brazing

Reusable rocket engines are a practical necessity facing NASA and other space agencies. Joint technology programs have incorporated Russian technology, most notably rotational compression brazing, in combination with channel wall construction to enhance nozzle reliability via machine-controlled processes and very low part count. Special Ag-Pd alloys (Fig. 1) are used to join the liner, jacket, stiffeners and manifold components together.

During the brazing process, a vacuum of 10^{-5} mbar (10^{-5} Torr) is pulled on the inner assembly while a positive pressure of 5–6 bar is applied to the outer surfaces. This pressure differential, in combination with rotating the part during heating and brazing to

Fig. 1. Channel-wall braze joints (Photograph courtesy of GenCorp Aerojet)

Fig. 2. Vacuum annealing of titanium coils .*(Photograph courtesy of Solar Atmospheres)*

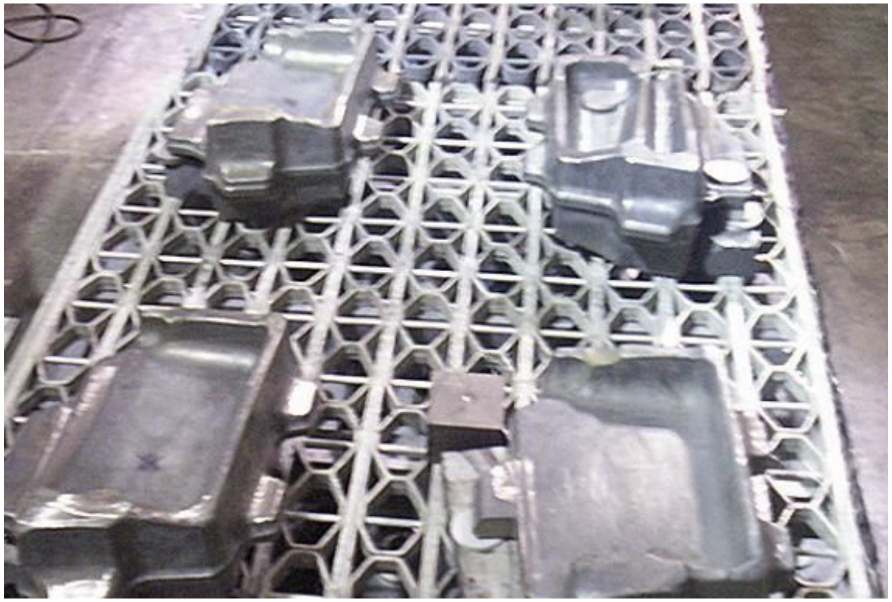

Fig. 3. Ti-5553 castings (Photograph courtesy of Solar Atmospheres)

counteract the forces of gravity on the braze joint, produces a superior part.

BASCA Process

Titanium and titanium alloys are annealed (Fig. 2) to produce an optimum combination of ductility, machinability, dimensional stability and structural stability. Like recrystallization annealing, beta annealing improves the fracture toughness of titanium alloys. Beta annealing is performed at temperatures slightly above the beta transus of the alloy being annealed to prevent excessive grain growth. Annealing times are dependent on the section thickness and are sufficient for complete transformation. Time at temperature after transformation is held to a minimum to control beta grain growth.

Russian and American aerospace manufacturers have collaborated to develop a unique beta-annealing process involving slow cooling and age hardening. The BASCA 160 process (Fig. 3) for alloys such as Ti-5553 (5%Al-5%V-5%Mo-3%Cr) is used to achieve tensile values in the 1,100 MPa

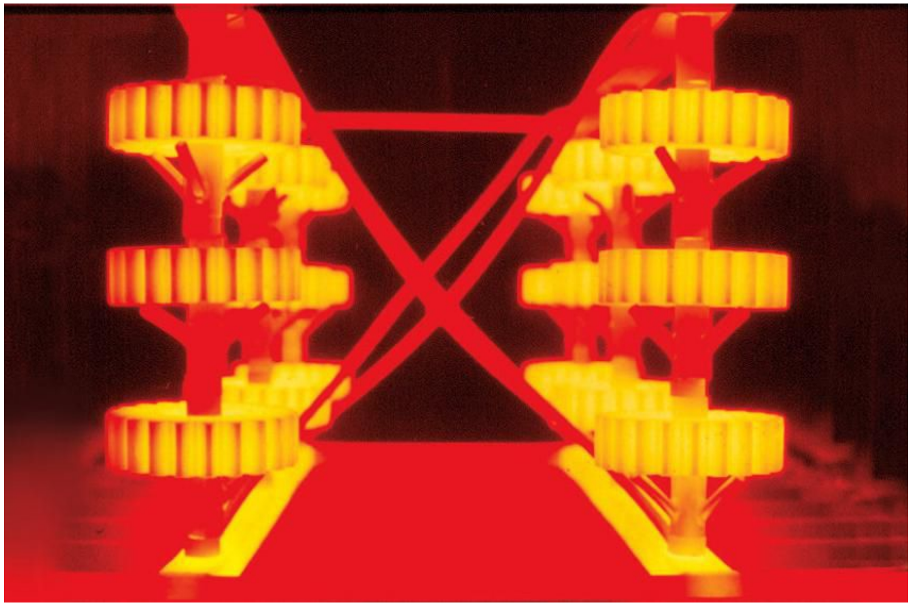

Fig. 4. First production vacuum-carburized load - circa 1969 (Photograph courtesy of C. I. Hayes)

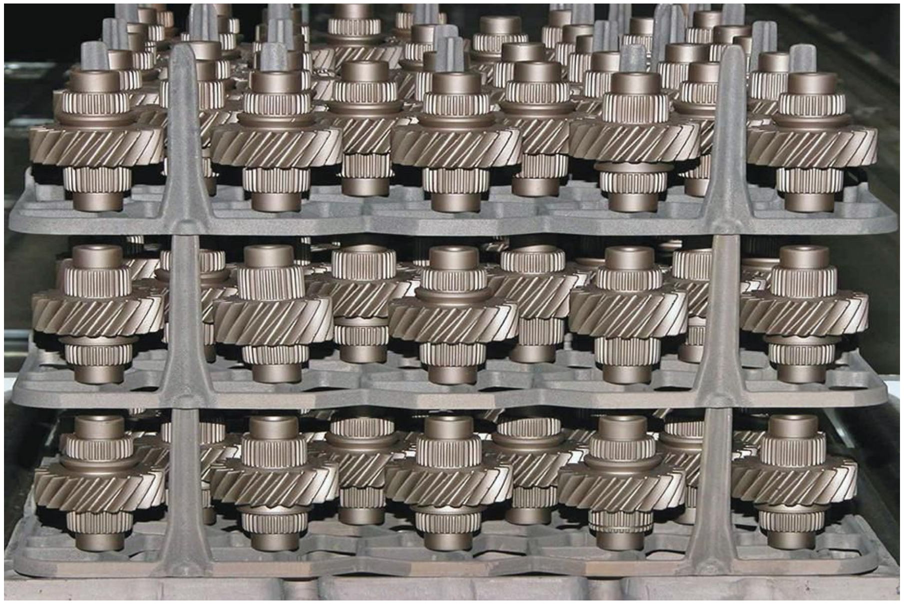

Fig. 5. Typical LPC vacuum-carburized components (Photograph courtesy of ALD Thermal Treatment)

(160,000 psi) range. The key to the process is a very precise and controlled slow cool through a defined critical range then age hardening to avoid both distortion and alpha (α) case formation.

Low-Pressure Vacuum Carburizing and Carbonitriding

Vacuum carburizing, invented and commercialized in the late 1960s (Fig. 4) was perfected with the advent of low-pressure carburizing – under 7.5 mbar (10 Torr) – in the mid 1990s due in large part to the pioneering work on the use of acetylene as the principle hydrocarbon gas for carburizing as reported in the late 1970s in Russia (USSR Patent No. 668978).

Today, the focus is on low-pressure vacuum carburizing for advanced applications in aerospace, motorsports and automotive (Fig. 5). Technology drivers include performance enhancement and production throughput. New part designs are specifying vacuum-carburized components, and the development and use of inexpensive Cr-Mn steels is just beginning to emerge.

Carburizing cycles for new materials, especially using carburizing temperatures above 980°C (1800°F), continue to be of great R&D interest. In some cases, cycle times have been reduced by 33–50% over atmosphere carburizing – processes requiring several hundred hours. Short-duration boost cycles (in the range of seconds) and long diffusion times (in the range of hours) provide just enough carbon to the surface of the part while avoiding the formation of retained austenite, carbide networks and necklaces.

Some of these materials include:

- Aubert & Duval: X12 VDW and XD15NW
- Carpenter Technology: Pyrowear 53, 675 and AerMet 100
- Böhler-Uddeholm: N360 Iso Extra, N695, R250, R350
- Questek Innovations: Ferrium C61, CS62, C69
- The Timken Company: CSS-42L, CSB-50NIL, CBS 223, CBS-600, BG42VIM/VAR, AF1410, HY180, HP-9-230, HP-9-430 and 300M
- Atlas Specialty Steels: BS970 and EN30B
- VSG Essen: Cronidur 30
- Teledyne Corporation: VascoMax C-250, C-300, C-350

High-Temperature Sintering and Vacuum Hardening

Fig. 6. Clutch and synchronizer hubs after LPC+HPGQ – SL-5506 material (Photograph courtesy of Stackpole Ltd.)

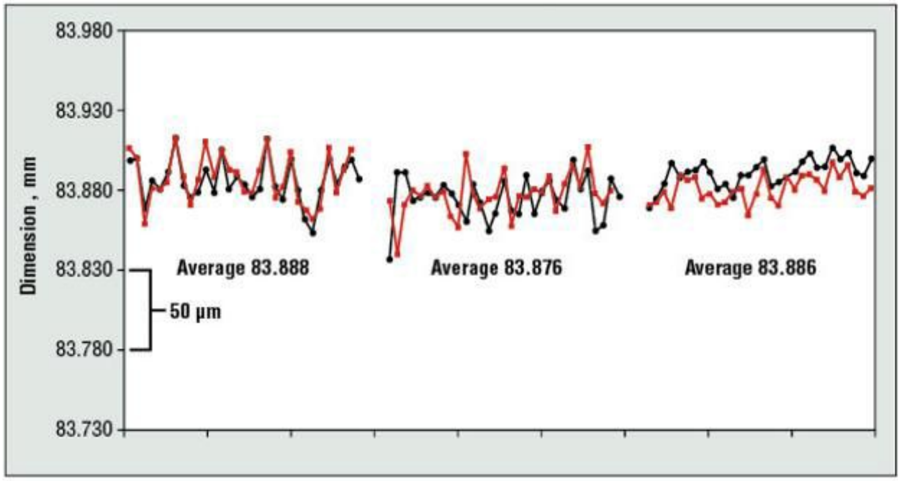

Fig. 7. Statistical process analysis – measurement over balls 0.036578 mm (0.0015 inches) – at two locations (Photograph courtesy of Stackpole Ltd.)

High-temperature sintering of automotive-transmission clutch and synchronizer hubs (Fig. 6) coupled with vacuum hardening and high-pressure gas quenching provide an example of the synergy between material selection and the heat-treating process. Product specifications mandate finish machining and heat treatment to 35 HRC. Conventional process solutions, including hard-machining operations, involve:

- FLN2-4405 (0.6%C, 1.85%Ni, 0.85% Mo) material hardened, oil quenched and tempered
- FLNC-4408 (0.75%C, 0.85%Mo, 2.0%Cu, 2.0%Ni) material sinter hardened

Finding a lower-cost technology alternative led to the investigation of SL-5506 (0.6%C, 0.5%Mn, 0.5%Cr, 0.4%Ni) material. High-temperature sintering was required at 1280°C (2340°F) followed by vacuum heat treating, 5-bar helium quenching and tempering. Benefits included a reduced number of manufacturing steps (from 9 to 7) and the avoidance of the hard-turning operation.

A comparison of the technologies (Fig. 7) revealed significant quality improvement (less statistical scatter). No qualification of the spline form was performed between sinter and heat treat. The profile was held within 10 μm (0.0004 inches) and

the lead within 15 μm (0.0006 inches) over 14 mm (0.55 inches).

Future Developments

The future for vacuum heat treatment is bright. Whether considering the technology from an energy or environmental standpoint or when looking to optimize material property development, vacuum processing offers the flexibility that few other technologies can match. Offering high-vacuum and high-temperature capability, precision and repeatability are only a few of its many attributes. Vacuum has the versatility to handle a handful of extremely tiny parts (Fig. 8) or massive loads (Fig. 9) and anything in between. Areas of particularly rapid growth are in the medical, aerospace and automotive industries.

Fig. 8. Vacuum-carburized medical fastener
(Photograph courtesy of Midwest Thermal-Vac)

Fig. 9. Vacuum homogenizing of titanium ingots
(Photograph courtesy of Solar Atmospheres)

Using Partial Pressure in Vacuum Furnaces

It is of primary importance to understand vapor pressure in the vacuum heat treatment of metals. Operating in vacuum can often lead to significant evaporation, or "boiling away," of elemental constituents in the materials being processed, affecting surface integrity and in some cases altering the chemical composition of the base (or filler) metal. One way to overcome this problem is to introduce a gas partial pressure higher than that of the material's vapor pressure. Different gas choices, introduction methods and controls are available to the heat treater. The natural questions are how and when should they be used? Let's learn more.

Vapor Pressure

The vapor pressure of a material is the partial pressure present in the atmosphere which surrounds it. In other words, the vapor pressure tells us how much vapor a material will produce; a high vapor pressure means that the material will readily evaporate. Every material has a characteristic vapor pressure associated with it, which varies with temperature; the vapor pressure increases with increasing temperature increases.

All metals evaporate as a function of temperature (first-order effect) and vacuum level (second-order effect). Equation 1 allows the determination of the evaporation rate (Q), and shows that the vapor pressure/temperature relationship is nearly logarithmic:

$$(1) \qquad Q\text{max}=0.058Pv\sqrt{\frac{M}{T}}$$

where Q_{max} = evaporation rate (g/cm^3-sec), P_v = vapor pressure (Torr), T = temperature (K) and M = molecular weight.

In vacuum furnaces, metals tend to volatize at temperatures below their melting points. Table 1 shows this relationship for a number of common metals. The longer parts are held at the temperature and at the vacuum level shown, the greater the loss of the metallic element by evaporation. Where the element is part of a metal alloy system the vapor-pressure relationship changes (total vapor pressure of the alloy is the sum of the vapor pressures of each constituent times the percentage in the alloy, although this relationship has been debated by those knowledgeable in the field).

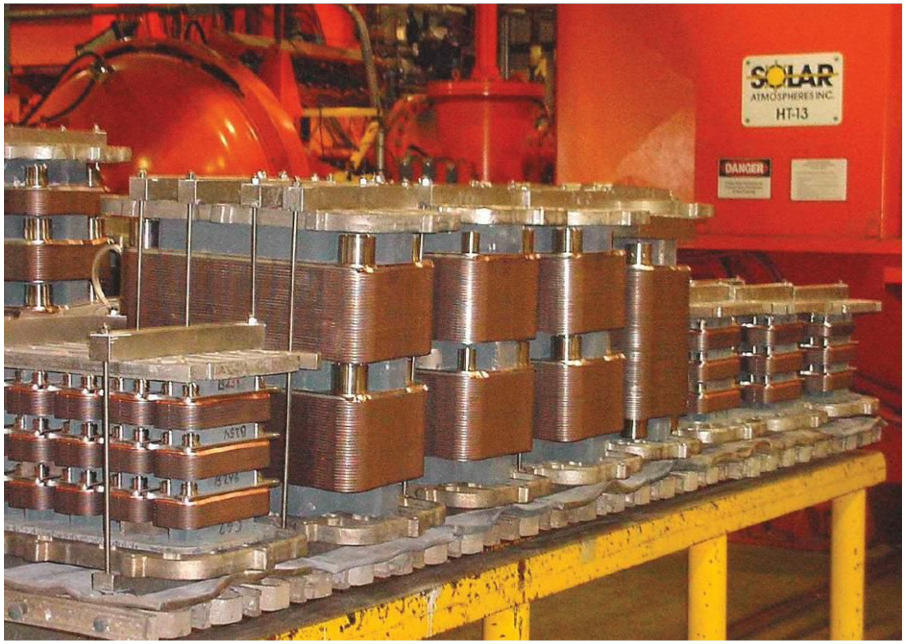

Fig. 1 Load of flat-plate heat exchangers ready for processing under partial pressure. Photo courtesy of Solar Atmospheres Inc, Souderton, Pa.

For example, processing aluminum, cadmium, magnesium, manganese and zinc or their alloys at temperatures as low as 400°C (750°F) may be marginally or totally impractical. This is why processing brass (Cu-Zn alloy) is normally not done in a vacuum system, or if it is, it is carried out at partial pressures near atmospheric. As the temperature increases, fewer materials can be run without being affected.

Stainless and tool steels and more exotic alloys often are processed in vacuum (Fig. 1). Chromium present in these materials evaporates noticeably at temperatures and pressures within normal heat treatment ranges. Processed above 990°C (1815°F), chromium will vaporize if the vacuum level is less 1×10^{-4} Torr and the parts are held for a prolonged time. Heat treaters often observe a greenish discoloration (chromium oxide) on the interior of their vacuum furnaces, the result of chromium vapor reacting with air leaking into the hot zone. Otherwise, the evaporation deposit is bright and mirror-like. To avoid this, an operating partial pressure between 0.3 and 5 Torr is typical for most chromium-containing parts.

For vacuum brazing (silver, copper, nickel), depletion of the filler metal alloy can be avoided by raising the pressure in the furnace to a level above the vapor pressure of the alloy at brazing temperature. For example, copper, having an equilibrium vapor pressure at 1120°C (2050°F) of 1×10^{-3} Torr is usually run at a partial pressure between 1 and 10 Torr. Nickel brazing normally is done in the 10^{-3} to 10^{-4} Torr range. However, in the 10^{-5} to 10^{-6} Torr range, you run the risk of losing some nickel, which has an equilibrium vapor pressure of 1×10^{-4} Torr at 1190°C (2175°F).

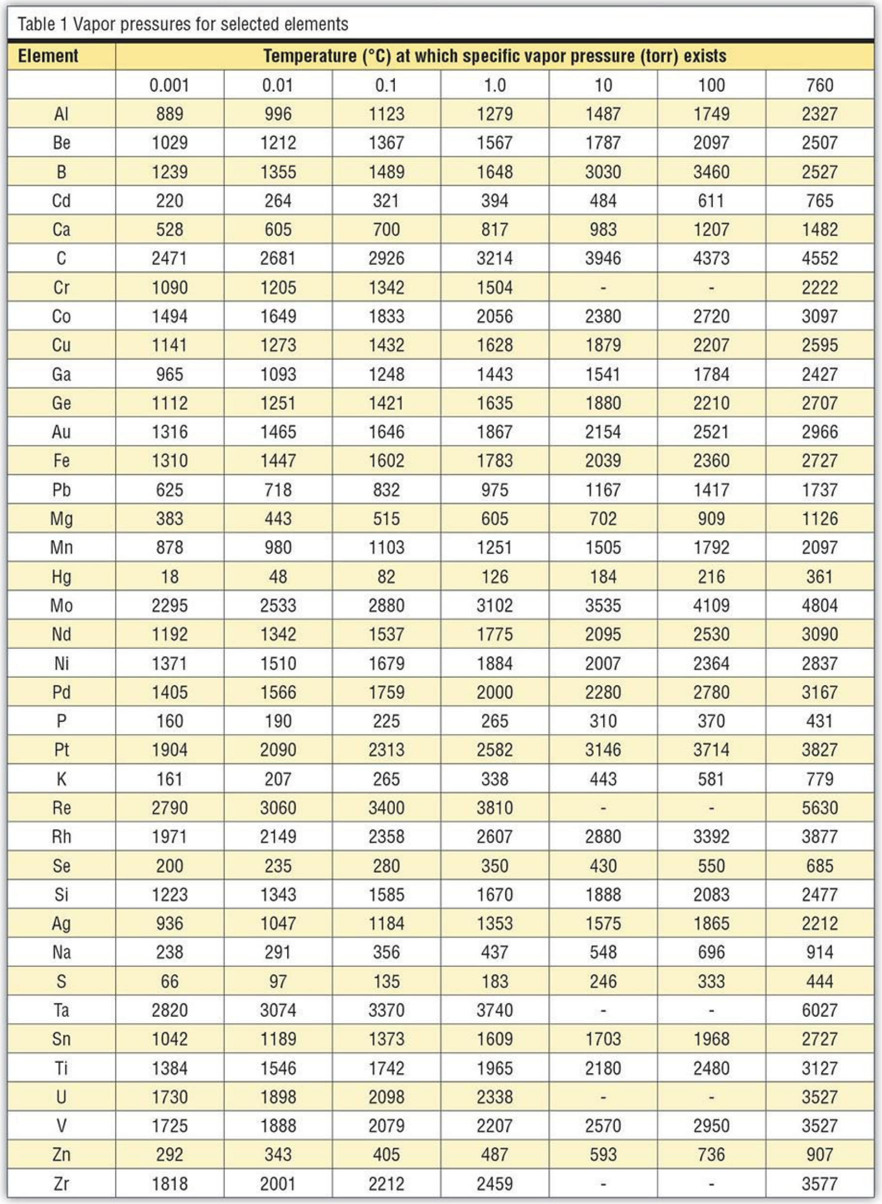

Table 1 Vapor pressures for selected elements

Element	Temperature (°C) at which specific vapor pressure (torr) exists						
	0.001	0.01	0.1	1.0	10	100	760
Al	889	996	1123	1279	1487	1749	2327
Be	1029	1212	1367	1567	1787	2097	2507
B	1239	1355	1489	1648	3030	3460	2527
Cd	220	264	321	394	484	611	765
Ca	528	605	700	817	983	1207	1482
C	2471	2681	2926	3214	3946	4373	4552
Cr	1090	1205	1342	1504	-	-	2222
Co	1494	1649	1833	2056	2380	2720	3097
Cu	1141	1273	1432	1628	1879	2207	2595
Ga	965	1093	1248	1443	1541	1784	2427
Ge	1112	1251	1421	1635	1880	2210	2707
Au	1316	1465	1646	1867	2154	2521	2966
Fe	1310	1447	1602	1783	2039	2360	2727
Pb	625	718	832	975	1167	1417	1737
Mg	383	443	515	605	702	909	1126
Mn	878	980	1103	1251	1505	1792	2097
Hg	18	48	82	126	184	216	361
Mo	2295	2533	2880	3102	3535	4109	4804
Nd	1192	1342	1537	1775	2095	2530	3090
Ni	1371	1510	1679	1884	2007	2364	2837
Pd	1405	1566	1759	2000	2280	2780	3167
P	160	190	225	265	310	370	431
Pt	1904	2090	2313	2582	3146	3714	3827
K	161	207	265	338	443	581	779
Re	2790	3060	3400	3810	-	-	5630
Rh	1971	2149	2358	2607	2880	3392	3877
Se	200	235	280	350	430	550	685
Si	1223	1343	1585	1670	1888	2083	2477
Ag	936	1047	1184	1353	1575	1865	2212
Na	238	291	356	437	548	696	914
S	66	97	135	183	246	333	444
Ta	2820	3074	3370	3740	-	-	6027
Sn	1042	1189	1373	1609	1703	1968	2727
Ti	1384	1546	1742	1965	2180	2480	3127
U	1730	1898	2098	2338	-	-	3527
V	1725	1888	2079	2207	2570	2950	3527
Zn	292	343	405	487	593	736	907
Zr	1818	2001	2212	2459	-	-	3577

Which Gases Can We Use?

Argon, hydrogen and nitrogen are the most common partial pressure gases. Often, argon is preferred as it tends to “sweep” the hot zone; that is, the heavy molecule tends to reduce evaporation compared with nitrogen and hydrogen. Specialized applications such as those in the electronics industry may use helium or even neon (if an ionizing gas is needed). Gases having a minimum purity of 99.99% and a dew point of -60°C (-76°F) or lower should be specified.

Certain cautions are in order. For example, nitrogen may react with certain stainless steels and titanium-containing materials resulting in surface nitriding. In the case of hydrogen, the normally near neutral vacuum atmosphere can be sharply shifted to a reducing atmosphere to prevent oxidation of sensitive process work or for furnace/fixture bakeout/cleanup cycles. Embrittlement by hydrogen is a concern for certain materials (e.g., Ti, Ta).

Measurement and control

It is critical to know the exact pressure, flow and type of gas being injected into the vacuum furnace so the process being run is under control. Thermocouples typically found on vacuum furnaces are affected by gas species, because they are calibrated for air. It is not uncommon to believe, for example, that you are running an argon partial pressure at 1 Torr when in reality you are running at 0.4 Torr, or with hydrogen (or helium) that you are at 10 Torr when you are really at 1 Torr. Absolute-pressure gages, such as the MKS Barocel® (MKS Instruments Inc., Wilmington, Mass.; www.mksinst.com) should be used to determine precise partial pressure values.

For flow accuracy, flowmeters should have a micrometer needle valve installed in the downstream line. On many units, the gas is pulsed in using a solenoid valve and set-point control on the vacuum gage, similar to continuous flow with a needle valve installed. Also, it is extremely important to inject the partial pressure gas directly into the hot zone so the gas does not short circuit the work area.

Summary

Partial pressure atmospheres are required in many heat treating and brazing operation to achieve the desired results. Introduction of the partial pressure gas into the furnace hot zone at one or more locations and controlling the partial pressure injection gas stream as a continuous flow rather than trying to operate at a specific pressure are critical considerations. The choice of partial pressure gas is also important both from a cost and quality standpoint.

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A Case for Higher Carburizing Temperatures

As heat treaters, we are always looking for ways to shorten our processing times. Increasing carburizing temperature has long been known to shorten cycle times, but grain growth has been our Achilles' heel. Today there is renewed focus on optimization of diffusion-related processes, and certain microalloying elements show promise for allowing us to raise carburizing temperatures. Let's learn more.

What Is Grain Growth, and Why Do We Care?

Grain growth, or an increase in the size of the grains, occurs by the movement of grain boundaries. A grain boundary is simply the interface between two adjacent grains. Grain boundaries can be thought of as imperfections in the crystal structure acting as barriers to the movement of dislocations (defects). Since these boundaries are regions of high energy, they make excellent sites for the nucleation of precipitates and contribute to the formation of secondary phases – such as martensite – in steel. As such, they influence mechanical properties – finer-grained steels have higher yield strength and do not harden quite as deeply and as a result have fewer tendencies to crack. Grain boundaries are also preferred sites for the onset of corrosion, and they influence the mechanism of creep.

It has long been held that grain size will remain small for steels held at temperatures slightly above the critical temperature, but at higher temperatures a wide variation in grain size occurs depending on chemical composition and the deoxidation method used in the steelmaking practice. Today microalloyelement additions of aluminum, niobium and titanium (Fig. 1) have been found to form fine precipitates (carbonitrides), and it is now possible to produce case-hardening steels that are resistant to grain growth up to temperatures as high as 2100°F (1150°C).[1]

These small carbonitride particles act to prevent the movement of grain boundaries by exerting a "pinning" pressure. Zener pinning is the influence of a dispersion of fine particles on the movement of grain boundaries and has a strong influence on recovery, recrystallization and grain growth.

Some Experimental Results

The grain-coarsening behavior of a modified 16MnCr5 (5120) steel chemistry (Table 1) has

Table 1. Chemical composition for various grades of 16MnCr5 (SAE 5120)

Steel	C	Si	Mn	Cr	Al	N	Nb	Ti
A	0.19	0.23	0.74	1.19	0.041	0.0264	0.045	-
B	0.22	0.22	1.20	1.08	0.026	0.0145	0.024	-
C	0.17	0.28	1.14	1.02	0.029	0.0129	0.030	0.007
D	0.17	0.30	1.35	1.12	0.016	0.0186	0.055	0.016

been investigated[2] by looking at the temperature at which precipitates form.

Over the entire temperature range (Fig. 2), steel B, the base composition, showed the lowest amount of precipitates while steel D showed the highest. At 1750°F (950°C), the amount of carbonitride precipitates present reached 236 ppm for steel B, 400 ppm for steel C and 814 ppm for steel D. The composition of the carbonitride precipitates revealed that in steel B only NbC precipitates are formed. In the steels C and D, which have significantly higher N contents, complex carbonitrides of Nb and Ti are formed, with Nb and N as the most prominent constituents.

Whereas Steel B shows undesirable grain growth at 1925°F (1050°C), the other microalloyed steel grades retain their fine-grained structure. Steel A – containing 450 ppm Nb and an increased amount of Al and N – shows the finest grain size distribution (ASTM 10) up to 1925°F (1050°C).

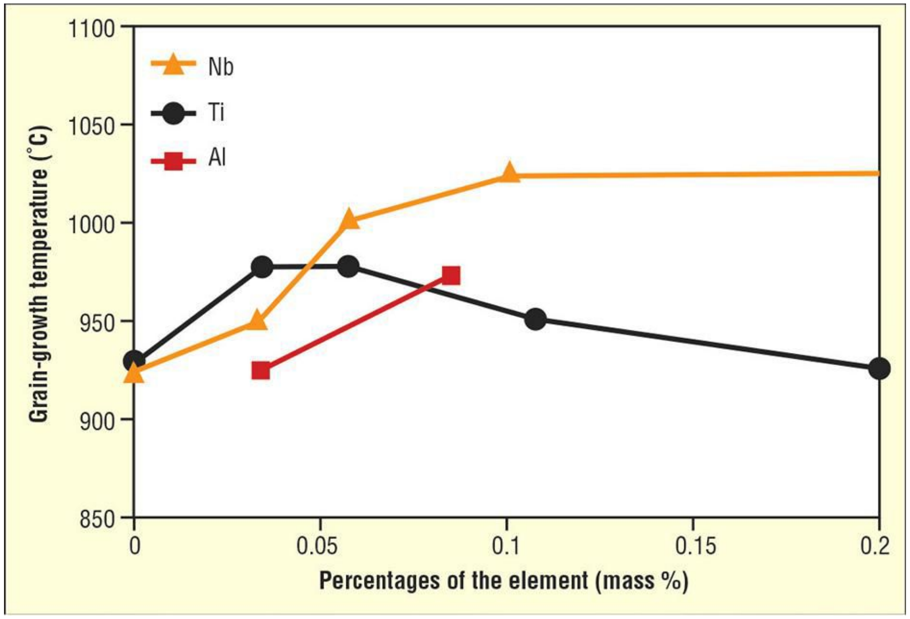

Fig. 1. Influence of alloying elements on grain-growth behavior (20MnCr5) [1]

Beyond 1925°F (1050°C), grain growth sets in with a 90% cumulative frequency of ASTM 1 at 2000°F (1100°C). Steels C and D show significantly less grain coarsening. With 90% of the grains being smaller than ASTM 6, they still remain fine grained up to 2000°F (1100°C).

Due to the increased amount of microalloying elements, the materials have a significant amount of Nb carbides and nitrides as well as Ti nitrides. Besides Al nitrides – that dissolve at temperatures above 1825°F (1000°C) – these precipitates act as grain boundary pinning particles. This gives improved grain size stability at higher temperatures that has been confirmed by various grain growth investigations. At temperatures above 2000°F (1100°C), even the Nb and Ti carbonitrides coarsen in a way that allows grain growth to take place.

Influence of Alloying Elements

When niobium is added to steels, it forms very stable carbides that facilitate grain refinement and allow precipitation hardening to achieve the strengthening mechanisms. The addition of as little as 0.02% Nb can increase the yield strength of medium-carbon steel by 70-100 MPa (10- 15 ksi). Alloy additions are usually in the range of 0.01%-0.20%. Small additions of niobium increase the yield strength and, to a lesser degree, the tensile strength.

Titanium additions refine the grain size and decrease hardenability. It is also a very strong carbide and nitride former. Titanium steels exhibit secondary hardening upon tempering due to the precipitation of TiC. The effect is increased when austenitizing temperatures are above 1800°F (980°C).

Aluminum has strong grain-refining properties but has a weak affect on hardenability and detracts from deep hardening. Aluminum, tends to enhance

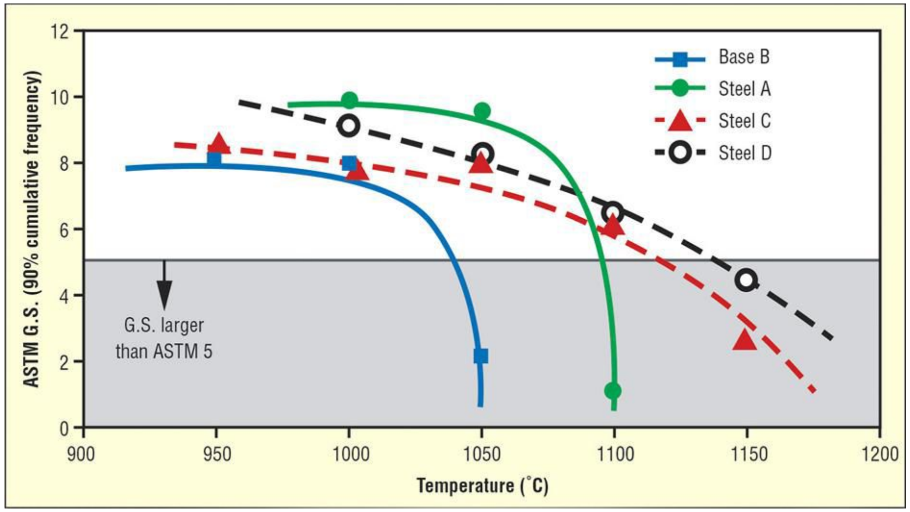

Fig. 2. Grain growth behavior of microalloyed steels[2]

creep, and it is widely used as a deoxidizer. Of all the alloying elements, aluminum is the most effective in controlling grain growth prior to quenching.

Nitrogen increases the tendency to form carbonitrides and has a positive effect on hardenability. Nitrogen increases the strength, hardness and machinability of steel but decreases the ductility and toughness. In aluminum-killed steels, nitrogen forms aluminum nitride particles that control the grain size of the steel, thereby improving both toughness and strength. The presence of various alloy nitride particles tends to retain a finer grain size during tempering.

Grain Size Control

Microalloying elements show great promise in preventing undesired grain growth by increasing the grain-coarsening temperature. Some studies[1] suggest that with the addition of microalloying elements such as niobium, aluminum and titanium, it is possible to raise the carburizing temperature before the onset of significant grain growth above 1900°F (1040°C) and possibly as high as 2100°F (1150°C). To achieve a fine grain structure, it appears that additions of at least 300 ppm aluminum, 450 ppm niobium or 100 ppm titanium are necessary to steels already containing about 175 ppm nitrogen.

Finally, a more homogeneous grain size and hardness distribution is reported to produce a reduction in distortion, reducing post-machining operations and thereby cost.[2]

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The Changing Face of Carburizing Alloys

If there was one constant in the universe, it was that carburizing alloys did not change. Many considered changing materials a sacred cow, a place where designers dare not go, but this is no longer the case. Today, the need to change materials is a necessity and the heat treater has been thrust onto the leading edge of this new frontier. Let's learn more.

The Lesson of the Past

Historically, materials development has paced the evolution of technology and has been driven by the need to go faster, reach higher, and last longer. Today's performance demands are no different as industries such as Aerospace (Aircraft, Rotorcraft) and Automotive (Car, Truck, Off-Road, Racing) are faced with demands for products to run at higher temperatures, in harsher environments, and in smaller, lighter packages while delivering more performance with higher reliability. New materials have emerged to meet these demands, and the Heat Treater must know how to process them.

Materials Development, Part 1

New carburizing alloys (Table 1) require new carburizing techniques. These materials often require custom designed carburizing cycles to obtain deep case depths in the range of 0.60 to 0.250 in. (1.50 to 6.35 mm). If specifications allow, reduced processing times can be achieved through the use of high-temperature (1900°F, or 1040°C, and above) carburizing. Quenching in oil and high pressure gas, including gas mixtures, is being used with success.

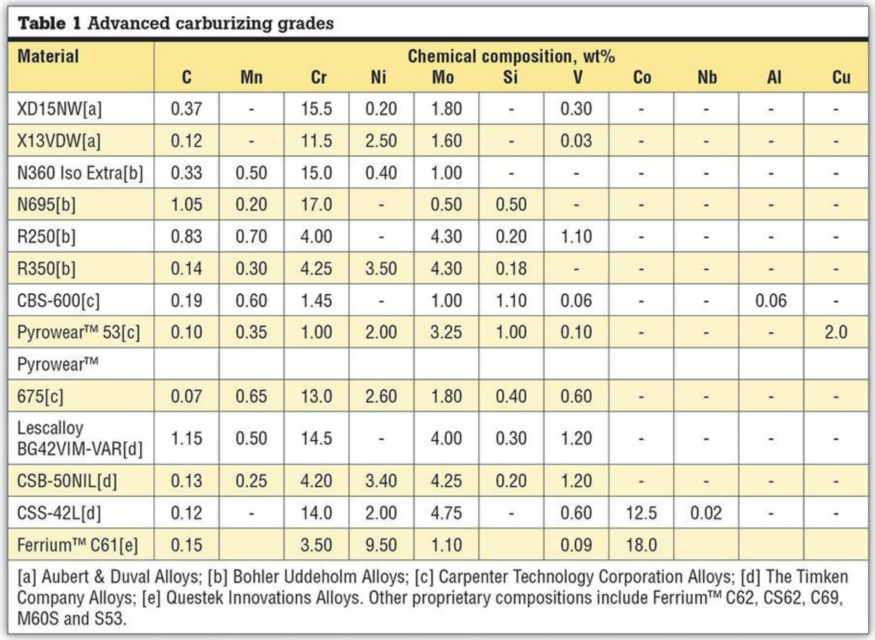

Table 1 Advanced carburizing grades

Material	Chemical composition, wt%										
	C	Mn	Cr	Ni	Mo	Si	V	Co	Nb	Al	Cu
XD15NW[a]	0.37	-	15.5	0.20	1.80	-	0.30	-	-	-	-
X13VDW[a]	0.12	-	11.5	2.50	1.60	-	0.03	-	-	-	-
N360 Iso Extra[b]	0.33	0.50	15.0	0.40	1.00	-	-	-	-	-	-
N695[b]	1.05	0.20	17.0	-	0.50	0.50	-	-	-	-	-
R250[b]	0.83	0.70	4.00	-	4.30	0.20	1.10	-	-	-	-
R350[b]	0.14	0.30	4.25	3.50	4.30	0.18	-	-	-	-	-
CBS-600[c]	0.19	0.60	1.45	-	1.00	1.10	0.06	-	-	0.06	-
Pyrowear™ 53[c]	0.10	0.35	1.00	2.00	3.25	1.00	0.10	-	-	-	2.0
Pyrowear™											
675[c]	0.07	0.65	13.0	2.60	1.80	0.40	0.60	-	-	-	-
Lescalloy BG42VIM-VAR[d]	1.15	0.50	14.5	-	4.00	0.30	1.20	-	-	-	-
CSB-50NIL[d]	0.13	0.25	4.20	3.40	4.25	0.20	1.20	-	-	-	-
CSS-42L[d]	0.12	-	14.0	2.00	4.75	-	0.60	12.5	0.02	-	-
Ferrium™ C61[e]	0.15		3.50	9.50	1.10		0.09	18.0			

[a] Aubert & Duval Alloys; [b] Bohler Uddeholm Alloys; [c] Carpenter Technology Corporation Alloys; [d] The Timken Company Alloys; [e] Questek Innovations Alloys. Other proprietary compositions include Ferrium™ C62, CS62, C69, M60S and S53.

Fig. 1. New carburizing steels are being used increasingly in the racing industry with excellent results [2]

For example, Pyrowear™ 675 is a relatively new carburizing stainless steel alloy with good corrosion, heat and fatigue resistance developed primarily for use in bearing and gear applications in the aerospace industry. It is vacuum induction melted and vacuum arc remelted (VIM/VAR) to meet aircraftquality cleanliness. Test results show it has corrosion resistance similar to that of AISI Type 440C stainless, and its rolling fatigue resistance is superior to that of M50Nil. Pyrowear 675 maintains a 60 HRC case hardness at operating temperatures to 400°F (205°C). Impact and fracture toughness are comparable or superior to those of conventional non-corrosion resistant carburizing steels such as SAE 8620 and 9310.

Other new alloy steels (Table 2) offer the possibility of lower unit cost by stock allowance savings, achieving higher productivity through reduced cycle times and reduced part distortion, while achieving substantial improvements in fatigue and wear performance well beyond the capabilities of conventional carburizing.

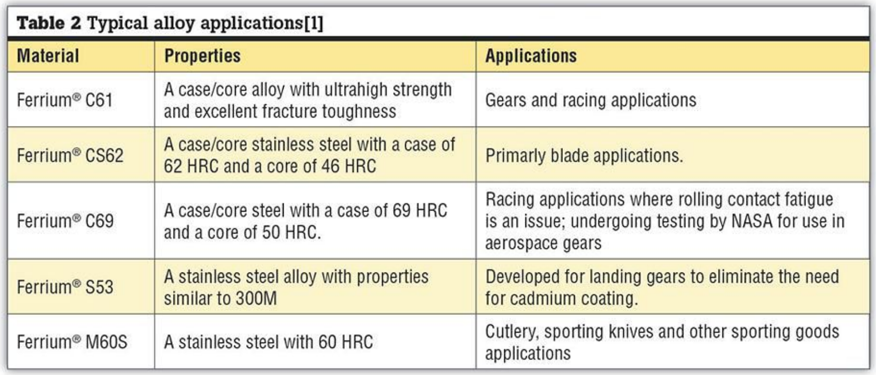

Table 2 Typical alloy applications[1]

Material	Properties	Applications
Ferrium® C61	A case/core alloy with ultrahigh strength and excellent fracture toughness	Gears and racing applications
Ferrium® CS62	A case/core stainless steel with a case of 62 HRC and a core of 46 HRC	Primarly blade applications.
Ferrium® C69	A case/core steel with a case of 69 HRC and a core of 50 HRC.	Racing applications where rolling contact fatigue is an issue; undergoing testing by NASA for use in aerospace gears
Ferrium® S53	A stainless steel alloy with properties similar to 300M	Developed for landing gears to eliminate the need for cadmium coating.
Ferrium® M60S	A stainless steel with 60 HRC	Cutlery, sporting knives and other sporting goods applications

Materials Development, Part 2

Popular steels in the 41xx, 51xx and 86xx families are being modified with additions of manganese, chromium and silicon to improve hardenability. Material cost is being offset by savings in manufacturing and heat treating since these modified chemistries allow for increased load sizes and the use of alternative carburizing and quenching methods, such as vacuum carburizing and high gas pressure quenching.

Other chemistry changes involving microalloying additions of titanium, niobium, aluminum and rare earth

Table 3 Racing results for cars and trucks using vacuum carburized parts

Event (through October 2004)	Race results Wins	Pole positions	Top ten finishes
Nextel Cup* (36 races)	22	11	275
Busch Series* (36 races)	22	17	157
Craftsman Truck Series (24 races)	7	6	81

*Including 2004 Nextel Cup and Busch Series Champions

ele ments allow the use of high-temperature carburizing; temperatures as high as 2200°F (1205°C) are being used on production parts.

Leading by Example

One application that illustrates the phenomenal performance enhancements possible by using advanced materials and/or heat treatments is in the racing industry (Fig. 1). Impressive race results (Table 3) have been achieved by vacuum carburizing components allowing increased rear wheel horsepower with equivalent or reduced rotating mass. The selection of the proper material for the application has improved both reliability and performance to such a degree that in drag racing, for example, aluminum parts failing due to excessive heat are now performance limiters.

Going Forward

To continue to meet the challenges ahead, a combination of talents and shared resources are required from heat treaters, steel suppliers, equipment manufacturers and researchers. Today, materials are being engineered by design. For example, in 1998, the President's Office of Science and Technology designated the computational design of materials as one of five critical technologies for the next decade.

Finally, new materials and manufacturing methods will require advanced steelmaking processes, improved steel cleanliness, better grain size and trace element chemistry control and the addition of elements to help bring about the performance for a given application. Advances in heat treatment including vacuum (hardening, carburizing), applied energy (induction, laser) and surface engineering methods (coatings, cladding) need to constantly evolve to meet new performance challenges. Exciting times are ahead in the world of materials and heat treating.

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Hardness and Hardenability Part Two: A Discussion on Hardenability and Hardenability Testing

For the heat treater, the concept of hardenability is often more difficult to grasp than that of hardness. Part of the reason for this is that we seldom perform the tests that measure or predict this property in our shops. The reason why it is important to measure the hardenability of steel is to make sure that we are making the right material choice for a specific engineering application. With the supply of raw material coming from multiple worldwide sources, there is renewed emphasis on predicting how a material will respond to heat treating. Let's learn more.

Hardenability is a material property independent of cooling rate and dependent on chemical composition and grain size. When evaluated by hardness testing, hardenability is defined as the capacity of the material under a given set of heat-treatment conditions to harden "in depth." In other words, hardenability is concerned with the "depth of hardening" or the hardness profile obtained, not the ability to achieve a particular hardness value. When evaluated by microstructural techniques, hardenability is defined as the capacity of the steel to transform partially or completely from austenite to some percentage of martensite at a given depth when cooled under known conditions.[1]

Effects of Alloying Elements

Certain alloying elements have a strong influence on the hardenability and response to heat treatment and ultimately on the ability of the product to perform its intended function. The main alloying elements that affect hardenability are carbon, boron, chromium, manganese, molybdenum, nickel and silicon.[2]

Carbon

Increasing the carbon content increases both the hardenability and hardness (Fig. 1) of steels by retarding the formation of pearlite and ferrite and encouraging the formation of martensite at slower cooling rates. It is more common to control hardenability, however, with other alloying elements and to use steels with carbon levels of less than about 0.4 wt%. This is due to the fact that while carbon controls the hardness of the martensite, as the carbon levels increase, the critical temperature for the formation of martensite is depressed to lower and lower

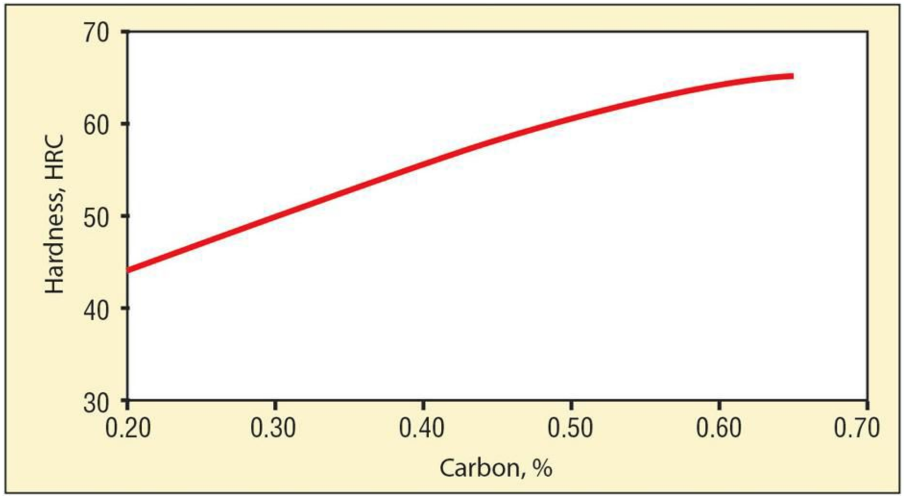

Fig. 1. Effect of carbon content on surface hardness (99% martensite condition)[3]

temperatures, promoting the formation of retained austenite. In addition, high-carbon steels are prone to distortion and cracking during heat treatment and can be difficult to machine in the annealed condition before heat treatment.

Boron

Boron – a very potent alloying element – is extremely effective as a hardening agent and as such has a dynamic impact on hardenability. Boron is typically added to steels in the composition range of 0.0005% to 0.003%. The effect of boron is greatest at lower carbon contents, and it is typically used with low-carbon steels. It does not adversely affect formability or machinability.

Since boron has a very strong affinity for oxygen and nitrogen, failure to tie up free nitrogen during the steelmaking process results in the formation of boron nitrides that will prevent the boron from being available for hardening. Boron must be in solution to affect the hardenability of the steel. Titanium and/or aluminum are added for this purpose. It is important, therefore, that the mill carefully control the titanium/nitrogen ratio. Both titanium and aluminum tend to reduce machinability of the steel. However, the formability typically improves. Boron content in excess of 0.003% has a detrimental effect on impact strength due to grain-boundary precipitation.

Other Alloying Elements

Common alloying elements in steel are chromium, manganese, molybdenum, nickel, silicon and vanadium. These elements retard the phase transformation from austenite, and the complex interactions between them affect the temperatures of the phase transformation and the resultant microstructure. To assist the metallurgist and heat treater, alloy-steel compositions are often described in terms of their "carbon equivalent," which describes the magnitude of the effect of all the elements on

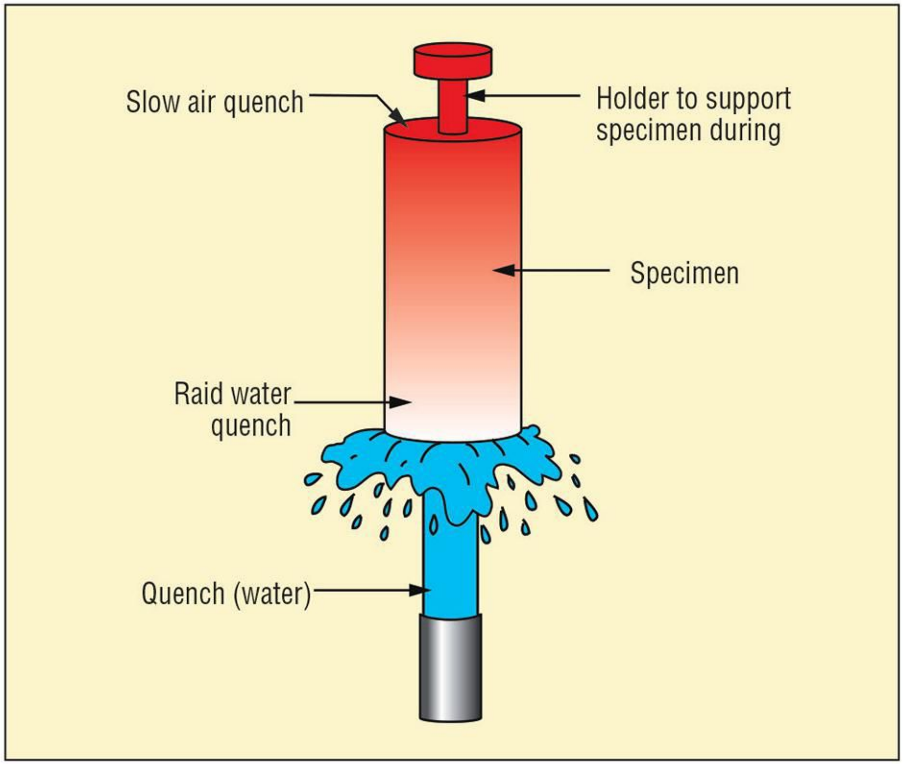

Fig. 2. Jominy test setup[3]

hardenability. Steels of the same carbon equivalent have similar hardenability. A commonly used formula for calculating the equivalent carbon content (E_c) is given below.

$$E_c = \%C + \left[\frac{\%Mn + \%Si}{6}\right] + \left[\frac{\%Cr + \%Mo + \%V}{5}\right] + \left[\frac{\%Cu + \%Ni}{15}\right]$$

Finally, attention should be paid to trace-element chemistry as many tramp elements (e.g., titanium, niobium and aluminum) influence a material's response to heat treatment.

Effect of Microstructure

As the austenite grain size increases so too does the hardenability of the steel. The steelmaking process controls the initial grain size (fine or coarse) by the use of such additions as aluminum. During heat treatment, however, the size of the austenite grains increases with the length of time above the steel's critical temperature and with higher temperatures. A larger (coarser) austenite grain size retards the rate of the ferrite/pearlite phase transformation. Since a substantial increase in hardenability only occurs at high austenitizing temperatures, one must be concerned about the resultant properties of the steel. Fine-grain steels promote a somewhat greater toughness and shock resistance and have less of a tendency to crack in heat treatment. Coarse-grain steels tend to harden more deeply.

Jominy and Other Hardenability Tests

Several test methods have been developed over the years to determine the hardenability of steel. Most of us have heard of the Jominy end-quench test (Fig. 2).

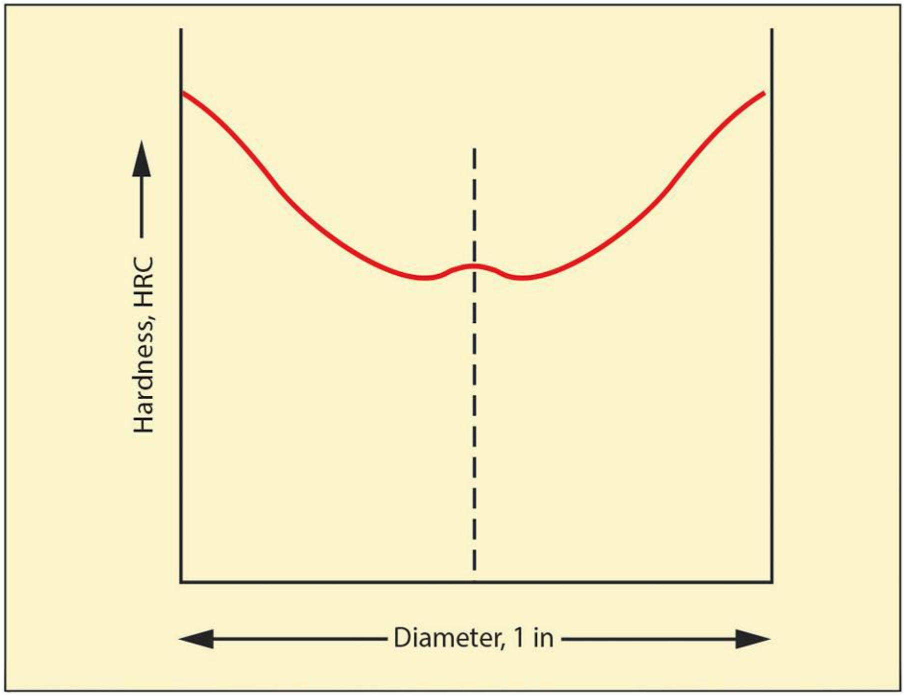

Fig. 3. Schematic representation of the S-A-C hardenability curve[4]

Another is the S-A-C test, which is often applied to steel of low hardenability. The advantage of the S-A-C test lies in the fact that the three numbers give a good visual image of the hardness distribution curve. These tests were developed as alternatives to the creation of continuous cooling transformation (CCT) diagrams.

The test sample for the S-A-C test is a cylinder with a length of 140 mm (5.5 inches) and a diameter of 25.4 mm (1 inch). After normalizing and austenitizing, the specimen is quenched into a water bath. A cylinder 25.4-mm long is cut from the test specimen, and the end faces are ground to remove any tempering effects induced by the cutting operation. Rockwell C hardness measurements are then made at four positions on the original cylinder face, and the average hardness provides the surface, or S-value. Rockwell testing is carried out along the cross section of the specimen from the surface to the center to develop a hardness profile (Fig. 3). The total area under the curve provides the area, or A-value, in units of "Rockwell-inch," and the hardness at the center gives the C-value.

Material Certification Sheets

Most people need not be experts in all the intricate details of hardenability or the test to determine hardenability. However, it is important that we pay attention to the material certification sheets provided with every lot of material we are treating. Understanding what this information is telling us about the material chemistry, grain size and hardenability provides invaluable information on how we must heat treat the material and how it will respond to our heat-treatment methods.

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Fundamentals of Heat Treating: Ideal Diameter

A quantitative measure of a steel's hardenability is expressed by its DI, or ideal diameter, value. This abbreviation comes from the French phrase "diamètre idéal" and refers to the largest diameter of steel bar that can be quenched to produce 50% martensite in its center (Fig. 1). The quench rate of the bar is assumed to be infinitely fast on the outside; that is, it has sufficient quench severity so the heat removal rate is controlled by the thermal diffusivity of the metal and not the heat transfer rate from the steel to the quenchant. Typically, water or brine provides these infinitely fast quench conditions. The larger the ideal diameter value, the more hardenable is the steel.

DI values are an excellent means of comparing the relative hardenability of two materials as well as determining if it is possible to harden a particular cross section (or ruling section) of a given steel. DI values are influenced by the hardenability (chemical composition) of a material, the grain size and the severity of quench.

It is important to note that hardness in steel is determined by carbon content while hardenability is determined by the alloy chemistry, which also includes carbon. Elements such as copper and vanadium play a large role in hardenability. Also, a number of customers today are using boron as an inexpensive, yet effective element, to increase hardenability. Therefore, DI values vary greatly depending on material, from less than one inch (25 mm) for say an AISI 1045 steel to over of 7 in. (178 mm) for an AISI 4140 steel.

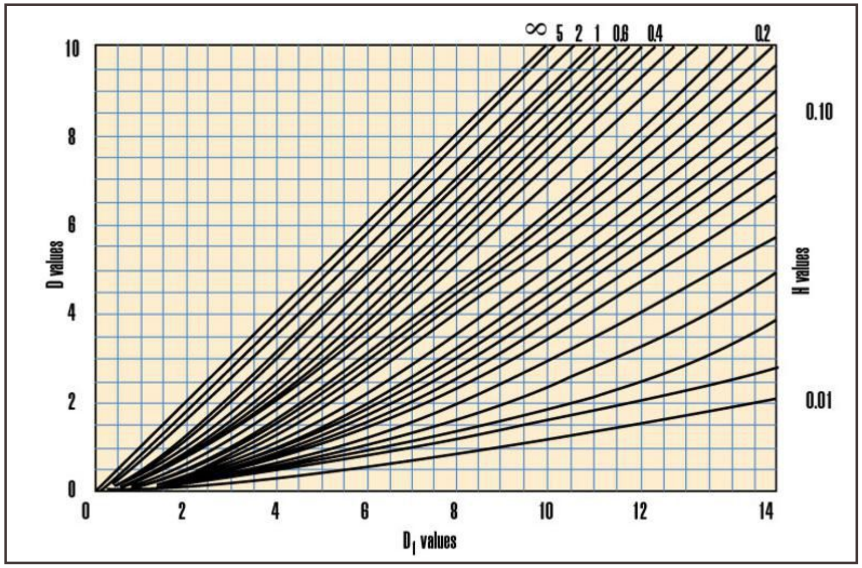

Fig. 1. Relationship between actual critical diameter, or D (the largest size bar that, after being quenched in a given medium, contains greater than 50% martensite) and ideal diameter, or DI (size of bar hardened to 50% martensite in a "perfect" quench medium) for determining the quench severity (H). In a perfect quenchant, the surface of the bar is assumed to cool instantaneously to the temperature of the quenching medium.

A more hardenable steel allows the heat treater to form martensite even in the middle of a thick bar (where cooling rates are slow), whereas a less hardenable steel can achieve a martensitic structure only in thin sections, where the cooling rates are fast. Steels that form martensite in thicker sections are those in which the nucleation and growth of ferrite and cementite is slow and can be avoided. Many solute additions slow down the formation of ferrite, as do larger grain sizes (e.g., ASTM 6 vs. ASTM 8) because fewer grain boundaries are present to act as ferrite nucleation sites. Therefore, alloy additions and grain-coarsening steps are used to make steel more hardenable.

While DI values are calculated for cylindrical bars, the hardenability of various product shapes can be estimated using DI values for equivalent rounds; cooling rates are influenced by surface-to-volume ratios. The hardness of steel having varying amounts of martensite can then be determined by means of a Jominy hardenability test. Jominy data can also be used to estimate the quench severity necessary to produce a given hardness distribution in a particular steel.

Calculating the Ideal Diameter

Ideal diameter can be calculated fairly precisely if you know the austenite grain size and alloying elements present in the steel of interest. ASTM Specification A255 (Standard Test Method for Determining Hardenability of Steel) assumes a grain size of 7 in its calculation because, statistically, most steels are close to this grain size value.

Let's calculate the DI value for a given steel with an ASTM No. 8 grain size and a chemical composition of 0.30%C, 0.20%Si, 0.40%Mn, 0.15%Ni, 0.95%Cr and 0.15%Mo. The ideal diameter is calculated using Equation (1) with individual values listed in Table 1. For elements other than carbon, locate the element percentage in the carbon column and then read across to find the alloy factor under the appropriate element listed.

$$\text{DI} = \text{DI Jominy} \times f\text{Mn} \times f\text{Si} \times f\text{Ni} \times f\text{Cr} \times f\text{Mo} \qquad \text{(Eq 1)}$$

Our calculation then becomes

$$\text{DI} = 0.17 \times 1.14 \times 2.33 \times 1.055 \times 3.052 \times 1.45 = 2.10 \text{ in. } (53.34 \text{ mm}) \qquad \text{(Eq 2)}$$

Thus, it is not possible to harden a cylindrical cross section or equivalent round representing an actual shape greater than 2.1 in. (53 mm) and still obtain at least 50% martensite in the core, no matter what the quenchant is!

Table 1 Alloy factors for calculating ideal diameter [4]

% C	Base ideal diameter (DI Jominy) for ASTM grain size No. shown			Alloy factor (fx): x = element				
	No. 6	No. 7	No. 8	Mn	Si	Ni	Cr	Mo
0.05	0.0814	0.0750	0.0697	1.167	1.035	1.018	1.108	1.150
0.10	0.1153	0.1065	0.0995	1.333	1.070	1.036	1.216	1.300
0.15	0.1413	0.1315	0.1212	1.500	1.105	1.055	1.324	1.450
0.20	0.1623	0.1509	0.1400	1.667	1.140	1.073	1.432	1.600
0.25	0.1820	0.1678	0.1560	1.833	1.175	1.091	1.540	1.750
0.30	0.1991	0.1849	0.1700	2.000	1.210	1.109	1.648	1.900
0.35	0.2154	0.2000	0.1842	2.167	1.245	1.128	1.756	2.050
0.40	0.2300	0.2130	0.1976	2.333	1.280	1.146	1.840	2.200
0.45	0.2440	0.2259	0.2090	2.500	1.315	1.164	1.972	2.350
0.50	0.2580	0.2380	0.2200	2.667	1.350	1.182	2.080	2.500
0.55	0.2730	0.2510	0.2310	2.833	1.385	1.201	2.188	2.650
0.60	0.2840	0.2620	0.2410	3.000	1.420	1.219	2.296	2.800
0.65	0.2950	0.2730	0.2551	3.167	1.455	1.237	2.404	2.950
0.70	0.3060	0.2830	0.2600	3.333	1.490	1.255	2.512	3.100
0.75	0.3160	0.2930	0.2700	3.500	1.525	1.273	2.620	3.250
0.80	0.3260	0.3030	0.2780	3.667	1.560	1.291	2.728	3.400
0.85	0.3360	0.3120	0.2870	3.833	1.595	1.309	2.836	3.550
0.90	0.3460	0.3210	0.2960	4.000	1.630	1.321	2.944	3.700
0.95	-	-	-	4.167	1.665	1.345	3.052	-
1.00	-	-	-	4.333	1.700	1.364	3.160	-

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Remember that the DI calculation is empirical. There are many different DI equations with each giving slightly different answers so companies often use more than one.

Practical Examples

A carburizing grade of steel was found to have low core hardness after final machining. However, this low hardness condition affected less than 20% of the thousands of parts manufactured. All of the material complied with the material specification for chemistry and Jominy hardness at the J8 location. Analysis of DI values calculated from the mill heat chemical composition for each batch predicted which batches were most likely to display low core hardness. A procedure was then developed to check steel mill heat lots for the subject parts prior to purchase of raw material to assure that a minimum DI value is maintained.

In another example, a gear manufacturer found it necessary to continually make size adjustments prior to carburizing in an attempt to control distortion and compensate for lot-to-lot material variation. The steel, while meeting specification requirements, was found to be at the extremes of the control range. For this material, typical "H" band steel at two points of Jominy control (J4 and J8) had a range of 12 to 14 HRC from high to low. Even "RH" (restricted hardenability) steel was found to have a 7 to 8 HRC hardness difference at the J4 and J8 positions. The solution was to specify DI values in a narrow control range, and the result was predictable part growth after carburizing and the elimination of a costly manufacturing step.

Where Do We Go From Here?

Today, to better predict lot-to-lot distortion, manufacturers are specifying (and steelmakers are producing) material to extremely tight DI ranges (e.g., 2.0 to 2.3) to ensure achieving size control on critical parts instead of trying to hold a tighter Jominy hardenability range. This is especially the case if no post heat treat operations will be performed. Ideal diameter is an invaluable tool in the heat treater's arsenal.

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Stainless Steels Part Two: Heat Treatment Techniques

Heat treating of stainless steels depends to a great extent on the type (wrought or cast) and grade of stainless steel, as well as the reason for the treatment, most often to ensure that the properties altered during fabrication are restored (e.g. corrosion resistance, ductility, or hardness) so that the stainless steel component can perform in its intended service environment. There are quite a variety of different heat treatments available. Let's learn more.

Cleaning

An often-overlooked step, cleaning is necessary before any heat-treating operations are performed on stainless steels to remove oils, grease, and other types of residue. Left on the stainless steel surface during heat treating, localized carburization may occur degrading the corrosion resistance or other properties of the material.

Annealing

A number of different annealing methods (full, isothermal, subcritical) are commonly used for stainless steel. Austenitic stainless steels cannot be hardened by heat treatment, but they do harden by cold working. Annealing not only allows recrystallization of the work hardened grains but also places chromium carbides (precipitated at grain boundaries) back into solution. Annealing can also be used for homogenization of castings or welds, and to relieve stresses from cold working. Annealing temperatures usually are above 1900°F (1040°C), although some stainless steel types may be annealed at closely controlled temperatures as low as 1850°F (1010°C) when fine grain size is desired. Time at temperature is often kept short to minimize surface oxidation and to control grain growth, which may lead to a surface phenomenon called "orange peel."

Quench Annealing

Annealing of austenitic stainless steel is occasionally called quench annealing because the metal must be cooled rapidly through the temperature range of 1900°F (1040°C) to below 1100°F (600°C), and preferably below 900°F (480°C), to prevent precipitation of carbides at the grain boundaries (sensitization). The exception is.

for stabilized and extra-low carbon grades. This can be achieved by very rapid fan accelerated gas or water quenching.

Subcritical (Process) Annealing

Subcritical annealing is recommended for all applications that do not require maximum softness. All martensitic and most ferritic stainless steels can be subcritical annealed by heating into the upper part of the ferrite temperature range or full annealed by heating above the critical temperature into the austenite range followed by slow cooling. Typical temperatures are between 1400°F (760°C) and 1525°F (830°C) for subcritical annealing. When material has been previously heated above the critical temperature, such as in hot working, at least some martensite is present, even in ferritic stainless steels (e.g. 430). Relatively slow cooling at 75°F (25°C) per hour from full annealing temperature, or holding for one (1) hour or more at subcritical annealing temperature, is required to produce the desired soft structure of ferrite and spheroidized carbides. However, parts that have undergone only cold working after full annealing can be subcritically annealed satisfactorily in less than 30 minutes.

The ferritic types retain predominantly single-phase structures throughout the working temperature range (e.g. 409, 442, 446) and require only short recrystallization annealing in the range 1400°F (760°C) - 1750°F (955°C).

Repeated Process (Stabilize) Annealing

A stabilize anneal is sometimes performed after conventional annealing for grades containin titanium or niobium (e.g. 321 and 347). Most of the carbon content is combined with titanium in grade 321 or with niobium in grade 347 when these are annealed in the usual manner.

A second anneal between 1600°F (870°C) and 1650°F (900°C), for several hours followed by rapid cooling, precipitates all possible carbon as titanium or niobium carbide and prevents subsequent precipitation of chromium carbide. This special protective treatment is sometimes useful in extremely corrosive service conditions, especially when service also involves temperatures in the 750°F (400°C) to 1600°F (870°C) range.

Bright Annealing

All grades of stainless steels can be bright annealed in highly reducing controlled atmosphere furnaces running under hydrogen, dissociated ammonia, or nitrogen/hydrogen atmospheres at dew points less than -60°F (-50°C) or in vacuum furnaces to prevent or minimize surface oxidation. Martensitic grades (depending on carbon content) and even some ferritic grades are susceptible to hydrogen embrittlement.

Hardening, Quenching, and Tempering

Martensitic stainless steels are hardened by austenitizing, quenching and tempering much like low alloy steels. Austenitizing temperatures between 1800°F (980°C) and 1850°F (1010°C) are typical. As-quenched hardness increases with austenitizing temperature to about 1800°F (980°C) and then decreases due to the presence of retained austenite. For some grades, the optimum austenitizing temperature may depend on the subsequent tempering temperature.

A slow heating rate, or preheating before austenitizing, is recommended to prevent cracking in high carbon grades (e.g. 440C) and in intricate sections of low carbon types. Preheating at 1450°F (790°C), followed by heating to the austenitizing temperature, is a common practice.

Air cooling from the austenitizing temperature is usually adequate to produce full hardness, but oil quenching is sometimes used, particularly for larger sections. Parts should be tempered as soon as they have cooled to room temperature, particularly if oil quenching has been used, to avoid delayed cracking. Tempering at temperatures above 950°F (510°C should be followed by relatively rapid cooling to below 750°F (400°C) to avoid embrittlement at 885°F (475°C).

Precipitation-hardening, martensitic grades (e.g. 17-4PH, 13-8Mo) typically require full annealing followed by austenite conditioning, transformation cooling, and age (precipitation) hardening.Semiaustenitic precipitation-hardening types (e.g. 17-7PH, 15-7PH) may require annealing, trigger annealing (to condition austenite for transformation on cooling to room temperature), sub-zero cooling (to complete the transformation of austenite), and aging (to fully harden the alloy). Hardening improves strength and toughness and typically takes place in the 900°F (480°C) to 1150°F (620°C) range.

Sub-Zero Treatment

Stainless steel components can be cryogenically treated before tempering to transform retained austenite, particularly where dimensional stability is important (e.g. 440C). Temperatures in the range of -100°F (-75°C) to -150°F (-100°C) are common, and deep cooling below -300°F (-185°C) is being used.

Stress Relief

Austenitic stainless steels are typically heated between 800°F (425°C) and 1700°F (925°C) to achieve an adequate stress relief. One (1) hour at 1600°F (870°C) typically relieves about 85% of the residual stresses. Stress relieving in this temperature range, however, can also precipitate grain boundary carbides, resulting in sensitization that severely impairs corrosion resistance. To avoid these effects, it is strongly recommended that a stabilized stainless steel (e.g. 321 or 347) or an extra-low-carbon type (e.g. 304L or 316L) be used, particularly if extended stress relief times are required.

Stress relieving reduces residual stresses, avoids stress corrosion cracking, improves notch toughness, and improves dimensional stability in service. Full solution treatment (annealing), generally by heating to about 1975°F (1080°C) followed by rapid cooling, removes all residual stresses, but is not a practical treatment for most large or complex fabrications. A great deal of stainless steel is welded during fabrication. When full annealing is not possible, such as on large components or intricate shapes, weldments can be heated to an intermediate temperature to decrease high residual stresses Stress relieving is performed when joining dissimilar metals such as austenitic stainless steel and low alloy steel. Stress relief of martensitic or ferritic stainless steel weldments will temper heat affected zones and restore some corrosion resistance.

Low Temperature Stress Relief

When austenitic stainless steels have been cold worked to develop high strength, low-temperature stress relieving will increase the proportional limit and yield strength (particularly compressive yield strength). Low temperature stress relief in the 325°F (160°C) to 775°F (415°C) range will reduce residual stress with little or no effect on the corrosion resistance and/or mechanical properties. Temperatures up to 800°F (425°C) may be used if resistance to intergranular corrosion is not required.

Carburizing and Nitriding

Low-temperature nitriding and carburizing processes have been developed for austenitic stainless steels and are rapidly gaining acceptance for improving resistance to wear and corrosion. The soft matrix hardness limits these processes in heavily loaded applications.

Coatings

The most commonly applied coating is titanium nitride (TiN), which is used both for improved surface durability and for aesthetic purposes, having an attractive gold color and mirror-like finish.

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Tool Steel Heat Treatment

For anyone involved with heat treating of tool steels, it is critical to remember that there is no such thing as acceptable shortcuts in the heat-treatment process. As such, applying best practices for preheating, austenitizing, quenching, deep-freezing and tempering is mandatory. Let's learn more.

Preheating

Slow heating rates and appropriate preheat steps for tool steels provide multiple benefits. First, most tool steels are sensitive to thermal shock, and reducing thermal gradients produced by rapid heat rates minimizes the tendency of tool steels to crack. Also, tool steels undergo a volume change when they transform from their annealed microstructure to austenite while heating to elevated temperature. If this volume change occurs non-uniformly, it can cause unexpected distortion, especially in cases where differences in section size exist.

For most tool steels, select a preheat temperature just below the material's critical transformation temperature (Ac_1) and hold long enough to allow the full cross section to reach a uniform temperature. Another preheat and hold just above the critical temperature allows the material volume change to occur uniformly, creating less distortion. Preheating should be followed by rapid heating to austenitizing temperature.

Austenitizing

The purpose of austenitizing is to allow carbide particles to partially or fully dissolve and diffuse into the matrix. Different types of carbides dissolve at different rates as a function of temperature, thus the appropriate austenitizing temperature depends primarily on the chemical composition of the steel. Also, the austenitizing temperature may be varied slightly to tailor the resulting properties to specific applications.

In general, higher temperatures allow more alloy to diffuse, permitting slightly higher hardness and strength. At lower temperatures less alloy diffuses, and the resulting matrix is tougher and less brittle, although it may not develop as high a hardness.

Soak times at austenitizing temperature are usually extremely short – in the neighborhood of one to five minutes once the tool has reached temperature. Often load thermocouples are placed inside parts or in representative cross sections – the soak time being initiated once the center of the part has reached temperature. The optimum

Fig. 1. M-series, high-speed steel reamers and blanks *(Photo courtesy of The Yankee Corp., Fairfax, Vt., www.yankeereamer.com)*

combination of properties is often obtained at the lowest hardening temperature that will produce adequate hardness for the intended application.

Quenching

After the alloy content has been redistributed during austenitizing, the steel must be cooled fast enough to transform to martensite. Most tool steels (Fig. 1) actually develop a martensitic structure in the temperature range of 600°F (315°C) to 200°F (95°C). How fast a tool steel must be cooled, and in what type of quench medium to fully harden, depends on the chemical composition. Higher-alloy tool steels develop fully hardened properties with a slower quench rate. As a rule, use the slowest quench rate appropriate to develop an optimized part microstructure and hardness while minimizing distortion and the risk of cracking.

For the higher alloyed tool steels processed over 2000°F (1095°C), the quench rate from about 1800°F (980°C) to below 1200°F (650°C) is critical for optimum heat-treat response and material toughness.

No matter how tool steels are quenched, the resulting martensitic structure is extremely brittle and under great stress. If put into service in this condition, there is a significant risk that the tool will fail. Some tool steels will spontaneously crack in this condition even if left untouched at room temperature. For this reason, as soon as tool steels have been quenched by any method to handling temperature, around 150°F (65°C), they should be tempered immediately, usually interpreted as within 15–30 minutes.

Deep-Freezing

For most tool steels, retained austenite is highly undesirable since its subsequent conversion to martensite causes a size (volume) increase creating internal stress and leads to premature failure in service. By deep-freezing to -120°F (-85°C) or in some instances cryogenic cooling to -320°F (-195°C), retained austenite is transformed. The newly formed martensite is similar to the original as-quenched structure and must be tempered. Often deep-freezing is performed before tempering due to concerns over cracking, but it is sometimes done between multiple tempers.

Tempering

Tempering is performed both to stress-relieve the brittle martensite that was formed during the quench and to reduce the amount of retained austenite present. Most steels have a fairly wide range of acceptable tempering temperatures. In general, use the highest tempering temperature that will provide the necessary hardness for the tool. The rate of heating to and cooling from the tempering temperature is usually not critical. The material should be allowed to cool below 150°F (65°C) and often completely to room temperature between and after tempers. A good rule of thumb is to soak for one hour per inch of thickest section after the entire tool has reached temperature, but in no case less than two hours regardless of size.

Multiple tempers are typical, especially for many of the more complex tool steels (e.g. M-series and H-series) requiring double or even triple tempering to completely transform retained austenite to martensite. These steels reach maximum hardness after first temper and are designated as secondary hardening steels. The purpose of the second or third temper is to reduce the hardness to the desired working level and to ensure that any new martensite formed as a result of austenite transformation in tempering is effectively tempered.

Other Heat Treatments

Annealing

Tool steels are usually supplied to customers in the annealed condition with typical hardness values around 200-250 Brinell (≈ 20 HRC) to facilitate machining and other operations. This is especially important for forged tools and die blocks where partial or full air hardening takes place, resulting in a buildup of internal stresses. Dies and tools that may need to be rehardened must be annealed.

Full annealing involves heating the steel slowly and uniformly to a temperature above the upper critical temperature (Ac_3) and into the austenite range then holding until complete homogenization occurs. Cooling after heating is carefully controlled at a specific rate as recommended by the steel manufacturer for the grade of tool steel involved. Cooling is normally continued down to around 1000°F (540°C) when the steel may be removed from the furnace and air cooled to room temperature.

Normalizing

The purpose of normalizing is to refine the grains and to ensure that the microstructural constituents are evenly dispersed throughout the matrix. Excessive segregation can lead to poor fracture toughness or distortion in tools due, in part, to segregation and differential transformation rates.

Normalizing involves heating slowly to the normalizing temperature (i.e. in the austenite range), holding at temperature sufficient to allow homogenization to occur and then air cooling to room temperature. Caution must be observed since many tool-steel grades air harden when cooled from austenitizing temperatures.

Stress Relief

In instances where tools have been subject to aggressive machining the build up of internal residual stresses must be removed. Stress relief is carried out at 925 - 1025°F (500 - 550°C) allowing the tools to cool to room temperature prior to heat treatment. Stress relief incorporated as a preheat step is often used.

Common Heat-Treatment Issues

Decarburization

This may occur during all heat-treatment processes (even in vacuum furnaces if leaks exist) and is to be avoided due to subsequent detrimental effect on the hardness of the finished tool (unless removed by machining). The use of vacuum or protective atmospheres will minimize or eliminate decarburization. Other techniques such as the use of a borax or glass coating have also been used.

Size Change

The heat-treat process results in unavoidable size change – either an increase or decrease in dimensions due to changes in the tool microstructure. A combination of variables often contributes, including high alloy content, improper preheats, long soak times, higher-than-necessary austenitizing temperatures, variations in quenching, inadequate cooling between tempers or other factors in the process.

References

1. *IMMA Handbook of Engineering Materials*, 5th Edition.
2. Tarney, Edward, “Heat Treatment of Tool Steels," Crucible Service Centers (www.crucibleservice.com)
3. Roberts, George, and George Krauss and Richard Kennedy, “Tool Steels, 5th Edition," ASM International, 1998.

Frequently Asked Questions about Tool Steel Heat Treating

Tool steel heat treatment is fun! But, like French cooking, everyone seems to want to use a slightly different recipe, and we all know that too many cooks can spoil the soufflé. Let's learn more.

Tool steel heat treatment today is based on a simple premise: To obtain the optimum performance from any given grade, every step of the heat-treating process – including stress relief, preheating, austenitizing, quenching and tempering – must be done correctly.

The selection of any tool steel (Fig. 1) depends on a combination of factors, including component design, application end use and performance expectation. For any given application the goal of heat treating is to develop the ideal microstructure to help us achieve the proper balance of desirable properties: hot (red) hardness, wear resistance, deep hardening and toughness (Table 1).

Frequently Asked Questions

Many questions arise as heat treaters strive for perfection. Here are a few of them:

1. How does the raw material affect the heat treatment?

Chemical non-homogeneity, size and distribution of alloy carbides, grain size, banding, surface decarburization and hardenability response are critical considerations when selecting a raw material. For example, if banding or agglomeration of carbides due to chemical non-homogeneity are present, neither the preheat temperature/time nor the time at austenitizing temperature will resolve this issue, and the service life of the product can be adversely impacted (Fig. 2). It is for this reason that a metallographic examination of the raw material is highly recommended prior to heat treatment.

2. Is one preheat enough?

It is often said that single or double preheating depends on how high the austenitizing temperature is. Preheating is necessary to ensure that the tools are uniformly heated throughout the load, minimize the time at austenitizing temperature and minimize distortion. A common practice is to heat and soak at a preheat temperature just below the lower critical temperature (A_1) of the steel and

Table 1. Effect of alloying elements on tool steel properties[2]

Tool Steel Characteristic[a]	Alloying Element (by potency)
Hot (Red) Hardness	W, Mo, Co (with W or Mo), V, Cr, Mn
Wear Resistance	V, W, Mo, Cr, Mn
Deep Hardening	Mn, Mo, Cr, Si, Ni, V[b]
Toughness[c]	V, W, Mo, Mn, Cr

Notes: [a] Distortion is best managed by additions of Mo (with Cr), Cr and Mn. [b] Tool must be austenitized at a high enough temperature to dissolve the vanadium carbide. [c] Toughness is achieved via grain refinement.

once again somewhere above this temperature after the crystallographic transformation has taken place to relieve these stresses. In the case of large load sizes, preheats can also serve to ensure that the center and outside of the load reach high-heat temperature at or around the same time.

3. What austenitizing temperature do I need, and how long should I stay there?

Heating to the proper austenitizing temperature (to adjust the chemistry of the austenite) and holding at this temperature just long enough (to control grain size and prevent coarsening with subsequent reduced fracture toughness) is key. This varies with each tool steel composition. It is important to be at temperature long enough to dissolve the various alloy carbides present, which go into solution at

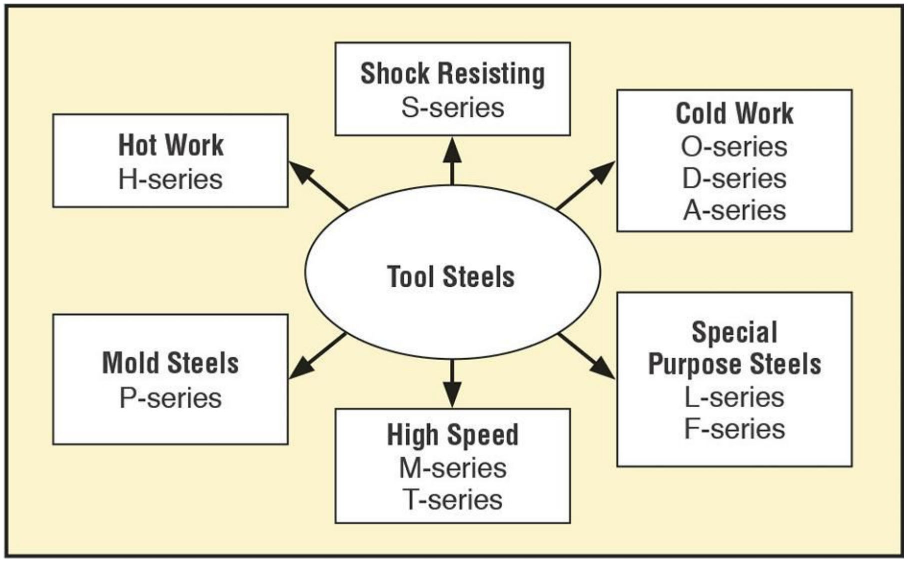


Fig. 1. Tool steel types[1]

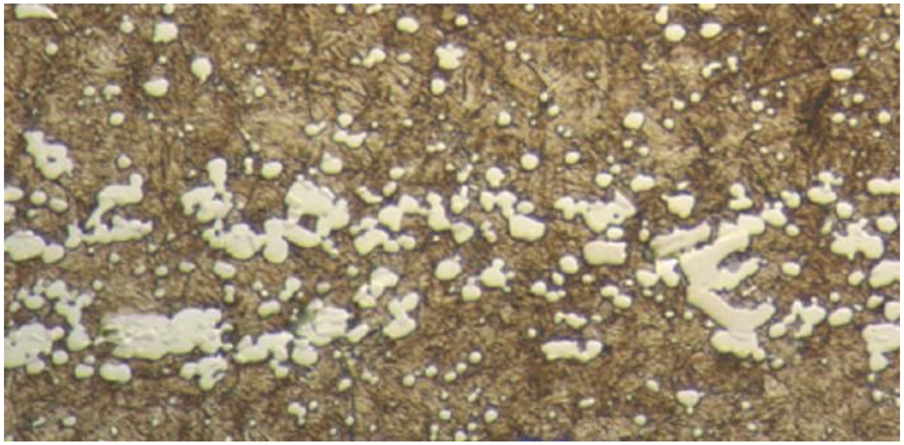


Fig. 2. Large M2 tool steel plate (3/4” thick x 8” wide x 12” long) exhibiting coarse and fine carbides in a martensite matrix – note heavy banding at 1000X. (Photograph courtesy of Aston Metallurgical Services Company, Inc.)[3]

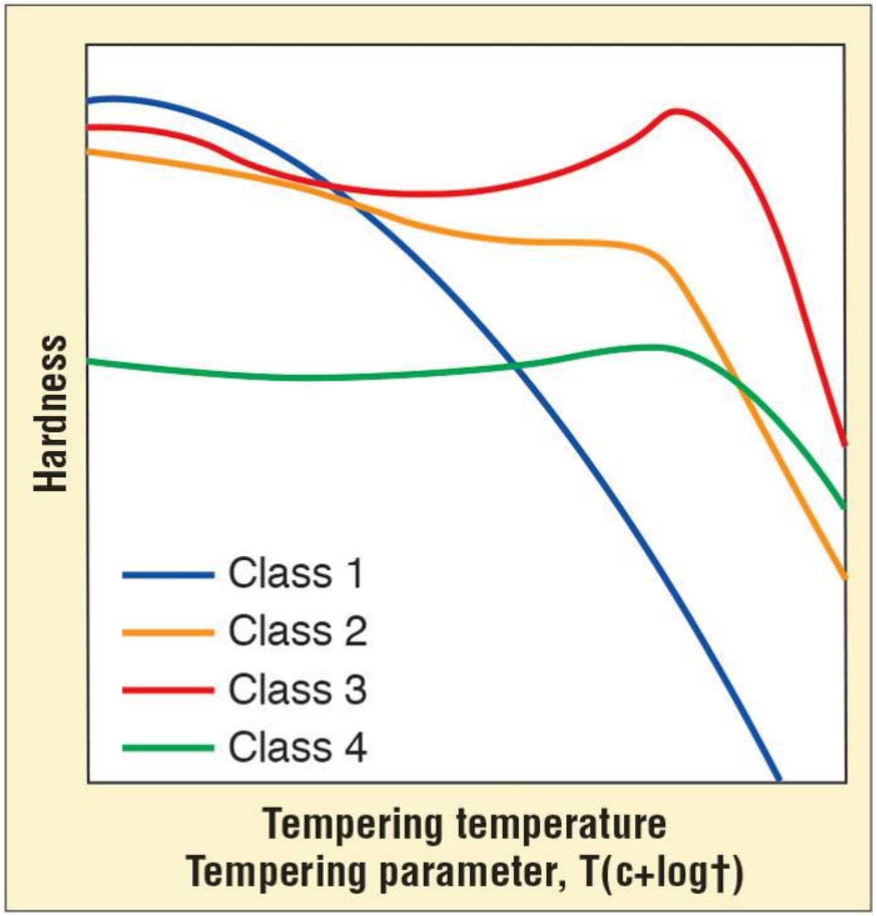


Fig. 3. Tool steel heat-treatment steps[2]

different rates, maximize martensite formation (on quenching) and balance the martensite/retained austenite ratio, but not stay so long as to allow excessive grain growth to occur.

4. What quench medium is the best (and why)?
As long as the rate of cooling achieves transformation to martensite and the critical-cooling velocity suppresses the formation of pearlite, any quench medium can be used. Traditionally, the composition of the steel (alloying elements present) and ruling (thickest) section of the tool have dictated whether water, oil, high-pressure gas, salt or air is needed. Grain size and the degree of carbide solution are important secondary issues.

5. Do all tool steels respond the same way to tempering?
Response to tempering[2] can be divided into a number of characteristic curves, or classes (Fig. 4). For carbon and low-alloy (Class 1) steels, tempering improves certain mechanical properties such as toughness and fracture resistance, except in certain temperature ranges where embrittlement can occur. By contrast, hardness decreases as tempering temperature is raised.

In more highly alloyed tool steels, various precipitation mechanisms produce tempering effects (Fig. 3) such as increased hardness and improved red hardness (i.e. resistant to coarsening due to temper, alloy, carbide dispersion). Medium-to-high alloy cold-work die steels are examples of

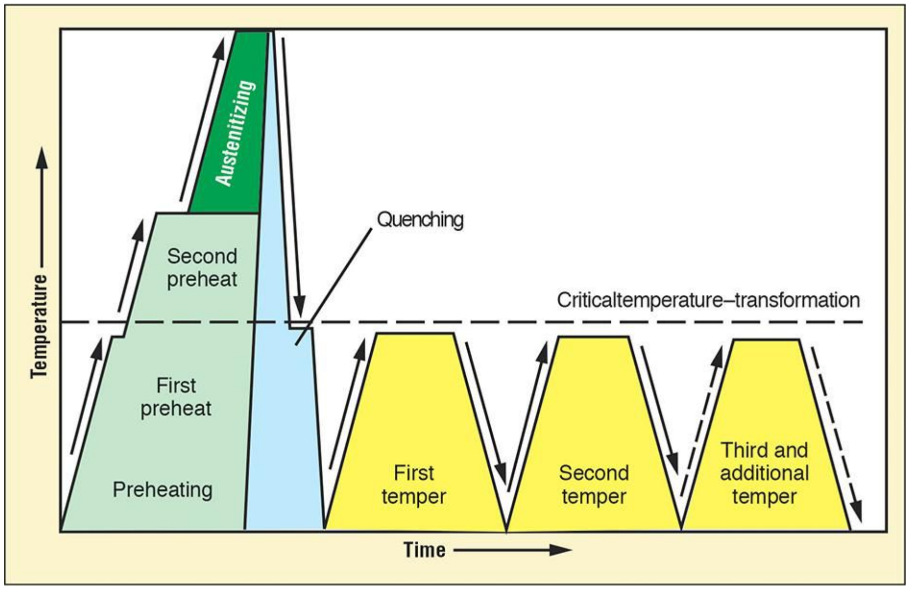


Fig. 4. Characteristic shape of tempering curves by class[2]

Class 2 tool steels in which carbide precipitation and softening are retarded by alloying elements. Steels with low to medium alloy additions fall somewhere between a Class 1 and Class 2.

Highly alloyed high-speed steels are examples of Class 3 tool steels that undergo secondary hardening associated with precipitation of alloy carbides in the tempered martensite matrix. Finally, Class 4 is representative of medium-to-high alloy hot-work die steels in which secondary hardening develops, but overall hardness is lower (than Class 3) due to lower carbon contents in these steels.

6. What does “temper immediately” really mean?

The Heat Treater’s Guide[4], among many other sources, tells us that it is important to “temper immediately” after hardening. One of the most common questions asked is,
“What does this really mean?” Do I have 5 minutes, 15 minutes, an hour, a shift, a day? And what are the consequences of not tempering immediately?

The risks of not tempering a tool within 5-15 minutes of cooling to “handling” temperature, 150˚F (65˚C), are cracking and failure to develop optimum properties. Practically speaking, being able to transfer a load into a tempering furnace within 2-4 hours seems to be a realistic shop-floor practice, but recognize the risk being taken. Oftentimes, there is a temptation to run multiple hardening loads and temper everything at one time so parts sit for a minimum of a shift, perhaps as long as 24 hours. This type of practice can get us into real trouble.

7. How many tempers are enough?

Double, triple or even quadruple tempers are mandatory on some types of tool steels (e.g., M-series, T-series), while single tempers are allowed in other cases (e.g., A-series). A general rule is that tool steels always benefit from multiple tempers. Tempering allows austenite to transform to coarse carbides and converts retained austenite to untempered martensite on cooling. Either of these conditions increases the sensitivity of a tool to brittle fracture. Multiple tempers improve toughness (by spheroidization of coarse carbides) and the tendency toward premature failure (by tempering the martensite).

8. Why do I have to cool down to 150˚F (65˚C) between tempers?

Balancing the important characteristics of tools (wear, hot hardness, toughness, deep hardening) requires completion of the transformation process at each step, and tempering is no exception. In addition, slow cooling minimizes the formation of internal (residual) stress.

9. What is secondary hardening?

Secondary hardening is a consequence of two reactions: the conversion (transformation) of retained austenite to untempered martensite on cooling from tempering temperature and the precipitation of finely dispersed temper (alloy) carbides throughout the matrix. Tungsten (W_2C), molybdenum (Mo_2C), vanadium (VC) and chromium (CrC) carbides are reported to contribute to this rehardening effect.

10. Can I achieve the same microstructure with vacuum hardening as I can with salt?

Advances in vacuum-furnace technology, particularly in the area of high gas-pressure quenching, produce microstructures with the type and distribution of carbides throughout the matrix equivalent to those microstructures traditionally limited to salt-bath heat treating.

11. What are the pros/cons of using salt?

Salt-bath technology has process capability over the full range of tool steel grades. It is capable of achieving temperature uniformity at austenitizing temperature in the range of ±5°F (±3°C). When using salt-bath equipment, the critical issues are:

- Control of operator-induced variability
- Properly maintaining bath chemistry and integrity

Salt Bath Advantages

- The heat-transfer rate between the liquid salt and the steel is in the order of 3-4 times faster than between a radiant or convection (gas) environment and steel.
- Work immersed in salt is "automatically preheated" as a "cocoon" of frozen salt is formed on immersion, which melts fairly rapidly.

- Uniformity of heat transfer (conduction) is more efficient for short process cycle times.

Other noteworthy features:

- Component distortion minimized by the buoyancy effect of the salt (e.g., a component that weighs 10 lbs. [4.5 kg] in air weighs only 3.3 lbs. [1.5 kg] in liquid salt)
- Finer (prior) austenitic grain sizes due in large part to shorter times at preheat temperature
- Use of convection or electro-chemical stirring can achieve temperature variations in the salt bath of ±3.5°F (±2°C)

Salt-Bath Disadvantages

- Limited protection of the tool steel parts from oxidation and decarburization either in the bath or during bath transfers
- Numerous environmental issues (air pollution, water pollution, waste disposal, permits, etc.)
- Operating range of the bath must be controlled by adjusting salt composition
- Control systems (programming, computer controls, etc.) lack sophistication
- High operator dependency for accuracy and repeatability
- High heat discharge to the environment
- Labor intensive
- Maintenance intensive – chemistry and equipment (type and complexity)

12. What are the pros/cons of using vacuum for tool steel heat treating?

Vacuum technology has process capability over the full range of tool steel grades. It is capable of achieving temperature uniformity at austenitizing temperature in the range of ±5°F (±3°C). When using vacuum furnaces, the critical issues are proper design and careful control of:

- Part loading and racking
- Ramp rates
- Preheating temperatures and times
- Austenitizing temperature and time at temperature
- Cooling rates of parts

Vacuum-Furnace Advantages

- Variable heating rates – convection heating in lower (<1600°F/870°C) temperature ranges
- Clean parts (extreme surface finishes), particularly if the parts are gas quenched
- Ability to handle physically large part sizes, complex shapes and heavy load weights
- Flexibility for intermediate heating and cooling

- Superior programming capability and cycle flexibility
- Load documentation (time/temperature cycles)
- Environmental, energy consumption and safety

Vacuum-Furnace Disadvantages

- Initial capital equipment cost
- Slight loss of surface hardness (0.5-1.0 HRC) for certain tool steel grades (Note: this can be overcome but special features/options are required)

References

1. Elgun, Serdar Z., *Tool Steels*, Farmingdale State College (http://info.lu.farmingdale.edu)
2. Roberts, George, George Krauss and Richard Kennedy, *Tool Steels, 5th Edition*, 1998.
3. *Heat Treater's Guide, 2nd Edition*, Chandler, Harry (Ed.), ASM International, 1995.

Induction Heating for the Rest of Us

Curiosity is a wonderful thing. It forces us to question, think and learn. Induction heating is one of those technologies that many of us who don't work regularly in the field are curious about, and it seems that you either understand it or you don't. To many, it's as difficult to learn as a new language. To others, it's pretty basic stuff. As heat treaters, we need to know something about induction heating and how to apply this technology to our needs. Let's learn more.

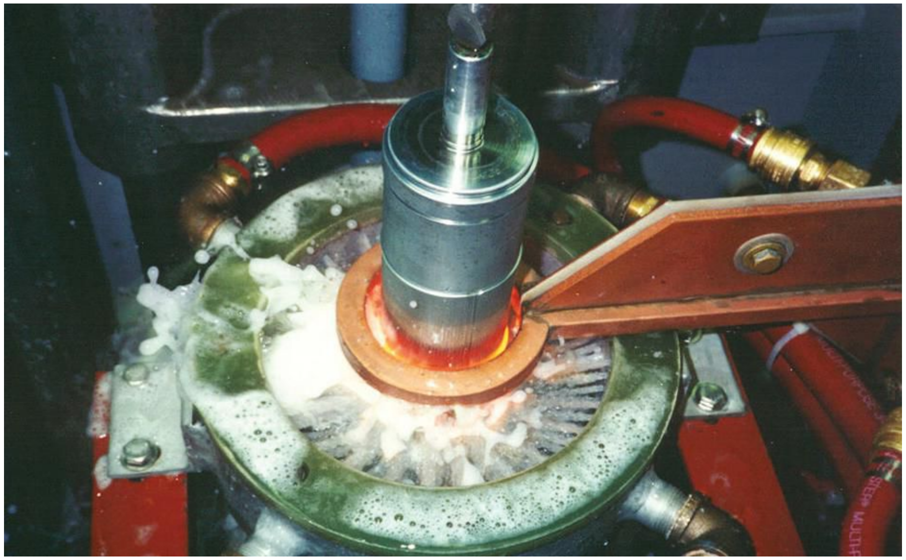


Fig. 1. Induction heating and quenching a rotary die.
(Courtesy of Ajax TOCCO Magnethermic)

What is induction heating?

Induction heating (Fig. 1) is a noncontact heating method; one in which an electrically conductive material (typically a metal) is heated by an alternating magnetic field. Invisible lines of force are created by a work coil when a current flows through it, the result of which is an induced current in the conductive workpiece. Heating results due to the Joule effect and, to a lesser degree, magnetic hysteresis (i.e., power loss other than by eddy currents in a magnetic material caused by reversals of the magnetic field). Joule's Law (Fig. 2 - 3) states that the rate at which heat energy is produced in any part of an electric circuit is measured by the product of the square of the current (I) times the resistance (R) of that part of the circuit.

What are the scientific principals involved in induction heating?

The principle of induction heating is a consequence of the Faraday effect, named for the physicist Michael Faraday who was the first to produce an electric current from a moving magnetic field.

How does induction heating work?

Induction heating is based on the principle of resistance to induced currents. These currents, called eddy currents, are similar in magnitude and opposite in direction to the current produced by the induction coil (also known as the inductor).

A number of other independent variables, including the workpiece's magnetic permeability (a measure of how magnetic the material is), the air gap (coupling distance between the inductor coil and workpiece) and the frequency influence the induction process and its efficiency.

How deep can I penetrate a part using induction?

The depth of (current) penetration during heating depends on the choice of frequency (for an alternating current, frequency is the number of complete cycles per second. The standard unit of frequency is the hertz, abbreviated Hz. For example, if an electric current completes one cycle per second, then the frequency is 1 Hz). The penetration depth is inversely proportional to the square root of frequency. In other words, for applications requiring deep case depth hardening, a low frequency is used.

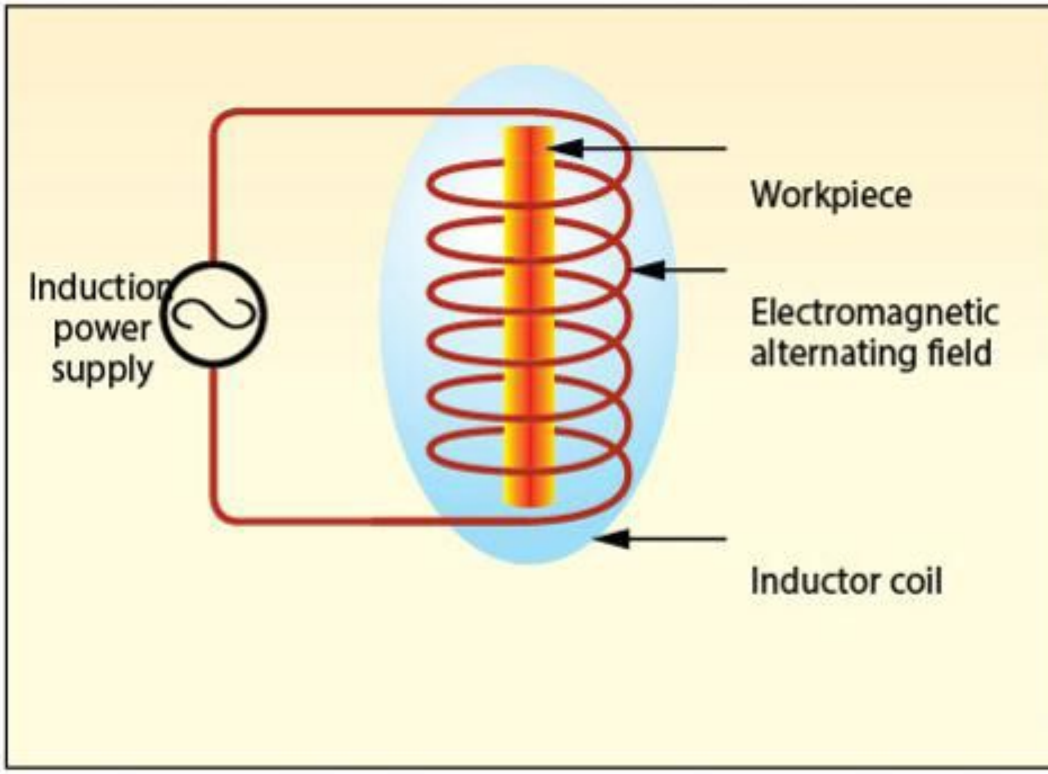
For surface heating or shallow case depth hardening, a high frequency is needed. Depth of heating is also influenced by such variables as the magnetic permeability and conductivity of a part. Finally, it is important to remember that for the same applied frequency, different metals have different penetration depths. For example, at 1 kHz (at ambient temperature), current penetration depth in stainless steel is about six times deeper than in copper.

What are induction coils?

Depending on application and workpiece geometry, the shape of an induction coil can be noticeably different. In some cases, induction coils are simple single-turn or multiturn geometries, where the workpiece is placed inside of the coil. In other cases, induction coil geometries are more complex, such as butterfly-shape, split-return, pancake, etc. Although any current carrying conductor can serve as an induction coil, coils usually are made of copper, which has good electrical conductivity, economy and availability.

Are all parts heated the same?

Different types of parts require different heating modes. For example, gears, camforms, camshafts and axle shafts are typically treated using a single-shot process (where the part is rotated in an inductor for uniform heating, followed by the appropriate quenching process). Longer parts such as shafts, rolls, ballscrews and bar stock are treated progressively, or



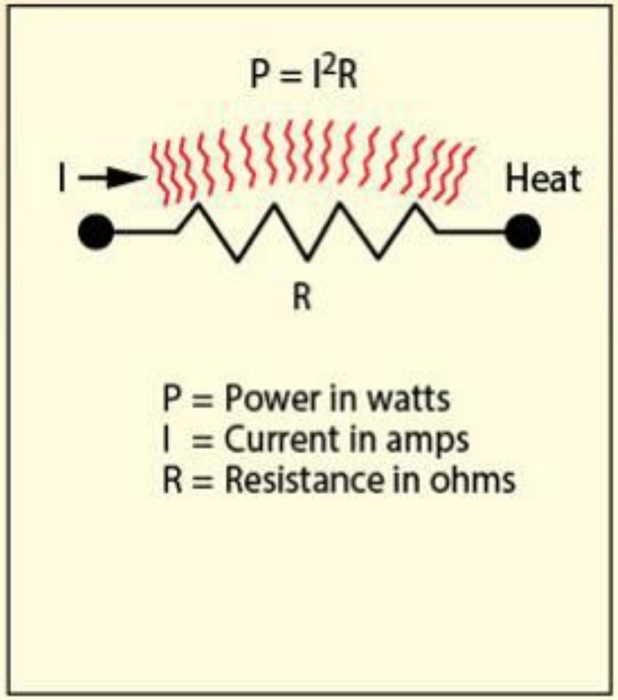


Fig. 2. Induction heating is a noncontact heating method

Fig. 3. Heat energy (E) produced in an electric circuit is equal to $I^2 \times R$.

scanned, where a minimal area is heated to achieve proper temperature, then either the part or inductor moves over the desired heat treat length.

What makes induction heating different from other methods of heating?

In induction heating, heat is generated within the work itself; it does not rely on transmission heat energy by radiation or convective as in a furnace or oven. Therefore, hardening to a specified depth below the surface is possible without excessively long process times or excessively high surface temperatures.

In addition, the heating can be applied to a specified area. Induction processes are accurate, consistent and repeatable. Because heating occurs in the part itself, induction heating is considered a very high (energy) efficiency process (and more efficient than the majority of alternative methods).

What makes up an induction heating system?

An induction heating system comprises a basic induction power source, which provides the required power output at the required power frequency, complete with matching components, an induction coil assembly, a method of material handling and some method of water cooling and quenching. Most induction heating systems are water cooled with the exception of small, low-power units. The methods of material handling and the induction coil arrangement depend entirely on the application. The choice of induction power source is related to the application requirements and to production rate.

What are the most common applications for induction heating?

Typical induction heating applications can be divided into the following general categories:

- Heat treating including hardening, tempering, stress relief, annealing and normalizing
- Mass heating of billet and bar, slab and bloom, strip and plate, wire and cable, tube and pipe and slug heating for semisolid forming
- Joining, brazing, bonding and soldering
- Preheating
- Shrink fitting
- Heating for deformation shaping including forging, swaging, upsetting, bending and piercing
- Melting
- Special applications such as crystal growing, cap sealing, sintering, spheroidizing, carbon vapor deposition, welding, levitation, epitaxial deposition and plasma generation

Where can I go to learn more?

There are a number of reference books on the subject, include the following that should be in everyone's library:

- V. Rudnev, D. Loveless, R. Cook and M. Black, *Handbook of Induction Heating*, Marcel Dekker, 2003
- *Induction Heating for Forging*, FIA Plant Engineering Committee, Forging Industry Association
- S. Zinn, and S.L. Semiatin, *Elements of Induction Heating: Design, Control, and Applications*, ASM International, 1988
- S.L. Semiatin and D.E. Stutz, *Induction Heat Treatment of Steel*, ASM International, 1986
- E.J. Davies and P. Simpson, *Induction Heating Handbook*, McGraw Hill, 1979
- M.G. Lozinskii, *Industrial Applications of Induction Heating*, Pergamon Press, 1969
- C.A. Tudbury, *Basics of Induction Heating, Volume 1*, J.F. Rider, 1960

Other sources of information include OEM manufacturers, commercial heat treaters and technical societies, such as: AISI (www.steel.org), ASM International (www.asminternational.org), AWS (www. aws.org), FIA (www.forging.org), SAE International (www.sae.org), SME (www.sme.org) and WAI (www.wirenet.org).

Summary

Induction heating is an important technology, but is not a solution to every application. There are jobs that can be done better by induction than by any other method, and there are jobs for which induction heating is a poor choice. The key, as with any technology, is to understand its limitations as well as its capabilities. Are you curious yet?

Acknowledgment: The author thanks the following persons for their valuable advise in putting this article together: the late Kelvin Spain; Radyne Corp. (www.radyne.com); Valery Rudnev and Daniel Williams; Inductoheat (www.inductoheat.com); the late George Pfaffmann; the late Fred Specht; Ajax-Tocco-Magnethermic (www.ajaxmag.com).

Principles of Gas Nitriding: The Nitriding Process (Part 1)

Nitriding is a case-hardening process in which nitrogen is introduced into the surface of a ferrous alloy such as steel by holding the metal at a temperature below that at which the crystal structure begins to transform to austenite on heating (Ac_1) as defined by the Iron-Carbon Phase Diagram (Fig. 1). The material typically is placed in contact with ammonia, which allows the transfer of nitrogen to the surface during its thermal decomposition to nitrogen and hydrogen. Other special nitriding processes are also used for certain types of stainless steels involving the decomposition of nitrogen gas at high temperatures, but these will not be the focus of this discussion. Let's learn more.

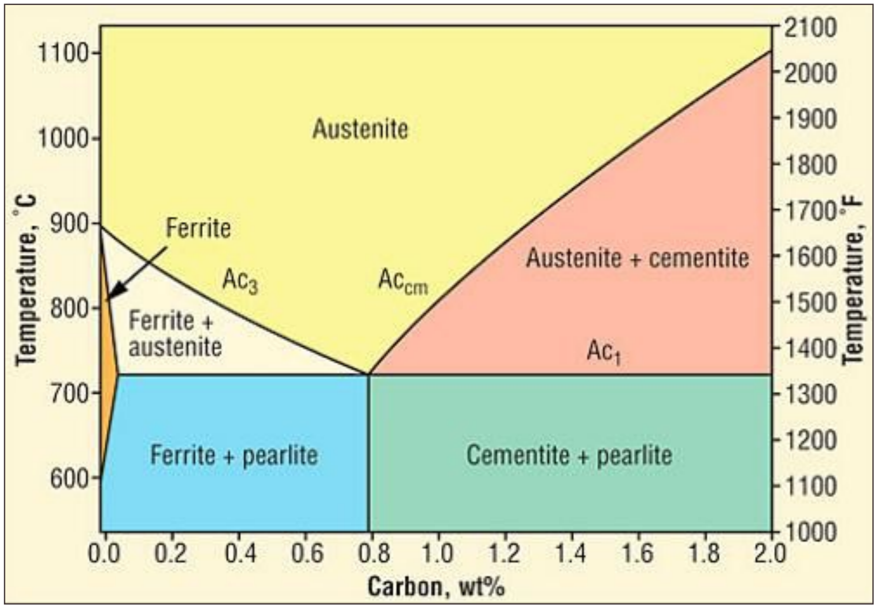


Fig. 1. Simplified iron-iron carbide phase diagram[1]

Several unique features of nitriding are:

- Nitriding is a (relatively) low-temperature process compared to other case-hardening processes (Fig. 2).
- Quenching is not required for a hard case.
- Part distortion is typically less than other case-hardening processes.
- Nitriding is relatively easy to control in terms of process parameters.
- In the gas nitriding process, a crystal structure that is ferritic rather than austenitic is highly desired.

Purpose of Nitriding

Nitriding is a diffusion-related surface treatment (Fig. 3) with the objective to increase surface hardness (among other properties) by the creation of a case on the surface of the part (Fig. 4).

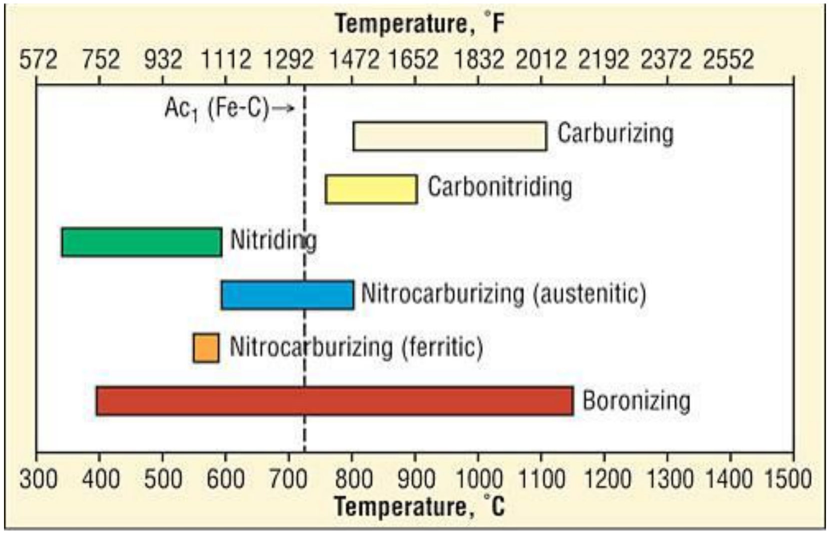


Fig. 2. Temperature for typical case-hardening processes[2]

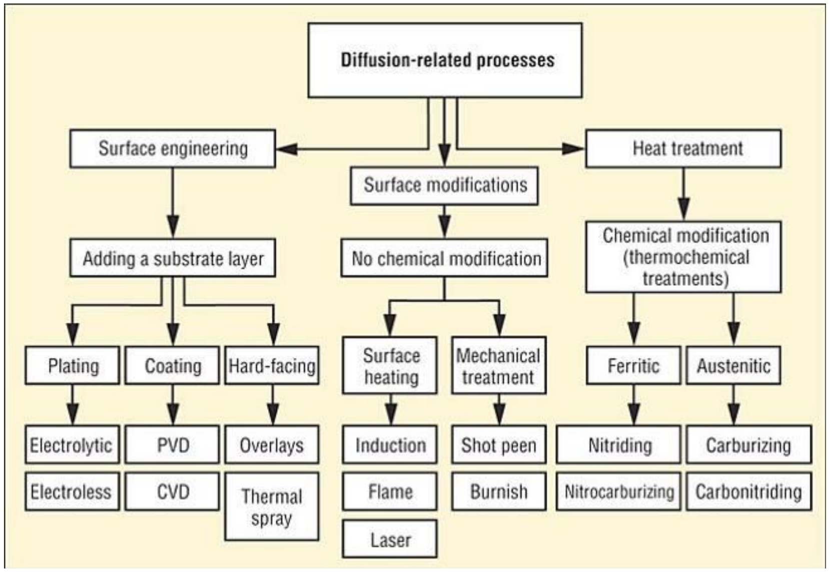


Fig. 3. Diffusion-related processes[2]

One of the appeals of this process is that rapid quenching is not required. Therefore, dimensional changes are kept to a minimum. It is not suitable for all applications. For example, one of its limitations is that the extremely high surface-hardness case is more brittle than that produced by the carburizing process.

A typical manufacturing sequence for gas nitriding (Fig. 5) consists of several heat-treatment steps, including pre-treatments and (optionally) stress relief between machining steps.

Nitriding creates a component that has the following properties:

- High surface hardness (typically > 67 HRC)
- Resistance to wear
- Anti-galling properties (for applications in poor lubrication conditions)
- A minimum of distortion and deformation (less than, for example, carburizing/hardening)
- Resistance to tempering (that is, resistant to softening)
- Stability of the nitrided case
- Improved fatigue life and other fatigue-related properties
- Reduction in notch sensitivity
- Resistance to corrosion (except for 300- series stainless steels)
- Small volumetric changes (some growth does occur)

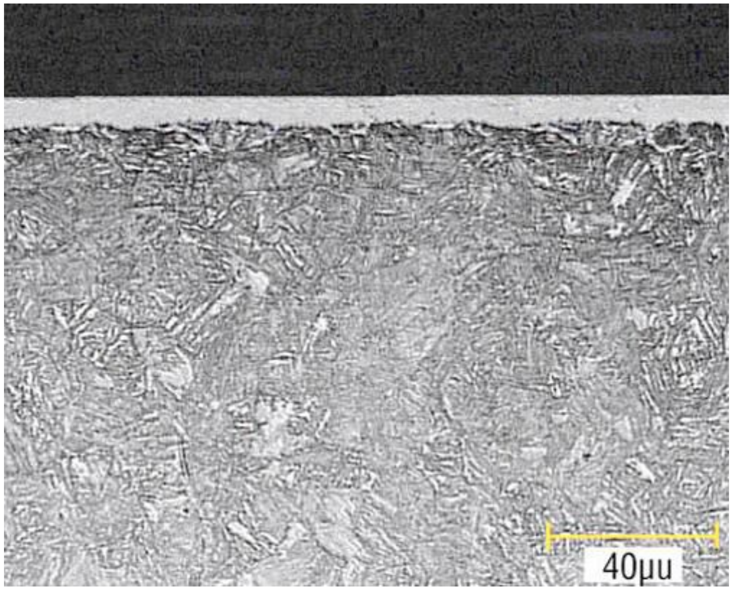


Fig. 4. Typical nitrided case showing a surface compound (white) layer and subsurface diffusion zone[2]

Properties that are considerably improved by nitriding are fatigue strength (resistance to dynamic loading), friction and resistance to wear, and corrosion resistance.

Types of Nitriding

Three methods of nitriding are commonly used in the industry today: gas nitriding (Fig. 6), plasma nitriding (Fig. 7) and salt-bath nitriding (Fig. 8). Each method is unique and has both advantages and limitations. Only gas nitriding will be discussed here.

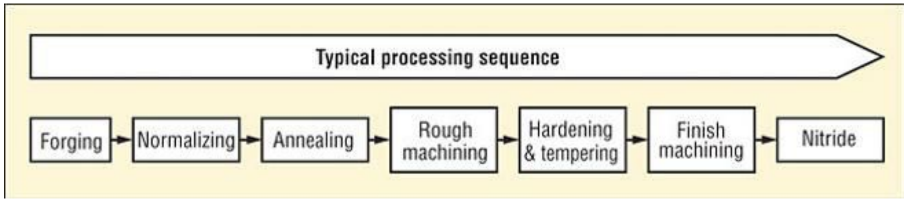


Fig. 5. Typical nitriding manufacturing sequence[2]

Prerequisites for Nitriding

To ensure the best nitriding results, the following precautions and recommendations should be followed. First, the steel should be hardened, quenched and tempered prior to nitriding so as to possess a uniform structure. Tempering temperature has an influence on the hardness of the case as well as the depth of nitriding (Fig. 9). The tempering temperature must be sufficiently high to guarantee structural stability at the nitriding temperature. The minimum tempering temperature should be 50°F (10°C) higher than the maximum temperature to be used for nitriding.

In addition, the following is recommended:

- Before nitriding, the steel must be free from decarburization. Precleaning is mandatory; residue on the parts will result in spotty cases.
- If freedom from distortion is of paramount importance, the internal stresses produced by machining or heat treating should be removed before nitriding by performing a stress-relief

operation, that is, heating to and holding at a temperature of 1000-1300°F (538-705°C) followed by slow cooling.

- Since some growth takes place on nitriding, this should either be allowed for in the final machining or grinding operation prior to nitriding or removed by lapping or careful grinding. If required, the removal of a slight amount of the nitride case should be anticipated in the nitriding case-depth calculation.
- If maximum resistance to corrosion is desired, the parts should be used as processed (with white layer intact).
- Nitrided steels of the Nitralloy type should not be used where resistance to the corrosion of mineral acids is encountered or where resistance to sharp abrasive particles at high velocities is required (as in sand nozzles).
- If straightening is required after nitriding, it should be done hot, if possible, in the temperature range of 1200°F (650°C). Cold-straightening techniques should be carefully reviewed as microcracking is a concern.
- If maximum hardness and maximum resistance to impact are desired, and the question of maximum corrosion resistance is not of vital

Fig. 6. Typical gas-nitrided load

importance, the removal of 0.001-0.002 inch (0.025-0.050 mm) of the nitrided case is desirable. The amount to be removed depends on the original case depth. This operation will remove the most brittle surface layer.

- If nitrided parts exhibit a shiny-gray surface after their removal from the furnace, the results should be viewed with suspicion. Invariably, the case will be shallow and below hardness. The parts should have a matte-gray appearance, although a slight discoloration does not indicate faulty nitriding. The opening of the furnace at too high a temperature or the presence of air leakage on cooling will account for the slight discoloration.

Prior Heat Treatment

In certain alloys, such as the 4100 and 4300 series, hardness of the nitrided case is modified appreciably by core hardness (Fig. 10). Observe that a decrease in core hardness results in a decrease in case hardness. In order to obtain maximum case hardness, these steels are usually provided with maximum core hardness by tempering at the minimum allowable tempering temperature.

All hardenable steels must be hardened and tempered before being nitrided. The minimum tempering temperature is usually at least 50°F (10°C) higher than the maximum temperature to be used in nitriding. Typical tempers range from 1150-1350°F (620-730°C).

Surface Preparation

Nitriding is to be considered a white (clean) glove treatment, that is, all residuals including oils and grit must be cleaned off the surface of the parts prior to nitriding. Even skin oils from handling parts without clean gloves can be problematic. If parts are not absolutely clean, spotty case depths will result.

One acceptable way to clean parts is by vapor degreasing and abrasive (aluminum-oxide grit) cleaning just prior to nitriding.

Another method involves a light phosphate coating. The steps involved are:

- Degrease
- Cold-water rinse
- Oxalic-acid bath dip
- Cold-water rinse
- Warm-water rinse
- Phosphate solution dip

If a decarburized surface is not removed before nitriding, the case will spall very readily.

Fig. 7. Typical plasma-nitrided load

Fig. 8. Typical salt-bath-nitrided load

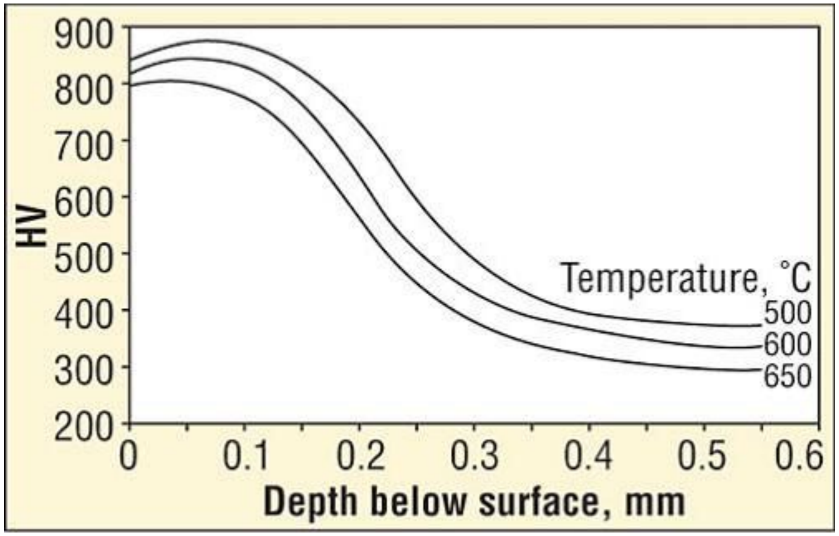


Fig. 9. Typical influence of tempering temperature on hardness and depth of nitrided case[4] (nitrided for 60 hours at 950°F/510°C)

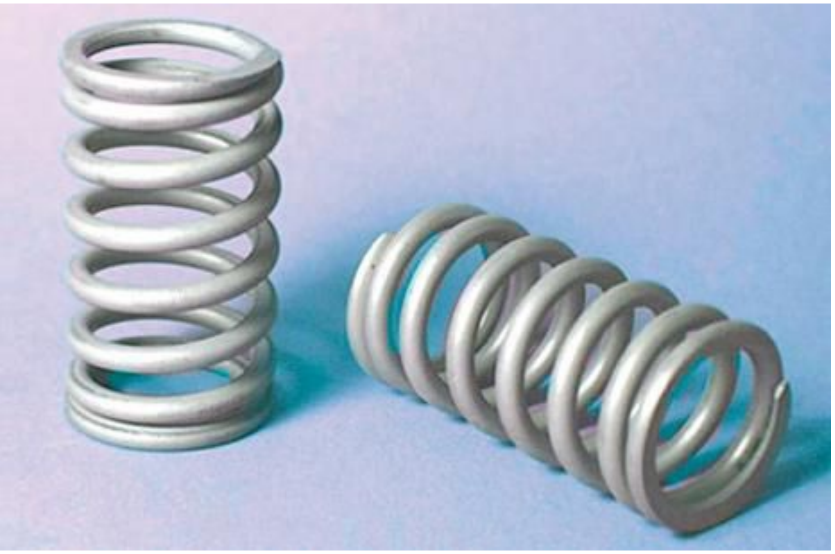


Fig. 11. Surface appearance of nitrided valve springs[5]

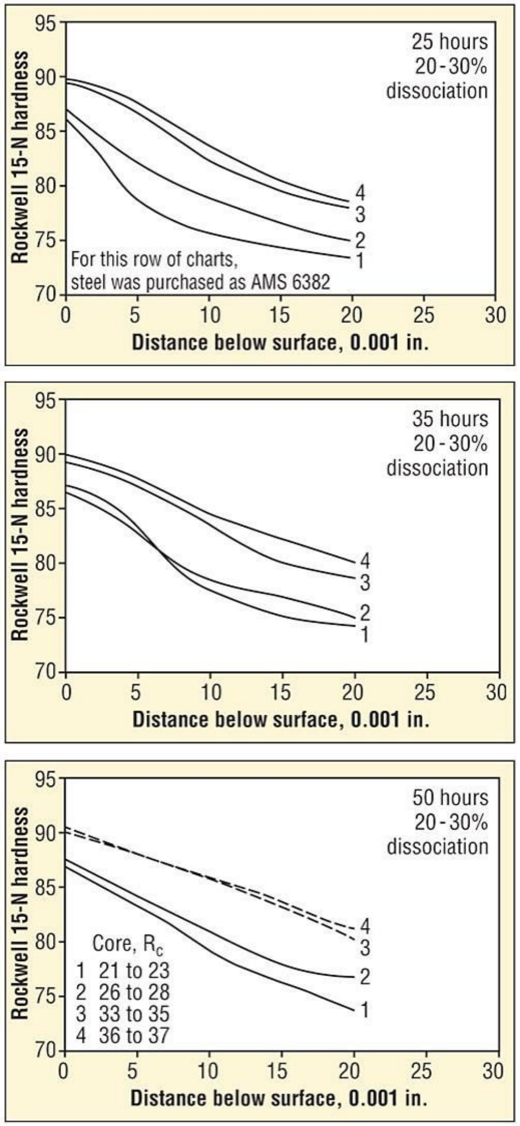


Fig. 10. Case depth as a function of hardness gradient for 4140 steel[12]

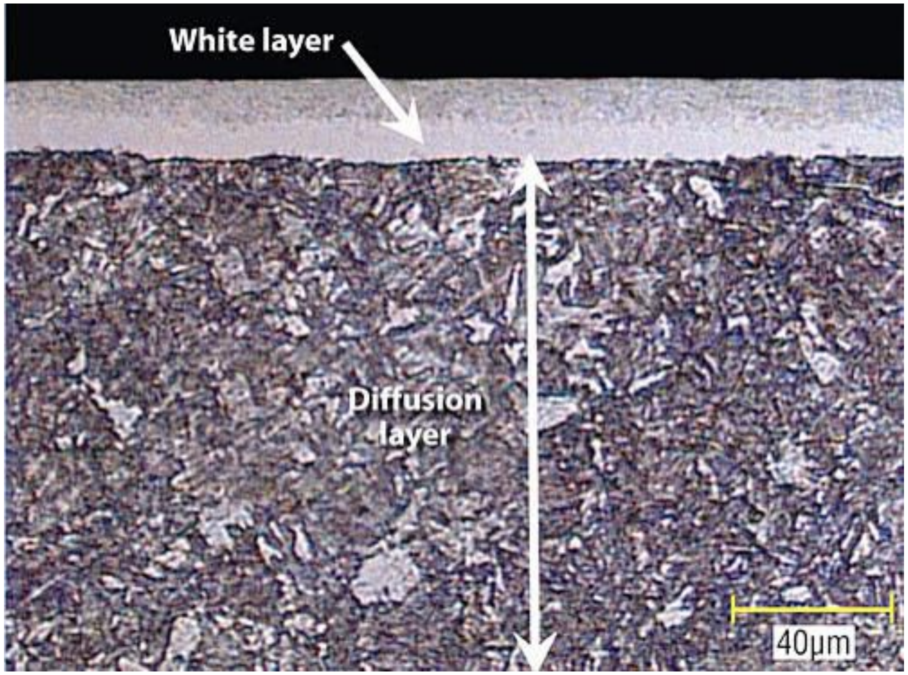


Fig. 12. Nitrided case[5]

Nitriding Results

The following results can be expected from the gas nitriding process:

Surface Appearance

Parts gas nitrided in ammonia should have a dull, matte-gray color (Fig. 11).

Structure of the Nitrided Case

In the nitriding process, the nitrogen that diffuses into the steel reacts with the nitride-forming elements present in solid solution. The hardening results from the reaction. The depth of case depends on how far beneath the steel surface nitrogen is able to diffuse during the nitriding period. The principle involved is that as the alloy elements are removed from solid solution, nitrogen (which is constantly being supplied from the surface) diffuses farther into the alloy and, thus, produces an increasingly deep case. The case depth for any given treatment time and temperature depends upon the amounts of alloy elements with which nitrogen must react before it can diffuse farther.

Nitrogen Concentration

The nitrided medium needs to contain only sufficient active nitrogen to maintain the white layer (Fig. 12). Any increase beyond this point serves to increase the depth of white layer and does not affect the thickness of the inner (diffusion) layer.

Corrosion Resistance

The white layer has excellent corrosion resistance. In certain applications, it does not need to be eliminated.

Dimensional Changes

During nitriding, parts increase slightly in size because of the increase in volume that occurs in the case. This change causes a stretching of the core, which results in tensile stresses in the core that are balanced by compressive stresses in the case after the parts are cooled to room temperature (e.g., notch-sensitivity reduction – a localized surface effect).

Tensile Stresses

Tensile stress originates at imperfections cancelled by compressive stresses.

Growth and Distortion

Dimensional change in nitrided parts are governed largely by composition, tempering temperatures, time/temperature of nitriding, relative thickness of case/core, shape of the part and areas marked off to prevent nitriding. The amount of growth is usually constant for identical parts nitrided in different batches by a fixed processing cycle. After the amount of growth for a particular part has been determined experimentally, allowance for it can be made during final machining (prior to nitriding).

Sharp corners or edges should be avoided on parts to be nitrided, because the projections formed at sharp corners are high in nitrogen content and susceptible to chipping. Sharp edges nitride through the section and are without support from a soft ductile core.

Parts nitrided by the two-stage process and not ground after nitriding have excellent dimensional stability.

Note: See Part 3 of this article for all references.

Principles of Gas Nitriding (Part 2)

Nitriding is a case-hardening process in which nitrogen is introduced into the surface of a ferrous alloy such as steel by holding the metal at a temperature below that at which the crystal structure begins to transform to austenite on heating (Ac_1) as defined by the Iron-Carbon Phase Diagram.

Gas Nitriding Reactions

Gas nitriding is typically done using ammonia with or without dilution of the atmosphere with dissociated ammonia or nitrogen (or nitrogen/hydrogen) in the temperature range of 925-1050°F (500-565°C).Ammonia (NH_3) is allowed to flow over the parts to be hardened.

Due to the temperature and the catalytic effect of the steel surface, the ammonia dissociates into atomic nitrogen and hydrogen in accordance with equation 1:

$$2NH_3 \rightarrow 2N + 6\ H\ (Eq1)$$

This is immediately followed by atomic nitrogen combining to form molecular nitrogen per equation 2:

$$2N + 6\ H \rightarrow N_2 + 3\ H_2\ (Eq2)$$

During the period in which this nitrogen passes through the atomic state, it is capable of being absorbed into the steel (Fig. 13).

So, the entire reaction – equation 3, Figure 14 – becomes:

$$2NH_3 \rightarrow N_2 + 3\ H_2\ (Eq3)$$

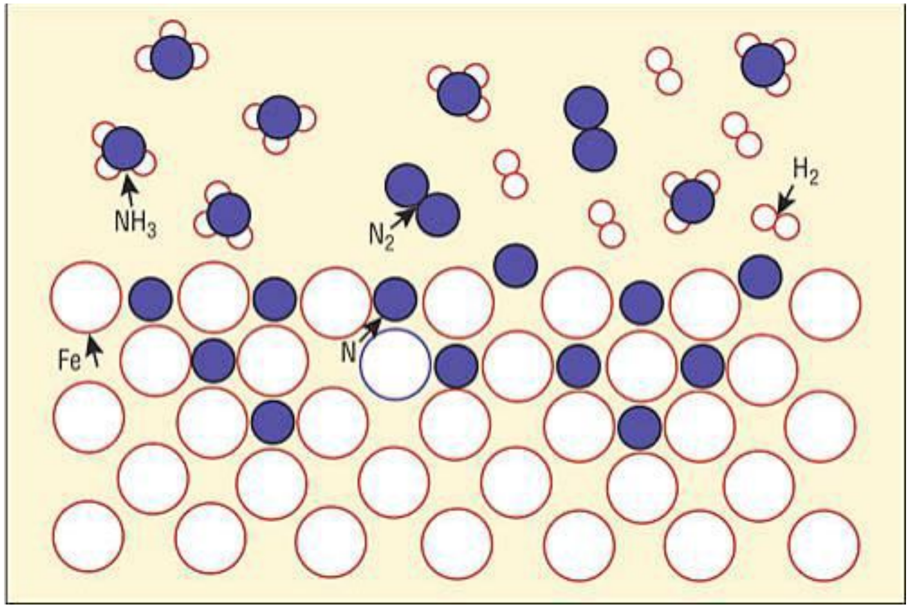


Fig. 13. Dissociation of ammonia and nitrogen pickup in steel during gas nitriding[4]

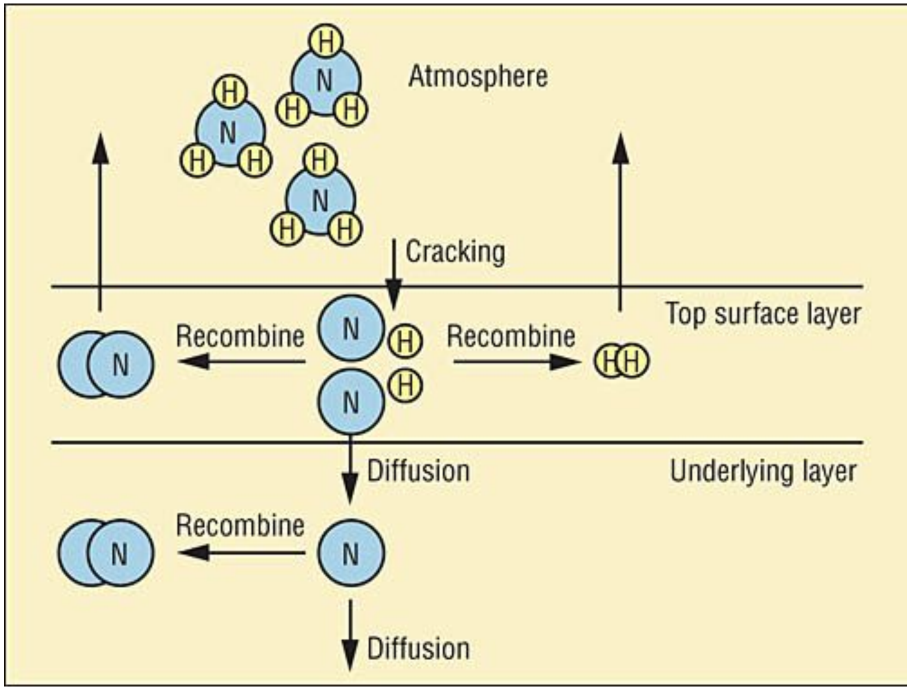


Fig. 14. Nitriding surface and subsurface reactions[5]

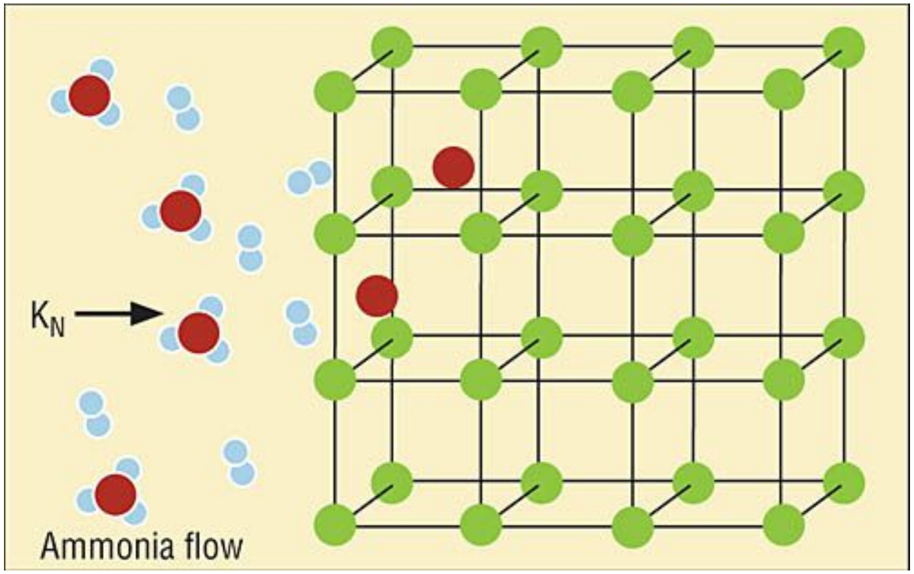


Fig. 15. Schematic representation of ammonia dissociation and nitrogen absorption[5]

Gas Nitriding Activity

In accordance with the laws governing diffusion (Fig. 15), the degree of nitrogen penetration is governed by the temperature and the amount of nitrogen that can penetrate and diffuse into and away from the outer layer of the steel.

In gas nitriding, the nitrogen activity is controlled by the degree of dissociation and the flow rate of the gas (Fig. 15). The nitrogen is supplied by the dissociation of ammonia at the steel surface in accordance with equation 4, a modified form of equation 1.

$NH_3 \rightarrow [N] + 3/2 \text{ volume } H_2$ (Eq4)

By comparison with gas carburizing, the nitriding atmosphere is not in equilibrium since the flow rate of ammonia is too high to allow equilibrium to be achieved.

The amount of ammonia present in the outlet gas is a measure of the degree of dissociation. The higher the flow rates of ammonia, the higher the ammonia percentage in the exiting gas stream and the lower the degree of dissociation. However, a greater percentage of ammonia is present at the surface.

Equation 5 provides an explanation of the nitrogen activity in which the activity constant (a_N) is directly proportional to the degree of ammonia dissociation and the flow rate.

$a_N \alpha a \cdot v$ (Eq5)

where a_N is the activity of atomic nitrogen
a is the degree of dissociation
v is the ammonia flow rate

Consequently, the nitrogen activity is a function of the number of ammonia molecules dissociated at the steel surface per unit of time. At constant pressure and temperature, the degree of dissociation is reduced as the flow rate increases, but the product ($a \cdot v$) increases and so does a_N.

Thus, the nitrogen potential (K_N) derived from equation 4 can be expressed as:

$K_N = pNH_3 / (pH_2)^{3/2}$ (Eq6)

The amount of white layer can be controlled by minimizing the nitriding potential. AMS 2759/10 (Automated Gaseous Nitriding Controlled by Nitriding Potential) indicates nitriding potential values (Table 1) for the various classes of white layer.

Nitrogen potential is also referred to as the nitriding parameter. At a constant temperature, the nitrogen activity, and consequently the nitrogen content, at the surface of the nitrided surface layer are determined by the nitriding potential. The various phases formed are expressed in the Lehrer Diagram (Fig. 16).

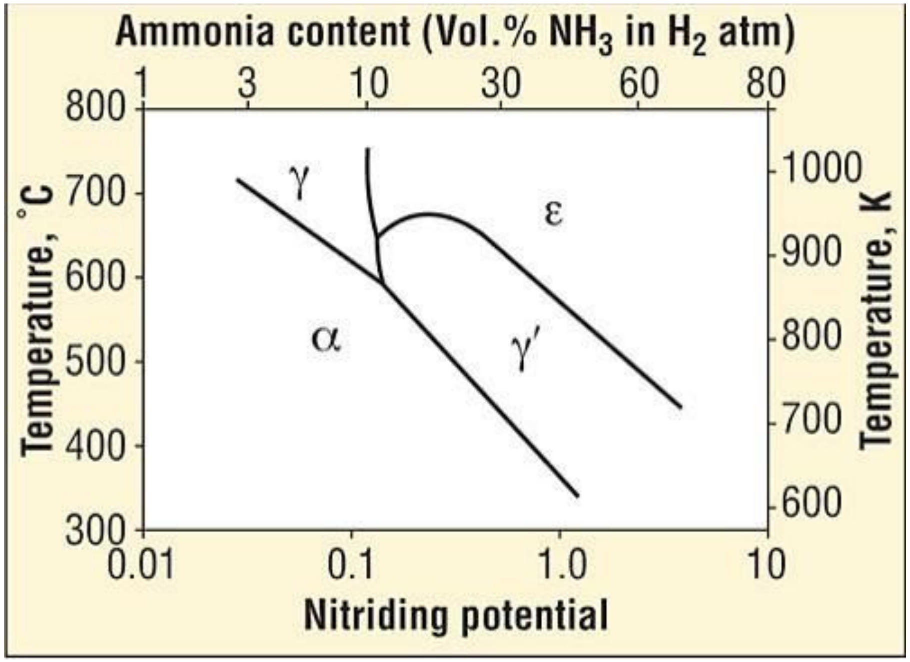


Fig. 16. Lehrer relationship between nitriding potential and the phase formed within the compound layer[4]

It is also important to guarantee that there is an adequate amount of nitrogen available during the process to harden the parts to specification. If there is not enough nitrogen available, the consequence will be low case depths and hardness, with related reductions in physical characteristics.

On the other hand, too much nitrogen at the part surface will result in formation of a brittle and excessively thick white layer, resulting in embrittlement of the nitrided case.

One of the keys to successful nitriding is controlling the percentage of ammonia available per square area of (work) surface that will supply atomic (nascent) nitrogen at the surface. It is important to realize that nitriding is due only to the dissociation of ammonia at the part surface, not due to the presence of molecular nitrogen (N_2) or dissociated ammonia ($N_2 + 3\ H_2$).

The nitriding reaction (Eq. 1, 2) will ultimately go to completion, but this is a very slow reaction. Empirical work has resulted in a rule of thumb that says if the furnace atmosphere is changed four times every hour, the amount of ammonia that is dissociated is 25±10%. An approximate relationship between ammonia flow rate and percentage dissociation exists (Fig. 17). The general shape of the curve will vary as a function of the furnace style, workload size and surface area.

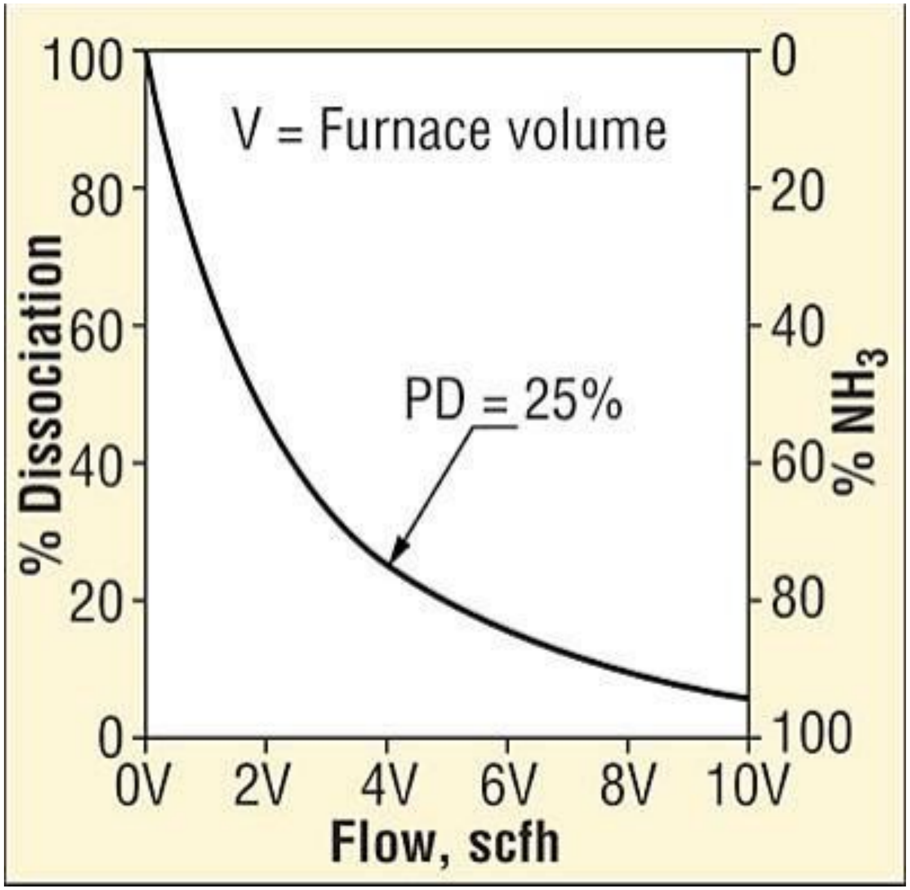


Fig. 17. Percentage dissociation as a function of furnace volume[6]

Hence, the best control method for the process is one that measures and controls the percentage of ammonia. When we talk about a 30% dissociation rate, we normally refer to a concentration of 70% ammonia and 30% dissociated ammonia in the exhaust gas. In reality, due to the volume change involved, only 82.3% is ammonia while 17.7% is dissociated ammonia.

To nitride successfully, an adequate supply of atomic nitrogen must be available at the part surface. Thus, in gas nitriding, it becomes very important to circulate the ammonia in such a way as to constantly resupply the active nitrogen on all areas to be hardened.

Gas Nitriding Cycles and Case-Depth Determination

Two types of nitriding processes are used: the single-stage process and two-stage or Floe (pronounced "flow") process named after its inventor, Dr. Carl Floe.

Case-depth and case-hardness properties vary not only with the duration and type of nitriding being performed but also with steel composition, prior structure and core hardness. Case depths are typically 0.008-0.025 inches (0.20-0.65 mm) and take 10-80 hours to produce.

Single-Stage Nitriding Process

In the single-stage process, a temperature range of 925-975°F (500-525°C) is typical. The dissociation rate of ammonia into nitrogen and hydrogen ranges from 15-30%. The process produces a brittle, nitrogen-rich layer known as the "white layer" (compound zone) at the surface and is comprised of various iron nitrides (FeN, Fe_4N, $Fe_{16}N_2$).

Two-Stage Floe Process (U.S. Patent No. 2,437,249)

The two-stage process was developed to reduce the amount of white layer formed by single-stage nitriding. The first stage is, except for time, the same as that of the single-stage process. In the second stage, however, the addition of a dilutant gas (dissociated ammonia or nitrogen) increases the percent dissociation to around 65-85%. The temperature is typically raised to 1025-1075°F (550-575°C), and the result is the reduction of the depth of the white layer, producing a deeper case of slightly lower hardness. If the two-stage method is used, it is frequently possible to meet dimensional tolerances without any final grinding operation.

Dissociated ammonia is generally required for high second-stage dissociation (otherwise erratic control may result), and it is commonly used as a dilutant (to change the percentage per square area that NH_3 molecules are exposed to). In some cases, nitrogen is used. However, white-layer control and porosity can be affected. Loading arrangement and the use of a furnace circulating fan are very important so that a high dissociation level may be achieved. The nitrogen potential varies with the composition of the gas mixture that is being sent into the furnace.

Table 5. K_N ranges

Class	Nitriding potential ranges, K_N	
	1st Stage	2nd Stage
0	4 – 15	0.2 – 0.8
1	4 – 15	0.4 – 2.6
2	4 – 15	1.2 – 5.5

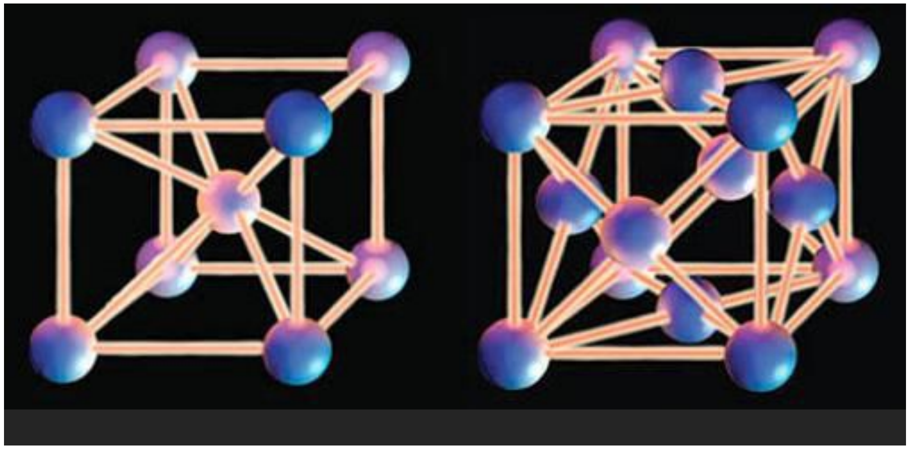


Fig. 18. Body-centered cubic (bcc) crystal structure; Face-centered cubic (fcc) crystal structure

Crystal (Lattice) Structure

Ferrite, or α iron, which is a body-centered cubic (BCC) in crystal structure (Fig. 18), dissolves 0.001% nitrogen at room temperature and 0.115% nitrogen at 1095°F (590°C). γ, or Fe_4N, has a face-centered cubic (FCC) crystal structure (Fig. 20) and dissolves 5.7-6.1% nitrogen. Fe_2N and Fe_3N are called ε, which has a hexagonal closed packed (HCP) crystal structure and dissolves between 8.0% and 11.0% nitrogen.

Control of the Nitriding Process

There are several methods of controlling the nitriding process based on analysis of the percentage of dissociation.

One method involves the use of an ammonia analyzer (Fig. 19), which is tied into ammonia and

Fig. 19. Ammonia control system[7] (courtesy of Super Systems Inc.)

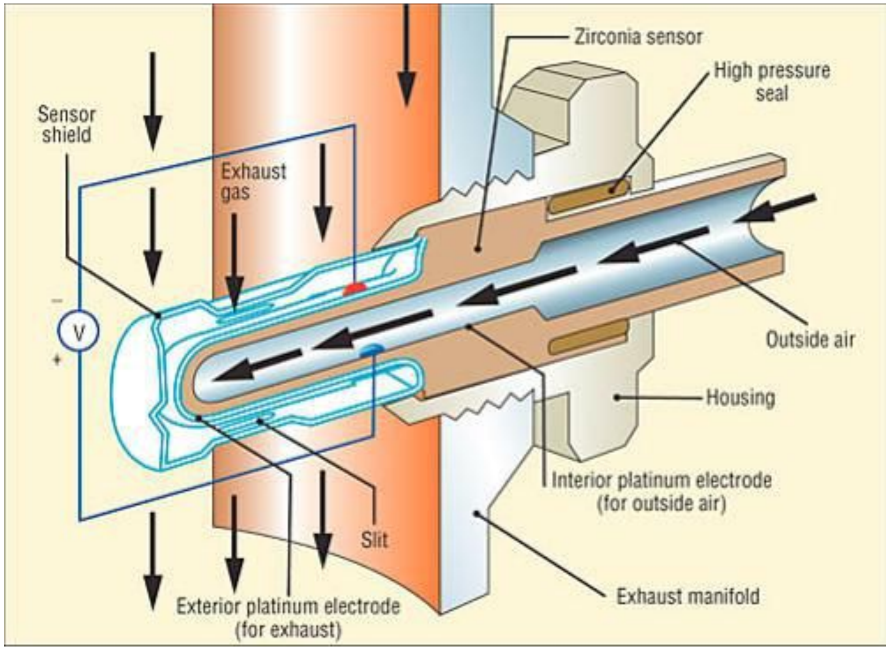


Fig. 20. Anatomy of a hydrogen sensor[5]

dissociated ammonia (or nitrogen) flowmeters (for use during the second stage of nitriding). Based on the output from the ammonia analyzer, the process can be accurately controlled.

Another method used to measure the degree of dissociation is an analysis of the amount of hydrogen in the exhaust gas (Fig. 20). From equation 4 we see:

1 volume $NH_3 \rightarrow 1/2$ volume $N_2 + 3/2$ volume H_2
(Eq7)

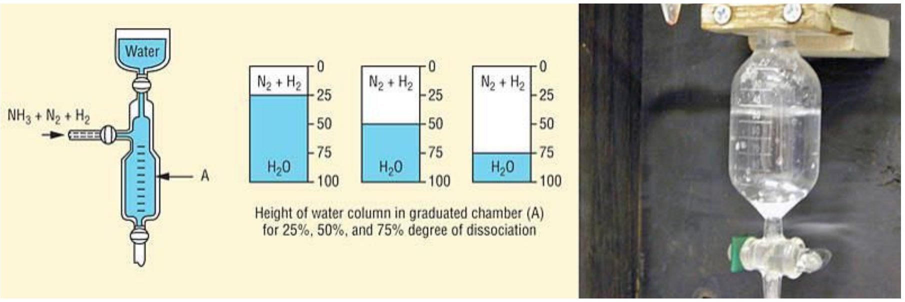


Fig. 21. Manual measurement of percentage dissociation[8]

For example, if the measured volume percentage of hydrogen is 30%, the volume percentage nitrogen is 10% (30/3), and the remaining ammonia volume is 60% (100% – 30% – 10%). Given the original volume of ammonia supplied (α) into the furnace chamber, equation 8 allows us to calculate the degree of dissociation (β) in the exhaust gas.

$1 - \beta/100 = (1 - \alpha/100) \div [(1 - \alpha/100 + 2(\alpha/100)]$
(Eq8)

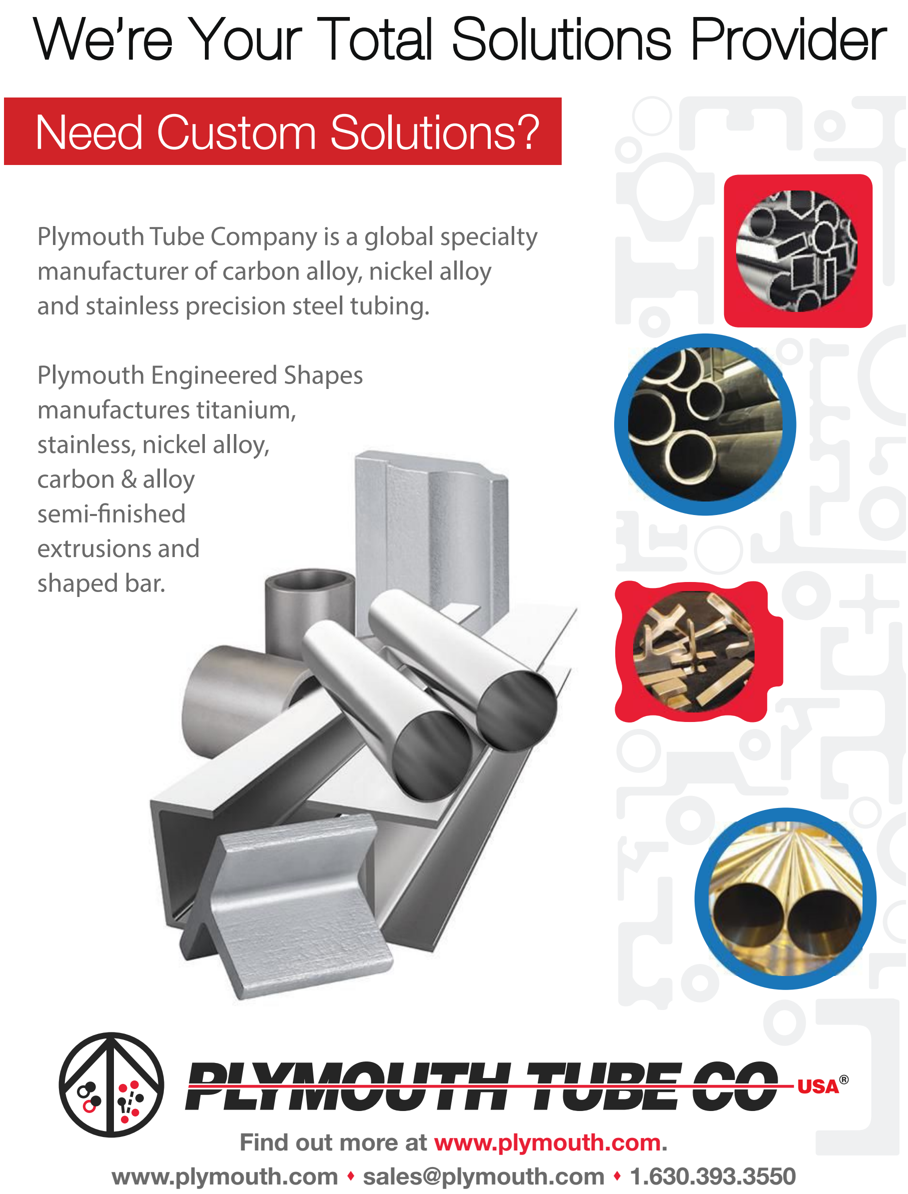
Instruments for in-situ measurement of the nitriding potential via the hydrogen content (and other methods) are commercially available and under development. These types of continuous-measurement devices are especially important for the short cycles – up to 20 hours.

Alternately, a manual method for the control of the nitriding atmosphere involves the use of a dissociation pipette or burette (Fig. 21).

Ammonia is completely soluble in water. When water is introduced into the dissociation pipette, any ammonia present dissolves, instantly forming ammonia hydroxide (NH_4OH). Water continues to enter until it occupies a volume equivalent to that previously occupied by the ammonia. The remainder of the exhaust gas, being insoluble in water, collects at the top of the pipette. The height of the water level is read directly from the scale of graduations, and this reading indicates the percentage of non-water-soluble hydrogen-nitrogen gas in the sample.

This reading, although not completely accurate, is the degree of dissociation. It should be noted that the dissociation of ammonia involves a twofold increase in volume as shown in equation 3.

Note: See Part 3 of this article for all references.



Principles of Gas Nitriding (Part 3)

This month, we focus on the materials used. Previous print articles have addressed the basics of the process (Part One) and the details of the process chemistry (Part Two).

Nitridable Steels

A variety of steels can be nitrided (Table 2). Steels with alloying elements such as aluminum, chromium, vanadium and molybdenum are more easily nitrided because they form nitrides that are stable at nitriding temperature. In general, the higher the percentage of alloying elements, the lower the nitriding temperature required and the higher the hardness achieved.

Effects of Alloying Elements

Individual alloying elements (Fig. 22) have different responses to nitriding. These include:

- Aluminum – Strongest nitride former (optimum at approximately 1.5% Al)
- Chromium – Low-alloy chromium-containing steels provide a nitrided case with considerably more ductility (than aluminum-containing steels) but with lower hardness. At high chromium percentages, the effect of chromium is approximately that of aluminum.
- Molybdenum – Forms stable nitrides; reduces the risk of embrittlement at nitriding temperatures
- Vanadium – Forms stable, hard nitrides
- Nickel, copper, silicon and manganese – These elements have little, if any, effect on nitriding.
- Lead – The addition of lead has a slightly negative effect, reducing case depth and hardness.

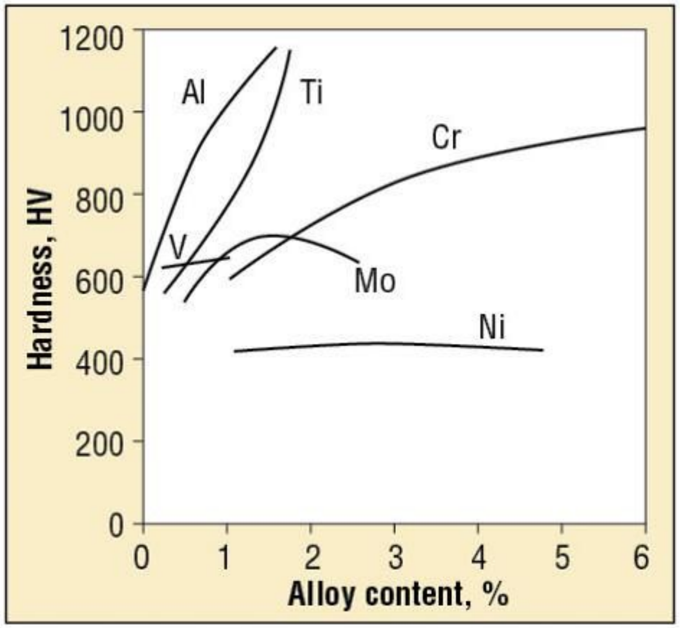


Fig. 22. Effect of individual alloying elements on nitriding

Table 2. Nitridable steels (U.S. & European)

Material	Alloy Type
Cast iron	GG25 GGG60
Unalloyed steels	1000 series (plain-carbon) CK15 C 45 St52
Alloyed steels (with aluminum)	Nitralloy G Nitralloy 135M Nitralloy N Nitralloy EZ 34CrAlNi7 34CrAlMo5
Alloyed steels (without aluminum and containing chromium)	4100 series (Cr-Mo) 4300 series (Ni-Cr-Mo) 5100 series (Cr) 6100 series (Cr-V) 8600 series (Ni-Cr-Mo) 8700 series (Ni-Cr-Mo) 9300 series (Ni-Cr-Mo) 9800 series (Ni-Cr-Mo) 16MnCr5 14NiCr14 31CrMoV9 31CrAlMo5 39CrMoV13 9
Alloyed cold-work steels	X165CrMoV12 X155CrVMo2-1
Hot-working die steels (5% Cr)	H11 H12 H13 X40CrMoV5 1 X30WCrV9 3
Stainless steels	Ferritic and martensitic (400 series) Austenitic (300 series) Precipitation hardening (e.g., 17-4, 17-7, A286)
Tool steels	Molybdenum (M) series (e.g., M2, M50) S6-5-2 S12-1-4-5
Other (European) steels	X2NiCoMo18 85 X2NiCoMo18 24 X10Cr13 X35CrMo17 X12CrNi18 8 X12CrNiNb18 9

Table 3. Selected properties of nitrided steels

Type of Steel			Processing Temperature	Case Depth	Surface Hardness	Tensile Strength
SAE/AISI	DIN	Material No.	°F (°C)	inch (mm)	HV1	KSI (N/mm²)
Cast Iron						
40B	GG25	-	980-1060 (530-570)	0.004 (0.1)	350-500	≈35 (≈250)
80-55-06	GGG60	-	980-1060 (530-570)	0.004-0.012 (0.1-0.3)	450-650	≈85 (≈600)
-	GTS55	-	980-1060 (530-570)	0.004 (0.1)	250-400	≈80 (≈550)
Structural Steels						
1010	C10	1.0301	1020-1080 (550-580)	0.012-0.032 (0.3-0.8)	200-350	50-75 (350-550)
1019	St 52 – 3	1.0570	1020-1080 (550-580)	0.012-0.032 (0.3-0.8)	200-450	80-100 (550-700)
1020	St 37-3	1.0116	1020-1080 (550-580)	0.012-0.032 (0.3-0.8)	200-350	55-65 (370-450)
1045	C45	1.0503	1020-1080 (550-580)	0.012-0.032 (0.3-0.8)	300-500	90-120 (670-820)
Free-Machining Steels						
1212	9S20K	1.0711	1020-1080 (550-580)	0.012-0.032 (0.3-0.8)	200-400	50-80 (250-550)
12L13	9SMnPb28	1.0718	1020-1080 (550-580)	0.012-0.032 (0.3-0.8)	200-400	50-80 (400-550)
Powder Metal						
-	SINT D30	-	1080 (580)	0.004 (0.1)	260-350	≥50 (≥350)
Cementation Steels						
4320	15CrNi6	1.5919	970-1020 (520-550)	0.012-0.024 (0.3-0.6)	500-650	80-100 (550-700)
5115	16MnCr5	1.5919	970-1020 (520-550)	0.012-0.032 (0.3-0.8)	500-700	80-100 (550-700)
Spring Steels						
-	67SiCr5	1.7103	≤790 (≤420)	≤0.004 (≤0.1)	700-800	218-233 (1500-1600)
Heat-Treatable Steels						
4140	42CrMo4	1.7225	930-1020 (500-550)	0.012-0.020 (0.3-0.5)	550-650	120-160 (800-1100)
4340	30CrNiMo8	1.6580	910-1000 (490-540)	0.012-0.020 (0.3-0.5)	600-700	120-160 (800-1100)
-	30CrMoV9	1.7707	910-1000 (490-540)	0.008-0.020 (0.2-0.5)	750-850	120-160 (800-1100)
Nitriding Steels						
900-1200	31CrMo12	1.8515	910-1000 (490-540)	0.008-0.020 (0.2-0.5)	750-900	130-175 (900-1200)
A355	34CrAlNi7	1.8550	970-1020 (520-550)	0.008-0.020 (0.2-0.5)	900-1000	125-160 (850-1100)
Heat-Resisting Steels						
~514	14CrMoV69	1.7735	910-1000 (490-540)	0.016-0.032 (0.4-0.8)	750-900	125-160 (900-1000)
L16	56NiCrMoV7	1.2714	840-1020 (450-550)	0.008-0.020 (0.2-0.5)	530-700	160-185 (1100-1300)
Hot-Working Steels						
H10	X32CrMoV33	1.2365	890-990 (480-530)	0.004-0.012 (0.1-0.3)	900-1100	200-235 (1400-1600)
H13	X40CrMoV51	1.2344	890-990 (480-530)	0.004-0.012 (0.1-0.3)	900-1100	200-235 (1400-1600)
Cold-Working Steels						
A2	X100CrMoV5 1	1.2363	890-950 (480-510)	0.004-0.008 (0.1-0.2)	800-1000	260-320 (1800-2200)
D2	X155CrVMo12 2	1.2379	890-950 (480-510)	≈0.004 (≈0.1)	900-1300	≥350 (≥2400)
High-Speed Steels						
M2	S6-5-2	1.3343	890-950 (480-510)	0.0008-0.04 (0.02-0.1)	1000-1250	≥350 (≥2400)
T1	S18-0-1	1.3355	890-950 (480-510)	0.0008-0.04 (0.02-0.1)	1000-1250	≥350 (≥2400)
Maraging Steels						
-	X2NiCoMo18 85	1.6359	860 (460)	0.02-0.04 (0.05-0.1)	850-950	≥305 (≥2100)
Stainless Steels						
304	X5CrNi189	1.4301	1020-1080 (550-580)	0.002-0.008 (0.05-0.2)	900-1200	75-110 (500-750)
420	X20Cr13	1.4021	1000-1060 (540-570)	0.004-0.008 (0.1-0.2)	850-1050	95-140 (650-950)
434	X35CrMo17	1.4122	1020-1080 (550-580)	0.004-0.008 (0.1-0.2)	950-1100	120-14 (800-950)
Valve Steels						
HN V3	X45CrSi93	1.4718	990-1040 (530-560)	0.0008-0.004 (0.02-0.10)	600-1000	130-160 (900-1100)
-	X55CrMnN208	1.4875	1020-1080 (550-580)	0.0008-0.004 (0.02-0.10)	700-1000	130-165 (900-1150)

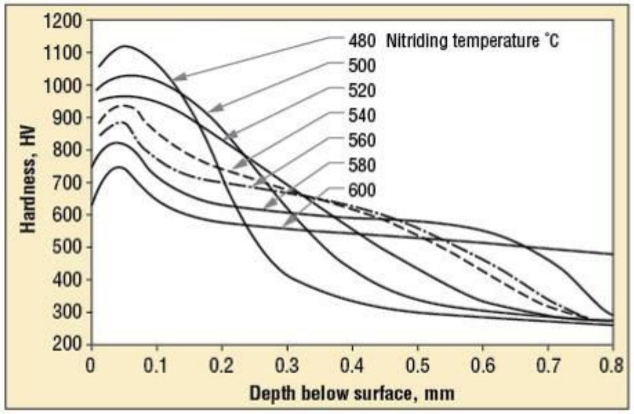


Fig. 23. Influence of nitriding temperature on surface hardness and depth of nitriding (Nitralloy 135M, 60 hour cycle)[4]

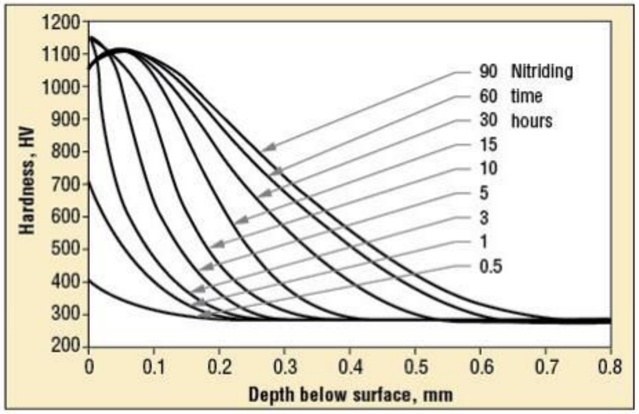


Fig. 24. Influence of nitriding time on surface hardness (Nitralloy 135M nitrided at 950°F (510°C)[4]

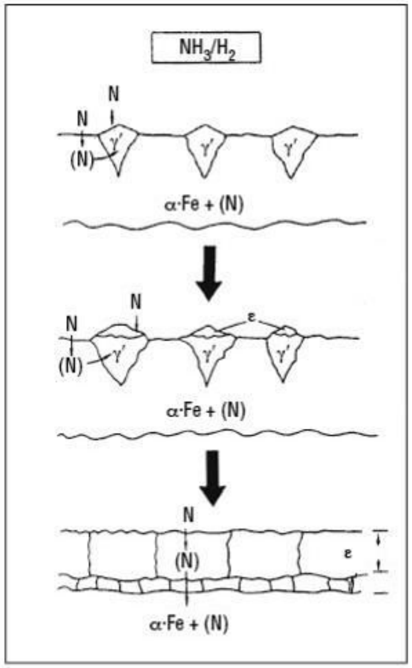


Fig. 26. Initiation of nitriding[9]

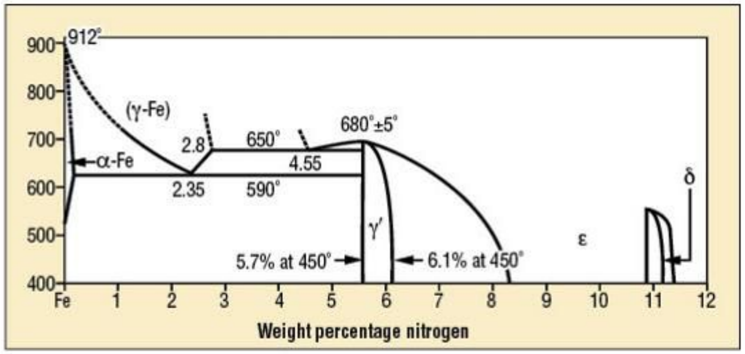


Fig. 25. Iron-nitrogen equilibrium diagram[10]

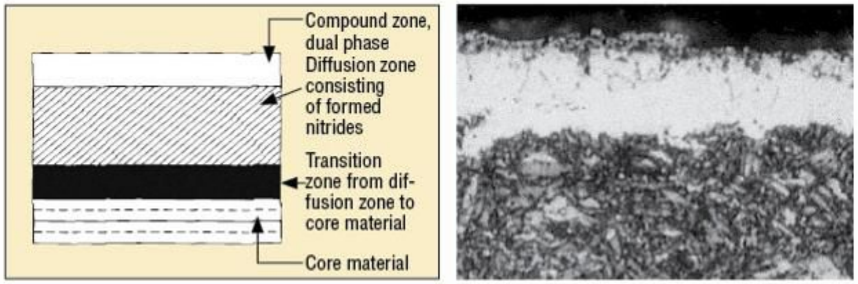


Fig. 27. Composition of nitrided layer[9]
(a) Layer representation; (b) Actual layers (compound and diffusion zones)

Table 4. Properties of some typical metal nitrides

Nitride	Properties and Applications
Aluminum nitride (AlN)	Good thermal shock resistance and a low coefficient of expansion. Excellent refractory properties. A very effective hardening agent in nitrided steel.
Alpha boron nitride (α-BN)	Excellent refractory properties with good electrical resistance.
Beta boron nitride (β-BN)	Very hard and often used as a diamond substitute. Useful in heat-resistance alloys and excellent tribological properties.
Chromium nitride	Very good hardness and wear resistance. Particularly effective as a hardening agent in nitrided steels.
Iron nitride	Very good hardness and wear resistance. Particularly effective as a hardening agent in nitrided steels.
Titanium nitride (TiN, Ti_2N)	Good high-temperature properties with thermal-shock resistance. Good abrasive and tribological properties, especially corrosion resistance.
Silicon nitride (Si_3N_4)	Very good high-temperature properties and oxidation resistance.

Properties of Nitrided Steels

Mechanical properties can be enhanced by gas nitriding.

Factors Affecting Case Depth

Users of the nitriding process (Table 3) are interested in the variables that determine the depth and properties (particularly the hardness) of the case produced. The key factors affecting the case depth are: the nature of the nitriding medium; nitriding time and temperature; the amount and nature of nitride-forming elements (Table 4) in solid solution in the steel; and the type, amount and nature of other elements in the steel (C, Ni, Si). In particular:

- The hardness of the case decreases with temperature. Lower temperatures decrease case depth for a given treatment time.
- The amount and nature of the nitride-forming elements in the steel affect the case depth to the extent that the penetration of nitrogen is inversely proportional to the amount of nitrogen that is precipitated (as the alloy nitrides) for a given cycle. The lower the alloy content, the deeper the case for a given time cycle.
- The major function of carbon is to combine with other alloying elements to form carbides, thus removing them from possible reaction with nitrogen.
- It is also known that nickel and silicon together reduce case depth slightly.

A change in nitriding temperature (Fig. 23) from 900° to 1110°F (480° to 600°C) influences the case depth and relative surface hardness. Similarly, a change in nitriding time at constant temperature can result in a variation in surface hardness (Fig. 24).

The Iron-Nitrogen phase diagram (Fig. 25) provides a "road map" to help determine the type of structures that will be produced in the nitriding process. This figure tells us what happens when nitrogen diffuses into the surface of pure iron.

At nitriding temperatures above 840°F (450°C), nitrogen will dissolve (interstitially) in ferrite (alpha-iron) but only up to a concentration of 0.1% at 1095°F (590°C). When nitrogen content exceeds this value, Fe_4N or gamma prime (γ') forms up to a nitrogen content of 5.7-6.1%. This nitride will precipitate at the grain boundaries and preferentially along certain crystallographic planes. As nitriding continues, these nitrides increase in size (as well as quantity) until the entire microstructure has been transformed into a layer of γ'. This is the so-called "compound or white layer."

As the concentration of nitrogen continues to increase, the nitrogen content in the layer also increases. When the nitrogen concentration exceeds 6.1%, γ' nitride starts to change to Fe_3N and Fe_2N, both referred to as epsilon (ε) nitride. This transformation starts at the surface (where the nitrogen concentration is greatest), and the γ' layer gradually transforms into epsilon nitride. Meanwhile, the γ' layer is driven deeper (Fig. 26).

Despite the high iron content of the epsilon layer (7.5-9%), the white (compound) layer is nonmetallic in nature. Simultaneously with the increase in thickness of the white layer, the nitrogen diffuses deeper into the steel and new nitrides are precipitated. The zone below the compound layer is called the "diffusion zone" (Fig. 27).

Since most steels also contain carbon, carbonitrides are formed as well. The nitrides and carbonitrides, in combination with the distortion of the lattice from interstitial atom addition, are quite hard, typically exhibiting ≥ 67 HRC (900-1100 HV) equivalent.

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Principles of Gas Nitriding (Part 4)

Nitrogen is a case-hardening process in which nitrogen is introduced into the surface of a ferrous alloy such as steel by holding the metal at a temperature below that at which the crystal structure begins to transform to austenite on heating as defined by the Iron-Carbon Phase Diagram.

Techniques for Inspection and Quality Control

What is the White Layer?

The "white layer" or compound zone is a very hard, brittle layer that does not diffuse into the steel but remains on the immediate surface. The process variables that control the depth (and make up) of the white layer are time, temperature and gas composition. It typically comprises two intermixed phases, gamma prime (γ') and epsilon (ε).

Thickness of the White Layer

The amount of carbon has a small effect on thickness, but it has a pronounced effect on the composition of the white layer (that is, the percentages of gamma prime and epsilon in the layer).

AMS (Aerospace Material Specification) 2759/10 identifies three classes of processes:

- Class 0: No white layer
- Class 1: White layer permitted, 0.00005 inch (maximum) or 0.0013 mm
- Class 2: White layer permitted, 0.001 inch (maximum) or 0.0254 mm

Measuring the White Layer

Hardness (Rockwell superficial) and microhardness (Knoop or Vickers) tests are commonly used to measure the case depth. Hardness of the white layer is done using microhardness techniques only (metallographic technique and proper selection of indentation load are critical to accurate readings).

The effective case depth in gas nitriding is defined as that point on the curve where the hardness is equal to the core hardness plus 4 HRC points (50 HV). For example, when the core hardness equals 30 HRC (300 HV), the effective case depth is defined as that point where the hardness is 34 HRC (350 HV).

Metallographic Evaluation

Case-depth determination (surface to a point of contrast between the case and core) can be done

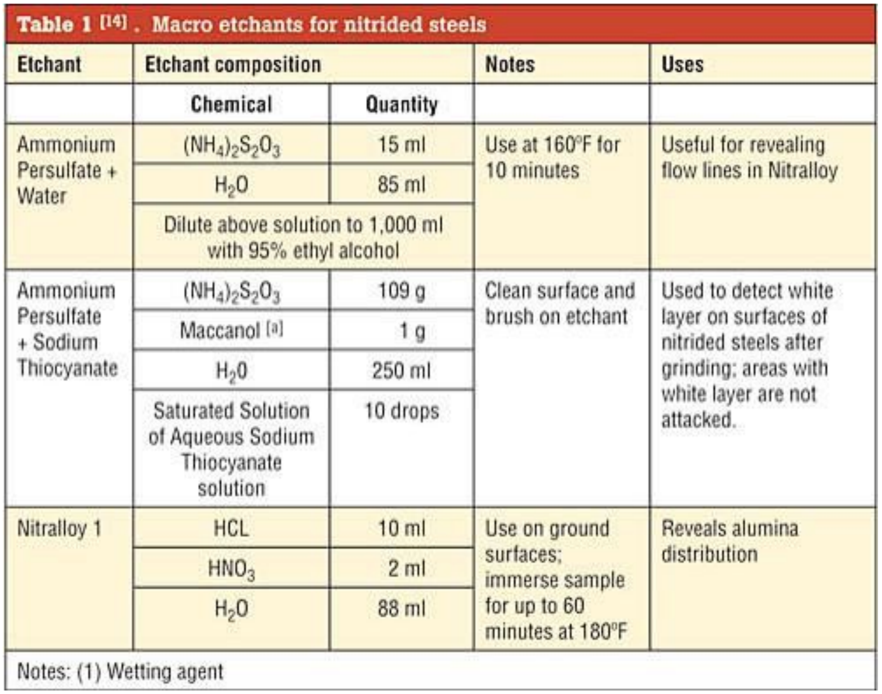

Table 1 [14] . Macro etchants for nitrided steels

Etchant	Etchant composition		Notes	Uses
	Chemical	**Quantity**		
Ammonium Persulfate + Water	$(NH_4)_2S_2O_3$	15 ml	Use at 160°F for 10 minutes	Useful for revealing flow lines in Nitralloy
	H_2O	85 ml		
	Dilute above solution to 1,000 ml with 95% ethyl alcohol			
Ammonium Persulfate + Sodium Thiocyanate	$(NH_4)_2S_2O_3$	109 g	Clean surface and brush on etchant	Used to detect white layer on surfaces of nitrided steels after grinding; areas with white layer are not attacked.
	Maccanol [a]	1 g		
	H_2O	250 ml		
	Saturated Solution of Aqueous Sodium Thiocyanate solution	10 drops		
Nitralloy 1	HCL	10 ml	Use on ground surfaces; immerse sample for up to 60 minutes at 180°F	Reveals alumina distribution
	HNO_3	2 ml		
	H_2O	88 ml		

Notes: (1) Wetting agent

by metallographic techniques. Suitable etchants for macroscopic (Table 1) and microscopic (Table 2) examination are shown.

Typical Results

White layer (iron nitride) can be 0.0002-0.0020 inch (0.005-0.050 mm) depending on length of cycle and whether single- or dual-stage nitriding is performed. Thickness is measured by metallographic methods: 5% nital or 10% ammonium persulfate. (Note: Etchants darken the case but not white layer.)

- The two-stage (Floe) process produces a shallower, softer and more ductile white layer than does the single-stage process. The white layer in the Floe process can be held to a maximum 0.0005-0.00075 inch (0.013-0.02 mm), which still may be excessive.

Elimination of White Layer

Engineering drawing specifications may call for the complete removal of the white layer, i.e. "no white layer" (0.0000 inch white layer depth). The ways to achieve this include:

1. The use of several patented processes:
    - U.S. Patent 2,960,421 achieves the removal of the iron-nitride white layer by a diffusion process. Parts are copper plated all over and then heated to and held at 975°F (525°C) for periods up to 40 hours depending on the thickness of the white layer to be removed.
    - U.S. Patent 3,069,296 achieves the removal of the iron-nitride white layer by use of a simple alkaline solution that decomposes the iron nitride, making it friable and removed by post nitriding blast cleaning. 200-mesh aluminum-oxide grit is normally recommended. Depending on the surface-finish requirements, either liquid-

abrasive or peening with glass bead may be substituted for grit blasting. The procedure does not harm the surface finish and has the added advantage of removing copper plate (during immersion in the alkaline solution) from parts plated for selected nitriding. Tests indicate no decrease in hardness, fatigue strength or impact strength, and etching or pitting of the surface does not occur if done properly.

2. The use of chemical techniques (Table 2):
   - Citric Acid – Parts are pre-cleaned then immersed in a citric-acid solution heated to 154-170ºF (68-77ºC) for several minutes. After rinsing, the parts are immersed in a neutralizing solution then rinsed again. Glass-bead blasting (400 mesh, 40-80 psig) is then performed, followed again by a cleaning operation. A stress relief operation at 335-365ºF (168-185ºC) for several hours (and within four hours of the acid immersion) completes the process.
   - Sulfuric Acid – Parts are pre-cleaned then immersed in a sulfuric acid solution heated to 165-195ºF (75-90ºC) for several seconds. After rinsing, the parts are immersed in a neutralizing solution, then rinsed again. Alumina oxide blasting (180 mesh, 40-60 psig) is then performed followed again by a cleaning operation. A stress relief operation at 335-365ºF (168-185ºC) for several hours (and within four hours of the acid immersion) completes the process.

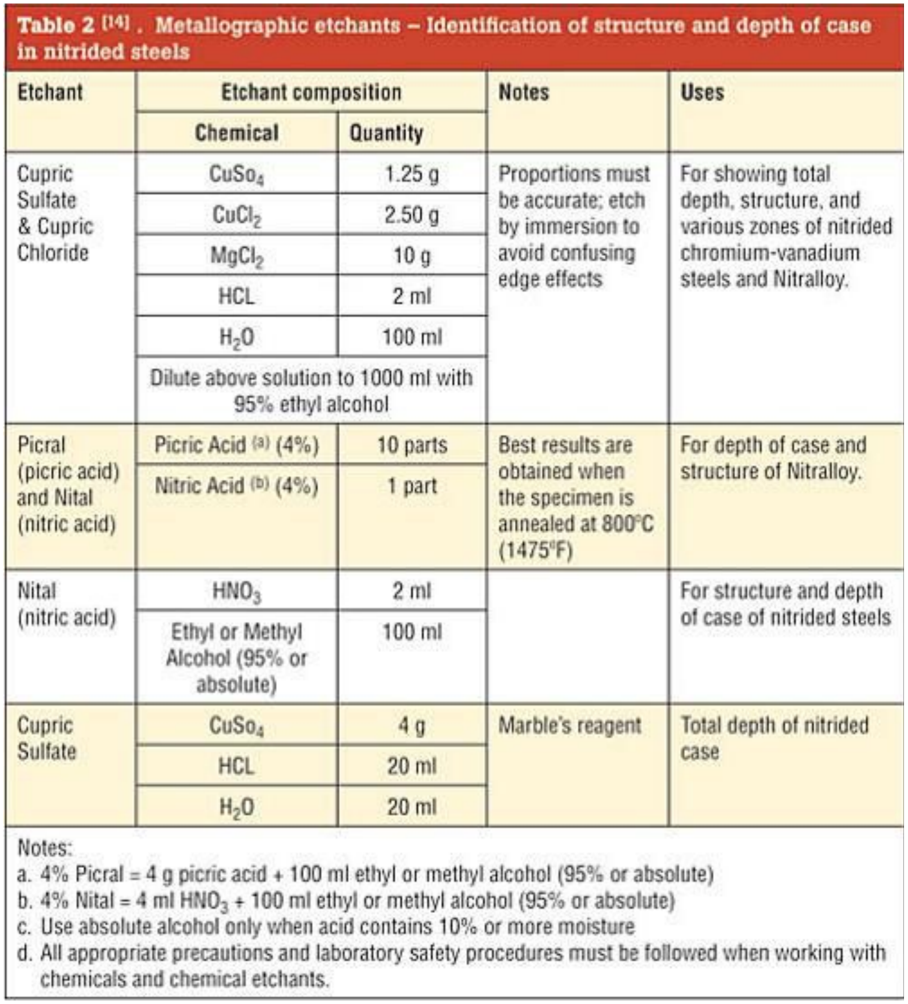

Table 2 [14] **. Metallographic etchants – Identification of structure and depth of case in nitrided steels**

Etchant	Etchant composition		Notes	Uses
	Chemical	Quantity		
Cupric Sulfate & Cupric Chloride	$CuSo_4$	1.25 g	Proportions must be accurate; etch by immersion to avoid confusing edge effects	For showing total depth, structure, and various zones of nitrided chromium-vanadium steels and Nitralloy.
	$CuCl_2$	2.50 g		
	$MgCl_2$	10 g		
	HCL	2 ml		
	H_2O	100 ml		
	Dilute above solution to 1000 ml with 95% ethyl alcohol			
Picral (picric acid) and Nital (nitric acid)	Picric Acid [a] (4%)	10 parts	Best results are obtained when the specimen is annealed at 800°C (1475°F)	For depth of case and structure of Nitralloy.
	Nitric Acid [b] (4%)	1 part		
Nital (nitric acid)	HNO_3	2 ml		For structure and depth of case of nitrided steels
	Ethyl or Methyl Alcohol (95% or absolute)	100 ml		
Cupric Sulfate	$CuSo_4$	4 g	Marble's reagent	Total depth of nitrided case
	HCL	20 ml		
	H_2O	20 ml		

Notes:
a. 4% Picral = 4 g picric acid + 100 ml ethyl or methyl alcohol (95% or absolute)
b. 4% Nital = 4 ml HNO_3 + 100 ml ethyl or methyl alcohol (95% or absolute)
c. Use absolute alcohol only when acid contains 10% or more moisture
d. All appropriate precautions and laboratory safety procedures must be followed when working with chemicals and chemical etchants.

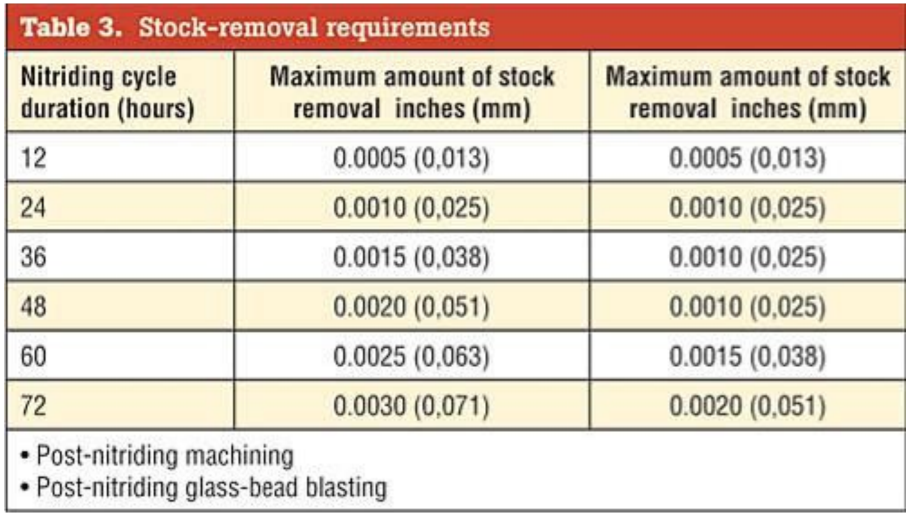

Table 3. Stock-removal requirements

Nitriding cycle duration (hours)	Maximum amount of stock removal inches (mm)	Maximum amount of stock removal inches (mm)
12	0.0005 (0.013)	0.0005 (0.013)
24	0.0010 (0.025)	0.0010 (0.025)
36	0.0015 (0.038)	0.0010 (0.025)
48	0.0020 (0.051)	0.0010 (0.025)
60	0.0025 (0.063)	0.0015 (0.038)
72	0.0030 (0.071)	0.0020 (0.051)

- Post-nitriding machining
- Post-nitriding glass-bead blasting

3. The use of ion nitriding techniques: post-nitriding machining and pos-nitriding glass-bead blasting

Re-Nitriding

Re-nitriding is not recommended. A nitrided part on which close dimensional tolerances are required must be correctly nitrided the first time. There is no satisfactory way of "de-nitriding" and then re-nitriding (only a brittle case is produced). The nitrided surface could be removed by grinding or sandblasting and the piece then re-nitrided if the resultant change in dimensions could be tolerated (it usually cannot).

Troubleshooting

The most common nitriding problems are detailed in Table 4.

Tips for Nitriding

A wide variety of steels, some stainless steels and certain tool steels can be nitrided using gas, ion (plasma) or salt bath methods. Three of the most common nitrided steels are:

- SAE 4140. This low-alloy steel is commonly used for nitriding applications. The combination of carbon and alloying elements allows core hardness in the range of 28-32 HRC developed by quenching and tempering at temperatures exceeding approximately 50°F (28°C) above the nitriding process temperature.
- SAE 4340. A higher-alloy steel used when a higher core hardness is required, up to 39 HRC, or heavier section sizes require a higher hardenability steel.
- Nitralloy. This family of steels was specifically designed for nitriding. Typical quenched-and-tempered core hardness is 25-35 HRC. The advantage of the Nitralloy steels is their excellent response to nitriding and the resulting (very) high surface hardness, typically in the 62-65 HRC equivalent range.

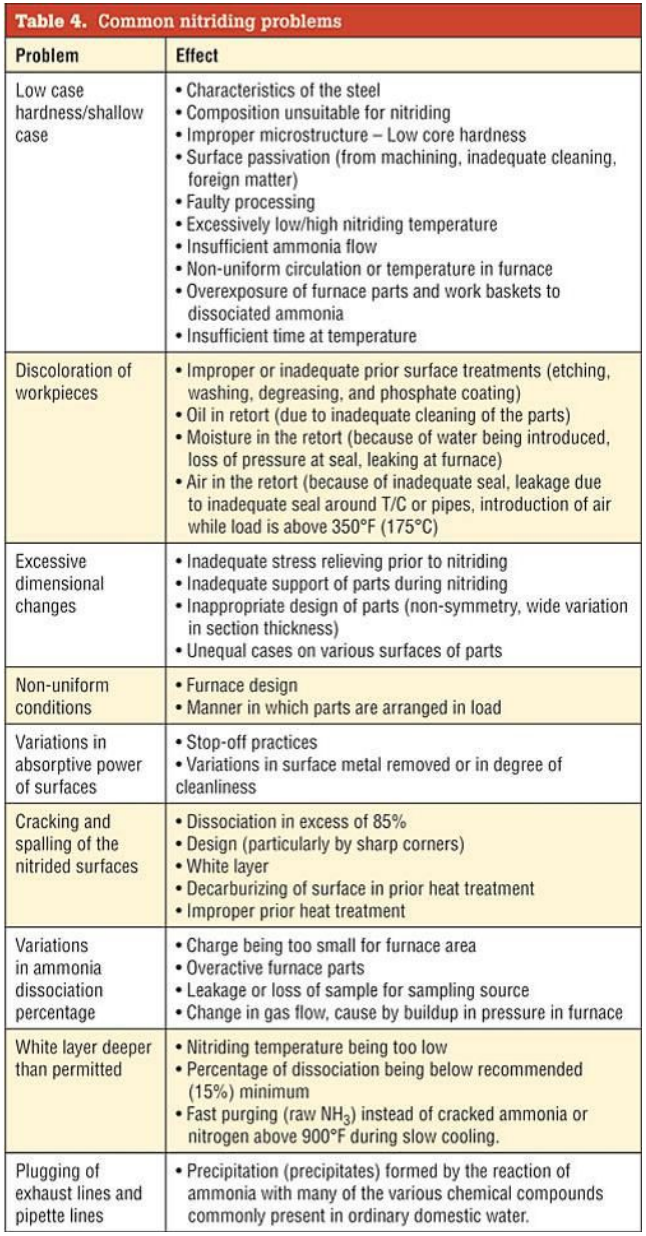

Table 4. Common nitriding problems

Problem	Effect
Low case hardness/shallow case	• Characteristics of the steel • Composition unsuitable for nitriding • Improper microstructure – Low core hardness • Surface passivation (from machining, inadequate cleaning, foreign matter) • Faulty processing • Excessively low/high nitriding temperature • Insufficient ammonia flow • Non-uniform circulation or temperature in furnace • Overexposure of furnace parts and work baskets to dissociated ammonia • Insufficient time at temperature
Discoloration of workpieces	• Improper or inadequate prior surface treatments (etching, washing, degreasing, and phosphate coating) • Oil in retort (due to inadequate cleaning of the parts) • Moisture in the retort (because of water being introduced, loss of pressure at seal, leaking at furnace) • Air in the retort (because of inadequate seal, leakage due to inadequate seal around T/C or pipes, introduction of air while load is above 350°F (175°C)
Excessive dimensional changes	• Inadequate stress relieving prior to nitriding • Inadequate support of parts during nitriding • Inappropriate design of parts (non-symmetry, wide variation in section thickness) • Unequal cases on various surfaces of parts
Non-uniform conditions	• Furnace design • Manner in which parts are arranged in load
Variations in absorptive power of surfaces	• Stop-off practices • Variations in surface metal removed or in degree of cleanliness
Cracking and spalling of the nitrided surfaces	• Dissociation in excess of 85% • Design (particularly by sharp corners) • White layer • Decarburizing of surface in prior heat treatment • Improper prior heat treatment
Variations in ammonia dissociation percentage	• Charge being too small for furnace area • Overactive furnace parts • Leakage or loss of sample for sampling source • Change in gas flow, cause by buildup in pressure in furnace
White layer deeper than permitted	• Nitriding temperature being too low • Percentage of dissociation being below recommended (15%) minimum • Fast purging (raw NH_3) instead of cracked ammonia or nitrogen above 900°F during slow cooling.
Plugging of exhaust lines and pipette lines	• Precipitation (precipitates) formed by the reaction of ammonia with many of the various chemical compounds commonly present in ordinary domestic water.

Regardless of the steel used for nitriding, two heat-treat methods are commonly used:

- Method 1. For minimal distortion: Quench-and-temper stock to specified core hardness – rough machine – stress relieve – finish machine – nitride – lap or lightly grind as necessary.
- Method 2. For maximum machinability: Rough machine – quench and temper to specified core hardness – finish machine – nitride – lap or lightly grind as necessary.

Typical nitrided case depths for steel are 0.010-0.020 inches (0.25-0.50 mm). Shallower or deeper case depths are possible. Significantly longer cycles are required for case depths about 0.020 inch due to the slow diffusion rate of nitrogen into steel. Stainless and tool steel case depths are typically limited to 0.001-0.003 inch (0.025-0.075 mm).

Case depth is generally specified as total case determined by etching a mounted sample, or it can be defined as the depth at which a certain hardness is obtained. The case depth hardness should be specified in terms of the actual core hardness (e.g., case depth at 110% of “core” or core hardness plus 3 HRC points), as the hardness gradient in a nitrided part depends heavily on the prior hardness.

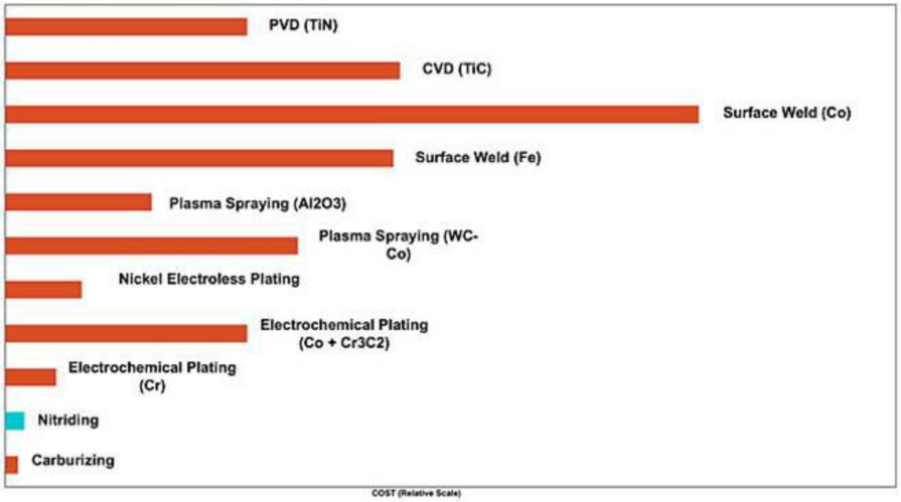

Fig. 28. Types of coatings

A typical by-product of nitriding is the white layer, a thin layer of extremely hard iron nitride. This layer may or may not be objectionable, but in most cases it must be kept thin.

Economic Considerations

Nitriding is often more expensive than other case-hardening processes (due primarily to the length of cycle), but the increased cost is often offset by the savings resulting from the small amount of distortion. Frequently, the increased cost of the nitriding process (over other methods of surface hardening) is justified by: simplification in manufacturing procedures; low-temperature process with associated low furnace maintenance; low labor costs; and cleanliness of the work. Nitriding is less expensive than most coating or other surface treatments (Fig. 28).

Nitriding should be considered for any application where wear resistance (particularly metal-to-metal wear), fatigue resistance and freedom from distortion are particularly important. Nitriding is not successful for applications involving erosion or low-stress scratching abrasion.

Typical Nitriding Applications

Basically, all iron and steel can be nitrided. Low- and non-alloyed steels can be nitrided in the annealed condition. Medium- and high-alloyed steels should be austenitized, quenched and tempered to develop optimum core properties prior to nitriding. Typical applications include:

- Cylinder barrels and liners
- Bushings
- Gears
- Piston pins
- Rotors
- Shafts
- Clutch hubs

- Shackle bolts
- Thread guides and spindles
- Cams
- Crankshafts
- Gauges
- Rubber and paper mill rolls
- Boring bars
- Camshafts
- Die-cast dies
- Clutch Plates

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Additive Manufacturing – The Next Industrial Revolution

Some say we are on the cusp of another industrial revolution, namely the decentralization of manufacturing heralded by the growth of additive-manufacturing (AM) technology. The Doctor agrees. So, what is additive manufacturing, how does it differ from other conventional manufacturing technologies and how will it affect the heat-treat community? Let's learn more.

Powder metallurgy has always been an attractive alternative to traditional manufacturing of products from wrought materials, and sintering (the bonding of adjacent powder particles together to form a cohesive metal component) is the thermal-treatment method associated with this technology. The automotive industry in particular has embraced its use. When higher densities are required, other sintering methods such as metal injection molding (MIM) are used.

What is Additive Manufacturing?

Additive manufacturing is not new, having been first introduced in the 1980s and developed for three-dimensional plastic parts with a thermoset polymer hardened by ultraviolet light. The technology was initially too slow for mass production and primarily used for rapid prototyping. Today, metals have been added to the list of materials that can be used, and the speed of the process has accelerated to the point where it is viable for high-volume manufacturing.

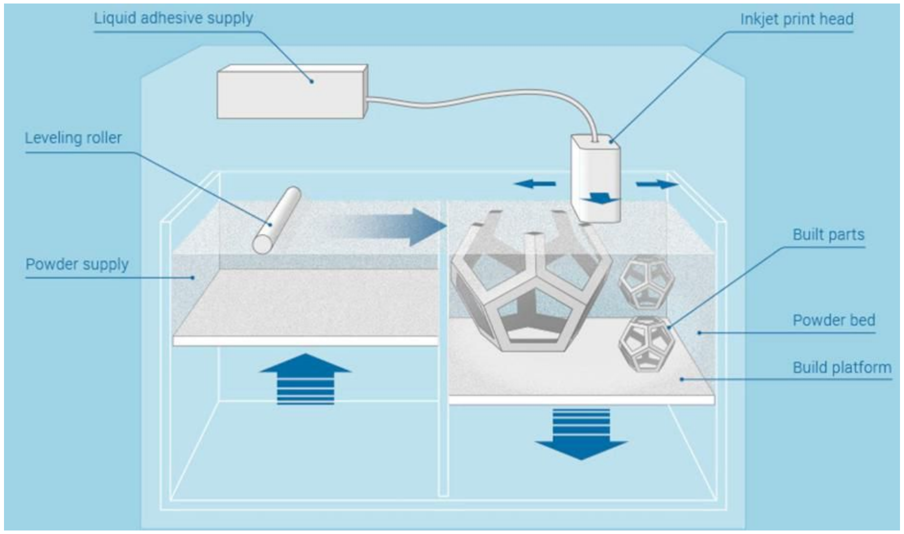

Fig. 1. Function of a binder-jetting printer[2]

AM refers to a process in which the raw material is added layer upon layer to create a component part. This is the opposite of machining, often now referred to as "subtractive manufacturing," which creates a part by removing material from a raw-material form.

One of the most promising versions of metal AM today is binder-jetting technology. Laser sintering and electron-beam methods are alternatives.[4] Binder jetting of metals is a process in which a liquid binding agent is selectively deposited onto a bed of powder-metal particles as the layers of the component part are being built up. The goal is to reduce the amount of liquid binder used since less binder allows easier access to the pores and more rapid binder removal.

A moving print head (Fig. 1) strategically blends binder into the powder while it is being deposited on the printing bed. After each pass, the bed height is lowered by the thickness of a print layer, 25-100μm, and another layer of powder and binder is added on top of the previous. As this is repeated, layers of bonded metal are successively deposited until the fully formed part is created.

After printing, sintering in a vacuum furnace is required, the same as with MIM technology. Binder jetting is used for creating parts made of Inconel, stainless steel, tungsten carbide, titanium, copper, brass and aluminum, among others. Since the printed layers can be extremely thin, the resulting part can be produced to an extremely high level of detail with very precise physical features.

Typical tolerances and specifications of metal binder-jetting technology include:[2]

- Maximal build envelope of 4,000 mm x 2,000 mm x 1,000 mm
- Minimum feature size of 0.1 mm
- Typical tolerance of ±0.13 mm
- Minimum layer thickness of 0.09 mm
- Fast build speed (in comparison to other additive technologies)

Binder jetting is the fastest metal AM method available. Maximum build speed is currently approximately 2,500 cm^3/hour, and one manufacturer is planning to introduce an 8,200 cm^3/hour machine in 2019. Car bodies and other large composite plastic parts have been printed using AM technology, and it is just a matter of time before this expands to metal printing.

AM is considered by most in the industry as a "disruptive" technology; one that will revolutionize many industrial sectors as it becomes faster and less expensive. It will also fundamentally affect how, when and where heat treatment is performed since sintering will become part of an AM manufacturing cell. As AM becomes more sophisticated and as understanding and awareness grows among manufacturers, machine shops as we know them will be fundamentally changed. AM offers clear advantages in that:

1. Small runs of unique or complex parts can be produced quickly and at low cost. Unlike MIM,

Fig. 2. A lightweight drone support structure created using AM[3]

casting or forging, no expensive molds are required. This reduces time to market, a very valuable commodity today.

2. Shrinkage is significantly less than that of MIM-produced parts, increasing accuracy and repeatability. One description of AM is that it is a MIM process without the distortion.
3. AM has the ability to pursue new innovations without extending the design cycle. This allows for many generations of design changes in the time that it would normally take to make a single change

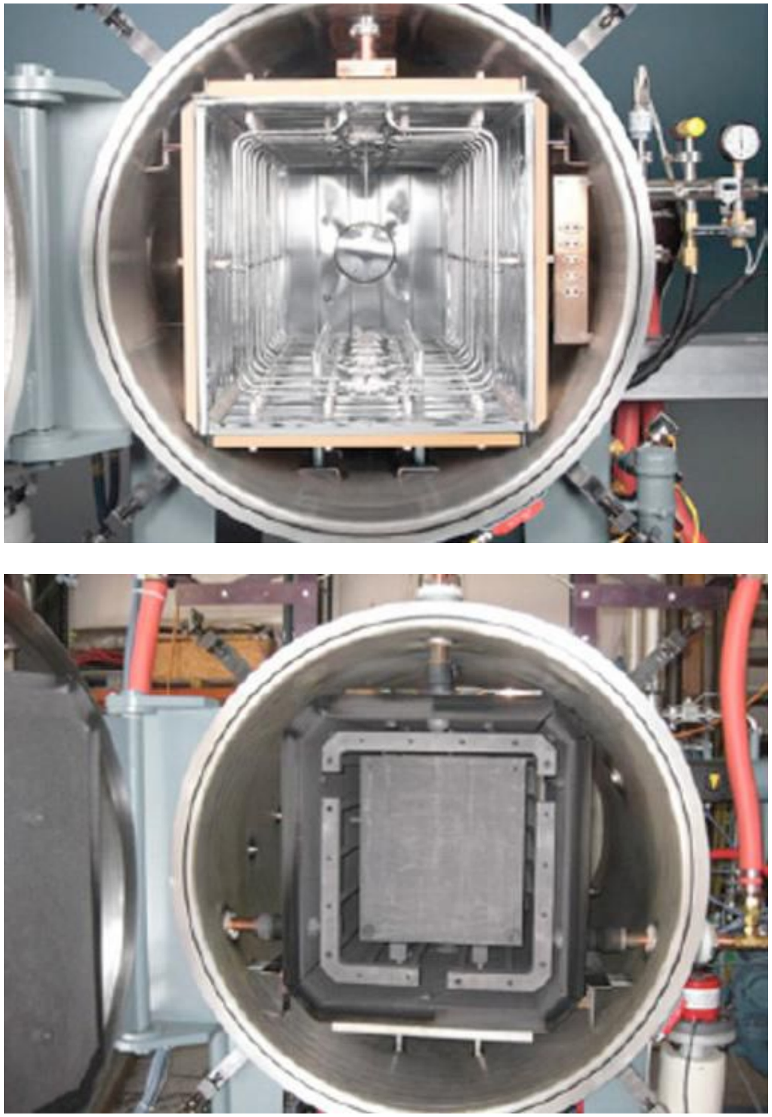

Fig. 3. Vacuum sintering furnaces for AM utilizing all-metal (top) or graphite (bottom) hot zones (courtesy of Centorr Vacuum Industries)

using conventional technologies. This might be the most revolutionary aspect of the technology.

4. Honeycomb designs are possible, reducing part weight while maintaining or even increasing strength.
5. AM offers the ability to make on-the-fly changes. If there's one thing design engineers can count on, it is customer revisions and design changes. With AM technology, the designer simply makes a change in the 3D digital model, and it is downloaded to the printer for manufacture.
6. Highly complex parts can be produced (Fig. 2) that would be literally impossible with any other technology. There are some shapes and intricate features that cannot be cast, molded or machined but can be printed. This opens up new possibilities for designers.
7. A high degree of customization is possible without added cost. AM technology allows the manufacture of one-of-a-kind designs like medical implants that are custom made to fit a specific individual.
8. AM generates no waste. Since it is an additive technology, only the material that is needed is actually used. When printing very expensive metals such as titanium, this makes a huge difference in the price of the finished product and the feasibility of the project.

AM has always been an attractive choice when production volumes are low, changes are frequent and complexity is high. As print speed increases and costs come down, AM applications will expand to include more mainstream component parts. Machine shops and in-house manufacturing departments will then be able to choose the most cost-effective technology, with sintering being performed as part of the AM manufacturing cell as opposed to a heat-treatment department or an outsource location. This will lead to new opportunities and challenges for heat treaters because more parts will require secondary debinding and sintering under vacuum.

For example, one of the primary challenges for vacuum furnaces (Fig. 3) used for sintering is dealing with the binder liberated from the material during the secondary debinding process. Dry pumps are preferred since the binder can contaminate the oil used in rotary oil-sealed pumps requiring frequent oil changes.

There must also be provisions made for removing the binder. One approach is to locate a binder trap prior to the pump, which collects the binder and requires periodic removal and cleaning. Manual or automated traps are available – the latter heats up to liquefy the binder residue, which then flows to the bottom of the trap. A valve is opened to allow residue to be collected. A third method involves the use of a condensing filter.

Summary

The additive-manufacturing revolution has begun! It will soon have an impact on all types of industries and their manufacturing strategies, representing a paradigm shift in design and engineering that will affect every process in the factory, including heat treatment.

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Heat Treatment of Cast Irons

Thinking back to our high-school days often brings a smile to our faces. As a freshman, the daunting task of picking a foreign language to study fell to The Doctor's mother, who immediately chose Latin for him. While perhaps not the most popular of choices today, it was a highly rewarding experience, especially for improving one's understanding of the English language and for making sense out of many scientific and scholarly works of the past. For example, did you know that iron (chemical symbol: Fe) derives its name and symbol from the Latin word *ferrum*? Its characteristics and heat treatment are equally interesting and much different from steel. Let's learn more.

What is iron?

Iron is an element that in its pure form is lustrous, silvery, soft and ductile. However, pure iron (<0.008% C) is a poor engineering material, generally not as strong as most plastics. Iron is, however, the fourth-most-abundant element on Earth and one of the most widely disbursed elements in the Earth's crust. In nature, it is found in various compounds with oxygen, sulfur or more complicated ores such as carbonates and silicates (Table 1).

Table 1. Common iron ores[1]

Mineralogical name	Chemical formula	Chemical composition	Class
Magnetite	Fe_3O_4	72.36% Fe, 27.64% O_2	Oxide
Hematite	Fe_2O_3	69.94% Fe, 30.06% O_2	Oxide
Ilmenite	$FeTiO_3$	36.80% Fe, 31.63% O_2, 31.57% Ti	Oxide
Limonite	$HFeO_2$, FeO(OH)	62.85% Fe, 27.01% O_2, 10.14% H_2O	Oxide
Pyrite	FeS_2	46.55% Fe, 53.45.5% S	Sulfide
Siderite	$FeCO_3$	48.20% Fe, 37.99% CO_2, 13.81% O_2	Carbonate

Iron has several unique properties that are of interest to metallurgists and engineers. First, iron requires relatively little energy to be extracted from its ore, making it the most cost-effective material choice (Fig. 1). Second, iron has asymmetric rotation of its electrons (that is, the electrons can spin in same directions). This allows iron to more easily combine with alloying elements, thus changing its properties. Finally, it is polymorphic, which means its crystal structure can "flip" or transform into different structures as a function of temperature and carbon content. Together these

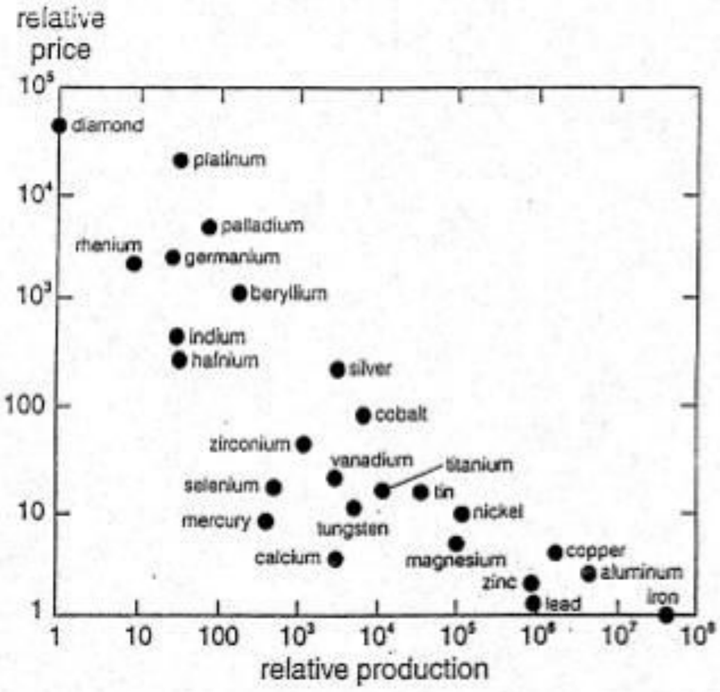

Fig. 1. Price vs. availability of common elements[2]

properties make iron one of the most attractive elements to use for the products we require in everyday life.

What is cast iron?

Cast iron (>2.11% C) is a generic term used to designate a family of metals with a wide variety of properties. All cast irons contain more than 2% carbon and an appreciable amount of silicon (usually 1-3%). The high carbon and silicon content means that they are easily melted, have good fluidity in the liquid state and have excellent pouring properties.

The basic types of cast iron are best differentiated by their microstructure, which is the form and shape in which the excess carbon separates during cooling. This determines the type of cast iron and establishes the nature of its properties – as opposed to their chemical analysis – because the various types overlap (Table 2).

Table 2. Typical composition of unalloyed cast irons[3]

Element	White iron (%)	Malleable iron (%)	Gray iron (%)	Ductile or nodular iron (%)
Carbon	1.8-3.6	2.00-2.60	2.5-4.0	3.0-4.0
Silicon	0.5-1.9	1.10-1.60	1.0-3.0	1.8-2.8
Manganese	0.25-0.80	0.20-1.00	0.25-1.0	0.10-1.00
Sulfur	0.06-0.20	0.04-0.18	0.02-0.25	0.03 max
Phosphorous	0.06-0.18	0.18 max	0.05-1.0	0.10 max

In *white* iron, the majority of carbon occurs as iron carbide. In *malleable* iron, the majority of carbon is in the form of irregularly shaped nodules of graphite. In *gray* iron, the carbon is present as graphite flakes, and in *ductile* iron, the graphite occurs in spheres. Because graphite has very little (cohesive) strength and reduces the effective

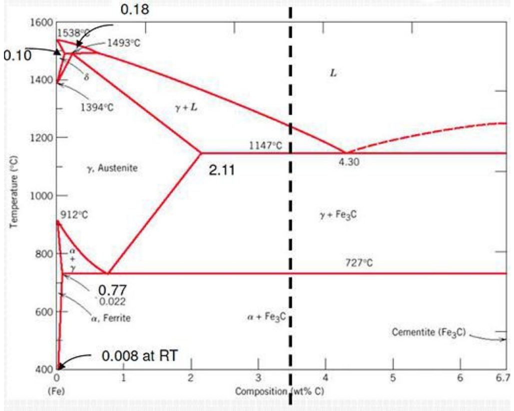

Fig. 2. Hypoeutectic cast iron[5]

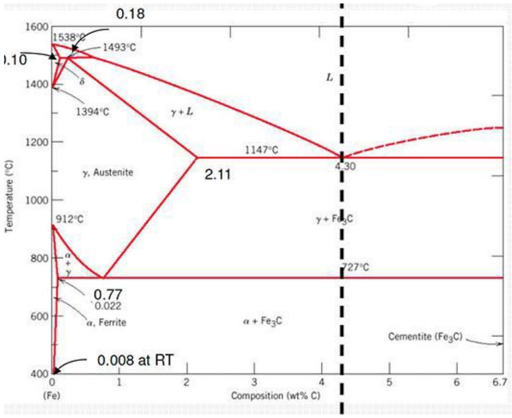

Fig. 3. Eutectic cast iron[5]

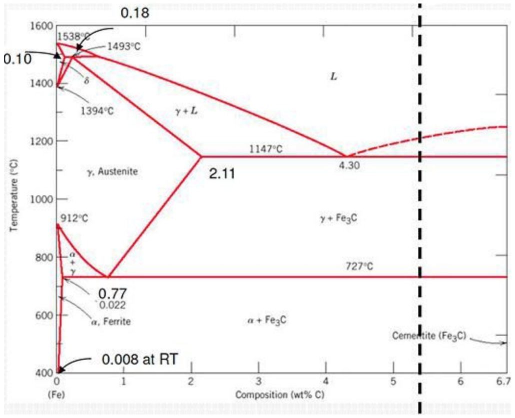

Fig. 4. Hypereutectic cast iron[5]

metallic cross section of the casting, both strength and ductility are affected.

Metallurgy of Cast Iron

The metallurgy of cast iron is much more complex than its economics. In fact, cast iron is one of the more complex metallurgical systems. It is interesting to note that iron-carbon alloys (with less than 2% carbon) are actually metastable. The true stable system is the iron-graphite phase transformation system (Fe-C). The solid-state transformations, on which cast-iron heat treatments are based, are similar to those applied to steels and can be characterized as:

- On cooling of *hypo*eutectic irons (Fig. 2), at a temperature just below 1147ºC (2097ºF), the microstructure consists of proeutectic austenite and ledeburite. On further cooling, excess carbon comes out as cementite from proeutectic and eutectic austenite. Finally, at 727ºC (1341ºF) both eutectic and proeutectic austenite decomposes into pearlite with a carbon content of approximately 0.77%.
- On cooling of eutectic cast irons (Fig. 3), ledeburite is formed. On further cooling, excess carbon comes out of solution as cementite from eutectic austenite. Finally, at 727ºC (1341ºF) eutectic austenite decomposes into pearlite.
- On cooling of *hyper*eutectic irons (Fig. 4), at a temperature just below 1147ºC (2097ºF), the microstructure consists of proeutectic austenite and ledeburite. On further cooling, excess

carbon comes out as cementite in the form of proeutectoid cementite and eutectic austenite. Finally, at 727°C (1341°F) eutectic austenite decomposes into pearlite.

If an iron alloy exceeds 2.11% carbon, the carbon does not have to nucleate from decomposition of austenite, but it can instead form directly from the melt by a eutectic reaction. Note that cementite (Fe_3C) can still nucleate at the eutectic more readily than graphite, but on sufficiently slow cooling, graphite itself is able to form and grow.

Consider the solidification of a 3% carbon cast iron. At a rapid cooling rate, dendrites of austenite form as the alloy cools below the liquidus and grow until the eutectic temperature is reached. At the eutectic point, graphite formation is suppressed, but austenite and cementite precipitate to form ledeburite – a form of eutectic that consists of spheres of austenite embedded in cementite. Ledeburite forms at the Fe-Fe_3C eutectic. On further cooling, the cementite grows as the austenite decreases in carbon content. At the eutectic, the remaining austenite transforms to pearlite. At room temperature, the iron is hard and brittle and is called white iron because the surface of a fractured piece of iron is white and (somewhat) lustrous.

Upon slow cooling of a 3% carbon cast iron, austenite forms from the melt, but eutectic freezing is now slow enough so the products of the eutectic reaction are austenite and graphite. The eutectic graphite tends to form flakes surrounded by eutectic austenite. As cooling continues, the austenite decreases in carbon content, while the remaining austenite transforms to pearlite. Because the fracture surface appears dull gray, the material is known as gray iron (or pearlitic gray iron).

Cooling at an extremely slow rate results in phase changes similar to those of a slow-cooled component, except the eutectoid cooling is sufficiently slow to permit graphite to precipitate rather than pearlite. No new graphite flakes will form, but the ones present will increase in size. The final microstructure consists of graphite flakes embedded in a ferrite matrix. The resultant material is called ferritic gray iron. It is noteworthy that cooling of actual castings is seldom slow enough to obtain this structure.

The cooling rate of a portion of a casting may sometimes vary, resulting in a structure containing patches of both white and gray iron called mottled iron.[4]

Types of Iron

Pig iron is the term that is generally applied to a metallic product that contains over 90% iron. Typically, it contains approximately 3% carbon, 1.5% silicon and lesser amounts of manganese, sulfur and phosphorus. Pig iron along with scrap metal is the base material for both cast iron and cast steel.

White Cast Iron

Chilled cast iron is produced by casting the molten metal in such a way as to produce a surface virtually free of graphitic carbon. The surface of chilled iron castings is extremely hard. The depth and hardness of the chilled portion may be controlled by adjusting the composition of the metal.

White iron is a type of chilled cast iron that is virtually free of graphitic carbon, which is achieved by selecting a specific chemical composition to inhibit graphitization for a given section size. The hardness of the casting may be controlled by selection of composition.

In both chilled and white irons, the depth of chill decreases and the hardness increases with increasing carbon content. Carbon varies from 2.5% to above 3.5%. It also is necessary to control silicon content; the chill is reduced by increasing the silicon content. The range of silicon content for white irons is governed by the section size. Silicon content varies from 0.6-1.5%. Alloying elements such as nickel, chromium and molybdenum are used to improve wear resistance.

Malleable Iron

Malleable irons are relatively soft and can be bent without breaking. They include ferritic (or standard) malleable iron and pearlitic malleable iron. Malleable iron is obtained by the heat treatment of white iron, so the hard iron-carbide structure of ledeburite is converted to a matrix of ferrite or pearlite and graphite, the latter being precipitated within the iron. This form of graphite is sometimes referred to as tempered carbon. A wide range of mechanical properties can be obtained in malleable iron with different matrix structures around the graphite.

Ferritic malleable iron has a ferrite matrix with interspersed nodules of tempered carbon and, depending on how it is produced, some combined carbon. Pearlitic malleable iron is designed to have combined carbon in the matrix, resulting in higher strength and hardness than ferritic malleable iron. The malleable irons typically contain from 2.0-2.65% C, 0.90-1.65% Si and 0.25-0.55% Mn as major constituents.

Table 3. Common grades of ductile iron[3]

Type (TS-YS -%El)	Tensile strength (ksi)	Yield strength (ksi)	Elong. (%)	Hardness (BHN)	Heat treatment	Typical microstructure
60-40-18	60	40	18	137-170	Annealed	All ferrite
65-45-12	65	45	12	149-229	-	Ferritic
80-55-06	85	55	6	179-255	-	Ferrite and pearlite
100-70-03	100	70	3	229-302	Normalized	All pearlite
120-90-02	120	90	2	250-350	Quench & temper	Tempered martensite

Gray Iron

Gray irons are alloys of iron, carbon and silicon, in which more carbon is present than can be retained in solid solution in austenite at the eutectic temperature. The carbon precipitates as graphite flakes. The gray irons typically contain from 1.7-4.5% carbon and 1-3% silicon as major constituents.

Ductile Iron

Ductile or nodular iron, also known as spheroidal graphite iron, is cast iron in which the graphite is present as tiny balls, or spherulites, instead of graphite flakes (as in gray iron) or compacted aggregates (as in malleable iron). The nodular irons typically contain from 3.2-4.1% C, 1.8-2.8% Si and up to 0.80% Mn as major constituents. Several types of matrix structures (including ferritic and pearlitic) can be developed by alloying and heat treatment. The various grades of regular, unalloyed ductile iron are designated by their tensile properties (Table 3).

Heat Treatment

The types of heat treatments for cast irons involve stress relief, annealing, normalizing and hardening (quench and temper). In each of these processes, it is important to understand the materials' response to its heat-treat process parameters (time, temperature and atmosphere).

Stress Relief

The need for stress relief is due to internal (residual) stress in the castings, which is often the result of cooling a complex or intricate shape or one with radical changes in cross-sectional area. In stress relief, the time-temperature relationship plays a significant role (Fig. 5), and higher temperatures will affect mechanical properties and often require the use of protective atmosphere to avoid oxidation.

Soak time at temperature is in the order of 1.5 hours per 25.4 mm (1 inch) of cross-sectional area for sections over 50 mm (2 inches) and 1 hour per 25.4 mm (1 inch) of cross-sectional area below this thickness. The cooling rate is also important, with furnace cooling to 260°C (500°F) adequate for most castings. However, those with intricate shapes should be cooled to 95°C (200°F).

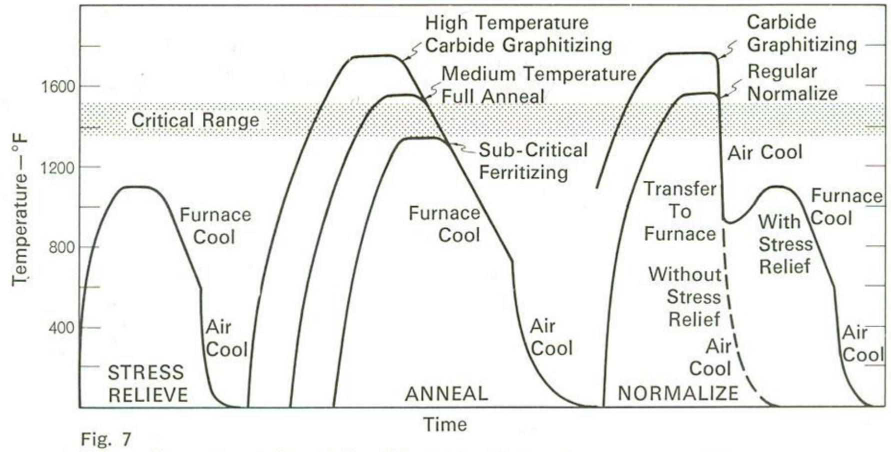

Fig. 7
Schematic representation of the different heating and cooling cycles used to stress relieve, anneal, and normalize iron castings⁴.

Fig. 6. Stress relief, annealing and normalizing cycles for cast irons[5]

Annealing

The annealing process is applied to castings primarily to improve machinability by softening the material. In the case of ductile iron, increases in ductility and impact resistance often result. Various heating and cooling cycles (Fig. 6) can be used each having a different purpose (Table 4).

Table 4. Annealing processes for gray and ductile iron castings[5]

Annealing type	Material	Purpose	Temperature[a] °C (°F)	Time	Cooling rate[c]
Ferritizing (low-temperature annealing)	Gray iron	Conversion of ferrite to pearlite in unalloyed irons for maximum machinability.	705-760 (1300-1400)	¾ hour per inch of cross-sectional area.	Furnace cool at 38°C/hour (100°F/hour) to 315°C (600°F). Cool in still air from 315°C (600°F) to room temperature.
	Ductile iron	In the absence of carbides to obtain grades 60-45-12 and 60-40-18.	720-730 (1325-1350)	1 hour per inch of cross-sectional area.	Furnace cool at 38°C/hour (100°F/hour) to 345°C (650°F). Air cool.
Full (medium temperature)	Gray iron	Conversion of ferrite to pearlite in unresponsive to low-temperature annealing; elimination of minor amounts of well-dispersed carbides in unalloyed irons.	815-900 (1500-1650)	1 hour per inch of cross-sectional area.	Furnace cool to 315°C (600°F). Cool in still air from 315°C (600°F) to room temperature.
	Ductile iron (lower Si-content iron)	In the absence of carbides to obtain grade 60-40-18.	870-900 (1600-1650)	Only long enough to equalize at control temperature	Furnace cool at 38°C/h (100°F/hour) to 345°C (650°F). Air cool.
Full (Graphitizing, high temperature)	Gray iron	Elimination of massive carbides in mottled or chilled irons and conversion of ferrite to pearlite in unalloyed irons for maximum machinability.	900-955 (1650-1750)	1-3 hours plus 1 hour per inch of cross-sectional area	Furnace cool to 315°C (600°F). Cool in still air from 315°C (600°F) to room temperature
	Ductile iron	In the presence of carbides to obtain grades 60-45-12 and 60-40-18.	900-924 (1650-1700)	2 hours minimum	Furnace cool at 95°C/hour (200°F/hour) to 705° C (1300°F). Hold 2 hours. Furnace cool at 38°C/hour (100°F/hour) to 345°C (650°F). Air cool.
Two-stage graphitization and ferritizing	Ductile iron	In the presence of carbides to obtain grades 60-45-12 and 60-40-18 where rapid cooling is practical.	900-924 (1650-1700)	1 hour per inch of cross-sectional area	Fast cool to 675°C (1250°F). Reheat to 730°C (1350°F) Hold 2 hours. Furnace cool at 38°C/hour (100°F/hour) to 345°C (650°F). Air cool.
Normalize Anneal	Gray iron	Elimination of massive carbides with the retention of pearlite for strength and hardness.	870-955 (1600-1750)	1-3 hours plus 1 hour per inch of cross-sectional area[b]	Air cool to below 480°C (900°F). May require subsequent stress relief.
Normalizing and tempering stress relief	Ductile iron		900-924 (1650-1700)	2 hours minimum	Air quench with fans. Temper at 540-675°C (1000–1250°F). Furnace cool at 38°C/hour (100°F/hour) to 345°C (650°F). Air cool.

Notes:
[a] Preferred temperature for castings is dependent on silicon, manganese and the alloy content.
[b] Time for graphitization will vary from 30-60 minutes in unalloyed irons to 2-3 hours in irons containing carbide stabilizers.
[c] Slow cooling from 540-315°C (1000-600°F) is necessary to minimize residual stress.

Normalizing

Iron castings are commonly normalized to obtain a microstructure of fine pearlite. The result is a microstructure with increased tensile strength and wear resistance. Normalized structures respond well to induction hardening. In general, the heating rate is not important, but excessive distortion and cracking must be avoided.

Normalizing temperature ranges vary from 815-870°C (1500-1600°F) for high-strength gray irons to 845-900°C (1550-1650°F) for low-strength gray irons and 870-925°C (1600-1700°F) for ductile irons. The silicon content determines the final temperature because it increases the critical temperature of the material. Cooling rates vary from still-air to fan-assisted cooling for large castings. Tempering is used if the final hardness is too high.

Hardening (Quench and Temper)

It is important to carefully select and control the process parameters when performing these operations so as to avoid problems such as distortion, warpage, overheating ("burning") or quench cracking. The lower-critical temperature for cast irons can be calculated (Eq. 1 and 2) or determined by sample analysis.

(1) Critical temperature (°C) = 730 + 28%Si – 25%Mn
(2) Critical temperature (°F) = 1350 + 50%Si – 45%Mn

In practice, castings to be hardened are austenitized at a temperature of 10-38°C (50-100°F) above the upper-critical temperature for one hour per 25 mm (1 inch). Heating should be gradual so as not to induce thermal stresses. After soaking, the parts are quenched. Typically, a severe quench is unnecessary. Oil or polymer quenching can also be used. Tempering follows and can be performed over a wide range, from 120-595°C (250-1100°F), to develop proper mechanical properties. Tempering time is typically shorter than for steels.

Cryogenic Processing

Many cast irons are cryogenically treated (-195ºC/-320ºF) to stabilize the microstructure and enhance properties (e.g., dampening and wear characteristics). A typical cycle consists of slowly reducing temperature over a period of 6-8 hours, stabilizing at temperature (typically 8-12 hours) and slowly raising the temperature back to room temperature again over a period of 6-8 hours.

Cryogenically treated gray cast-iron brake rotors (Fig. 7) in automotive applications have been shown to improve service life. In police cars, where a combination of high speeds and frequent braking translates to brake-component replacement around 12,875 km (8,000 miles), cryogenic processors report having extended the time between replacement of these components consistently up to 38,625 km (24,000 miles).

Common Heat Treatment for the Specific Cast-Iron Types

High-alloy (e.g., high chromium, high nickel-chromium) white irons can be heat treated. For example, nickel-chromium white-iron castings are given a stress-relief heat treatment because, properly made, they have an as-cast martensitic matrix structure. Tempering is performed between 205°C (400°F) and 260°C (450°F) for at least 4 hours. This tempers the martensite, relieves some of the transformation stresses, and increases the strength and impact toughness by 50-80%. Malleable cast iron can be heat treated to the same microstructures as ductile cast irons.

Fig. 7. Pearlitic structure of a cryogenically treated gray cast-iron brake rotor; 100X; 2% nital etch (courtesy of Controlled Thermal Processing Inc.)

The most common heat treatment applied to gray cast irons is stress relief because of non-uniform cooling of castings and annealing to improve machinability. Subcritical heating is used for both. Stress relief is done at temperatures between 550-650°C (1020-1200°F) without significantly lowering strength and hardness. Heating at temperatures between 700ºC (1290°F) and 760°C (1400°F) reduces the hardness for improved machinability.

Heat treatments of ductile cast iron include stress relief and annealing, and heat treatments used for steels include normalize and temper (for higher strength and wear resistance), quench and temper (for the highest strength) and austempering. Ferritizing (for the most-ductile microstructure) is done by austenitizing at 900°C (1650°F) followed by holding at 700°C (1290°F) to completely transform austenite to ferrite and graphite.

Summary

The heat treatment of cast iron deserves special consideration and is unique from steel heat treatment in many ways. The heat treater must understand these alloys in order to design recipes to ensure they uniformly achieve the required mechanical and metallurgical properties.

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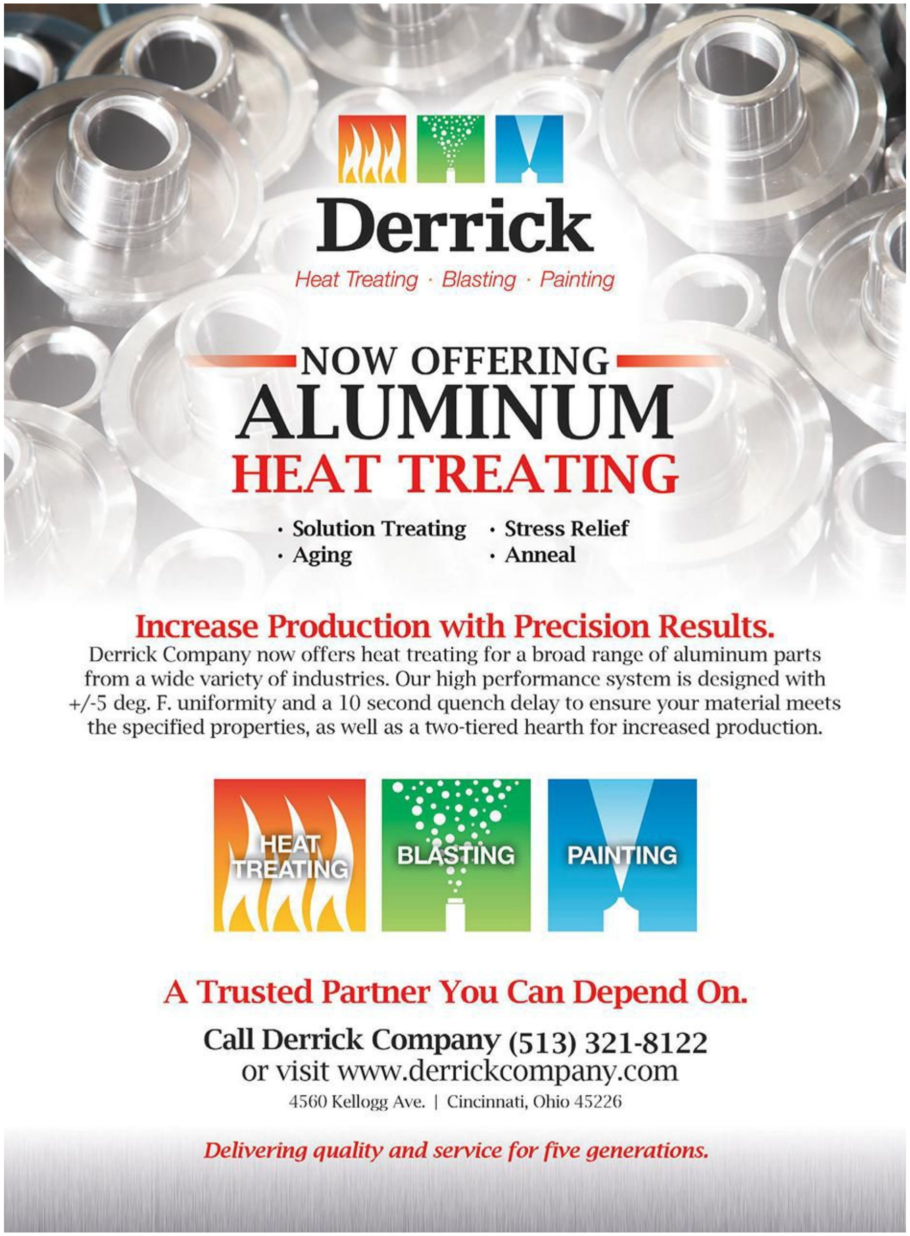

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Brazing of Nonferrous Materials

Brazing is not a heat treatment process, but a joining process that uses heat to help produce structurally sound assemblies. Because so many products we use every day rely on the assembly of two or more individual components that are then joined together, it is an important process to understand. When we think about brazing, we often focus on ferrous base materials and nonferrous filler metals such as copper brazing of steel. But there is a broad range of applications where nonferrous base materials must be brazed. Let's learn more.

Brazing Aluminum And Aluminum Alloys

The brazeability of aluminum and its alloys is generally considered difficult—rated as fair to good. Non-heat treatable alloys such as the 1xxx and 3xxx series, as well as low-magnesium alloys of the 5xxx series, are successfully brazed. Be aware that a certain amount of annealing occurs during brazing of these alloys and is dependent on the specific alloy, time/temperature cycle and the mass of the area subjected to heat.

Of the heat-treatable wrought alloys, brazing of the 6xxx series (e.g., 6061, 6063, 6101 and 6951) is common. Less common is brazing some of the 7xxx series (e.g., 7005 and 7072 used as a cladding material). Brazing followed by quenching, and then followed by natural or artificial aging tempers these heat-treatable alloys. Specific temper gain (or loss) depends on the alloy, on the heated area and temperature.

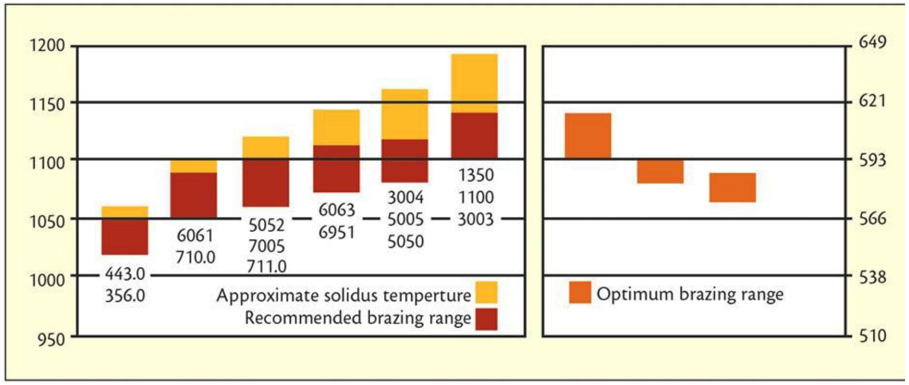


Fig. 1 Brazing temperature ranges of aluminum alloy base and filler metals [1]

High-quality castings (e.g., 443.0, 356.0, 406, 710, 711 and 850.0) can also be brazed. However, die casting alloys are difficult to braze. Also, not all aluminum alloys can be brazed, including the 2xxx series (e.g., 2024 and 2219) and some 7xxx series alloys (e.g., 7075) except as noted above.

All commercial filler metals are themselves aluminum-base alloys. The optimum brazing

Fig. 2 CAB brazed aluminum heat exchanger assembly. Courtesy of SECO/WARWICK Corp.

temperature for an aluminum-base filler metal not only is a function of the filler metal-melting range, but also is dependent on the amount of molten filler metal needed (to fill the joint), and is typically limited by the mutual solubility of the filler metal and the base metal being brazed (Fig. 1).

Brazing Copper And Copper Alloys

Table 1. Considerations in brazing copper and copper alloys

Material	Processing Concern
Aluminum bronzes	Difficult to braze. Alloys with ≤ 8% Al can be brazed with special fluxes.
Brasses, general	Dezincification (loss of zinc by vaporization) is a common problem during heating. Brasses are subject to cracking and should be heated slowly and uniformly. Design is an important consideration.
Brasses, leaded	Susceptible to hot cracking, which increases as a function of increasing lead content. Low residual stresses before brazing are required.
Brasses, naval/leaded naval/admiralty	Brasses containing aluminum require special fluxes.
Brasses, nickel-silver brazed.	Highly susceptible to hot cracking; should be stress relieved before being brazed.
Brasses, red	Flux is normally required when the zinc content is above 15%.
Brasses, yellow	Low melting filler metal should be used to avoid dezincification of the base metal.
Coppers, general	Coppers containing small additions of silver, lead, tellurium or sulfur will have wetting action improved by use of fluxes.
Copper nickels	Susceptible to both hot cracking and stress cracking by molten filler metal. Stress relief is required prior to brazing and care should be taken not to introduce stress during brazing.
Copper, oxygen free and phosphorous-deoxidized	Fluxes are required when using silver-base filler metals containing zinc, cadmium or lithium.
Coppers, precipitation hardenable	Fresh machined or mechanically abraded surfaces improve wetting action. Brazed in the aged condition reduces mechanical properties.
Coppers, tough pitch	Susceptible to embrittlement when heated and exposed to atmospheres containing hydrogen. Heated by open flame or torch may also result in hydrogen diffusion and embrittlement.
Phosphor bronze	Should be stress relieved to avoid cracking, and should be supported while being brazed to avoid reintroduction of stresses.

The ability to braze copper and its alloys is generally rated from good to excellent. They are commonly brazed using copper- and silver-base filler metals. A key concern is the softening of the base metal, which frequently occurs during brazing, because many copper-base alloys derive their properties as a result of heat treatment at relatively low temperatures or through cold working, or both.

The base metals include copper, copper-zinc alloys (brasses), copper-silicon alloys (silicon bronze), copper-aluminum alloys (aluminum bronze), copper-tin alloys (phosphor bronze), copper-nickel alloys and several others. Each has certain characteristics that must be carefully considered so the correct brazing equipment (e.g., furnace, torch, induction, resistance and dip), techniques and protective atmospheres are chosen.

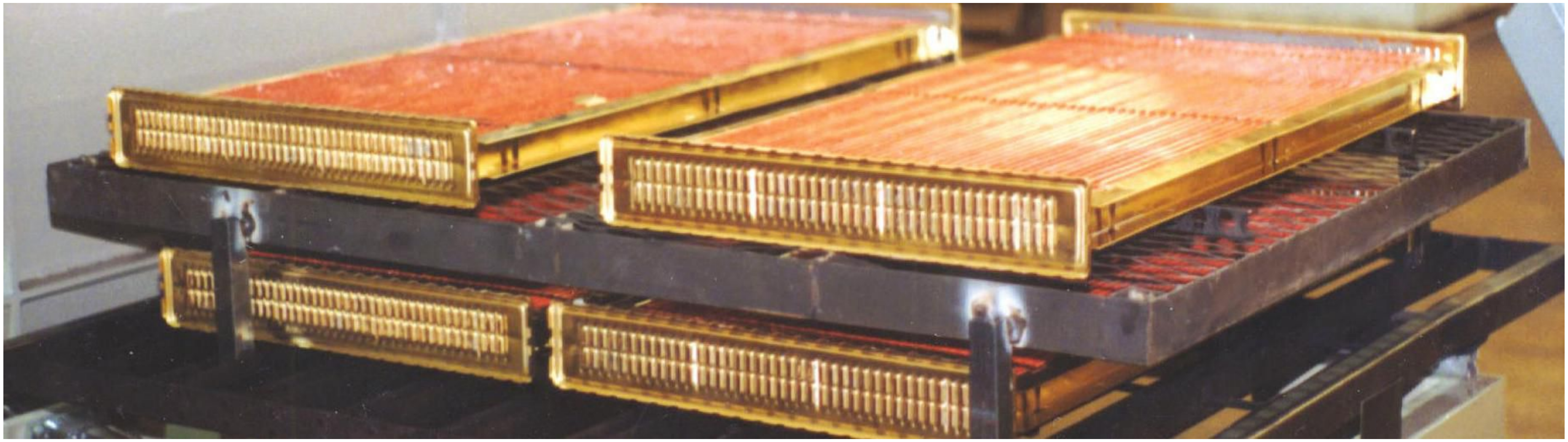


Fig. 3 CUB brazed copper/brass heat exchanger assemblies. Courtesy of SECO/WARWICK Corp.

This requires an understanding of the processing concerns of each material, listed in Table 1.

Brazing Of Heat-Exchanger Components

Today, an important application of aluminum and copper brazing is in the production of components such as heat exchanger cores (Figs. 2 and 3). Two common methods of atmosphere brazing are controlled atmosphere brazing (CAB) of aluminum using the Nocolok® process and the Cuprobraze® (CUB) process for brazing copper/brass heat exchangers under nitrogen based atmosphere. Aluminum brazing in vacuum furnaces is another common method for producing these products, especially for heat exchangers constructed of aluminum alloys containing magnesium.

To meet final product expectations, careful consideration must be given to material selection, forming and machining techniques and part tolerances, as well as fixturing and assembly. The furnace systems used can be very large to meet the needs of high-production manufacturers, such as the automotive industry, or they can be of a smaller scale for the aftermarket producers. Furnace design and cycle selection must also be approached with care. These processes can be done in batch, semicontinuous or continuous designs depending on product mix and production volumes.

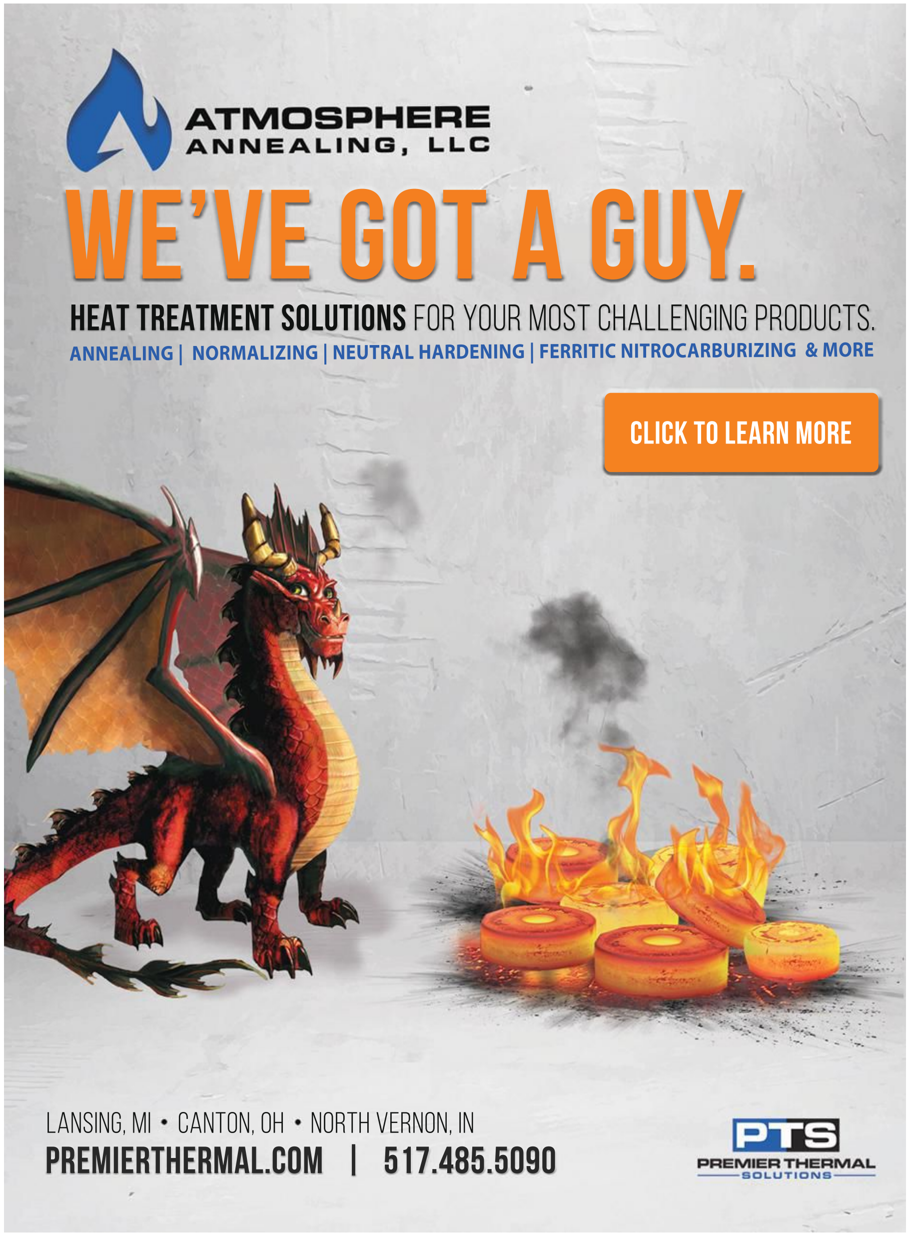
Summary

Brazing of all nonferrous base materials requires special techniques; a careful choice of filler metals; and the use of very low dew point inert atmospheres (e.g., argon), reactive atmospheres (e.g., hydrogen) or vacuum. This is true for all types of nonferrous materials including:

- Beryllium and its alloys
- Magnesium and its alloys
- Nickel, cobalt and superalloys
- Niobium and its alloys
- Reactive metals
- Refractory metals and alloys

Concerns with these materials include the formation of stable oxides and nitrides (that interfere with the flow of the braze alloy), absorption of gases (e.g., oxygen, hydrogen and nitrogen), galvanic corrosion, outgassing and/or the presence of contaminants, moisture, embrittlement by sulfur and low-melting metals (e.g., zinc, lead, bismuth and antimony) and stress cracking. Additional concerns that affect the ability to braze some of these materials include ductile-to-brittle transition behavior, recrystallization temperature and reactivity with carbon.

Filler metals used must be selected on the basis



of service conditions, application, maximum joint strength required and compatibility with the base metal (or coating, if used).

As with all brazing, variables that need to be controlled to produce mechanically sound braze joints include base metal selection and characteristics, filler metal selection and characteristics, component design, joint design and clearance, surface preparation, filler metal flow characteristics, temperature and time, and rate and source of heating.

Remember also that there are a number of factors affecting the ability to produce a metallurgically sound braze joint. Some influence the behavior of brazed joints, while others affect the base metal properties, and in some cases, the interactions between the base metal and filler metal. Effects on the base metal include carbide precipitation, hydrogen embrittlement, nature of the heat-affected zone, oxide stability and sulfur embrittlement. Filler metal effects include vapor pressure, alloying, phosphorous embrittlement and stress cracking. Interaction effects include post brazing thermal treatments, corrosion resistance and dissimilar metal combinations.

Successful brazing of nonferrous base metals depends on considering and planning for all aspects of the process.

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Considerations in Glass-to-Metal and Ceramic-to-Metal Sealing – Part One

There are areas of heat treatment that have been around for a long time, but the knowledge about them is now known by relatively few. Processes for glass-to-metal sealing and ceramic-to-metal sealing are good examples. Let's learn more.

Glass-to-metal seals (Fig. 1) are very important to the construction of vacuum tubes, bomb detectors, electric discharge tubes, incandescent light bulbs, glass-encapsulated semiconductor diodes, reed switches, pressure-tight glass windows in metal cases and metal or ceramic packages for electronics. Glass-to-metal sealing encompasses two types of hermetic (airtight) seals: compression and matching. In a compression seal, the outer member has the highest expansion factor and the inner components have a slightly lower expansion. In a matching seal, all components have a somewhat similar expansion. Heavy-gauge metals are used in a compression-type glass-to-metal seal, while thin-gauge metals are used for matching seals.

Fig. 1. Refrigeration site glasses

A metal that can be attached to glass to produce an airtight (hermetic) seal must have the following general characteristics:

- The melting point must be higher than the working temperature of the glass
- The material should be free of as many non-metallic inclusions as possible
- The thermal-expansion characteristics of both the metal and the glass should be similar (in the case of matched seals)

- No allotropic transformations creating significant changes in the thermal-expansion rate should occur over as wide a range as possible, typically 50-2000˚F (10-1090˚C)
- Any oxide layer formed during the process of making the glass-to-metal seal should adhere firmly to both the metal and the glass
- High electrical and thermal conductivity are required if the metal has to carry substantial electrical current (otherwise the strain rate may increase)
- Ease of joining to other metals by welding or soldering is highly desirable (and often essential)

Glass is used because it has several outstanding properties, including:

- Glass is stable. It is homogeneous, non-porous, smooth, hard and resistant to a wide variety of chemical attack.
- Glass has good electrical properties. Glass has very high dielectric strength (greater resistance to high voltage surges).
- Glass is affordable. It comes in a wide range of dielectric-constant and power-loss-per-unit-volume values at little cost differential.
- Glass has very high volume and surface resistivity values.

One of the most important characteristics of glass-to-metal or ceramic-to-metal seals is that each step of the manufacturing process is critically important. Failure to meet optimum specification requirements will destroy any chance of success. For glass-to-metal sealing (and joining), the following manufacturing steps are generally employed:

- Preparation (cleaning)
- Decarburization (if necessary)
- Oxidation
- Glass application
- Fixturing
- Sealing

In some cases, a stress relief (annealing) operation (for the metal alloy) is also used. For ceramic-to-metal sealing, the following additional steps are also included:

- Metallizing (of the ceramic to provide a wettable surface suitable for joining)
- Cleaning (of the metal component)
- Plating (typically Ni or Cu)
- Joining (soldering or brazing of the metalized ceramic to the metal alloy)

Preparation

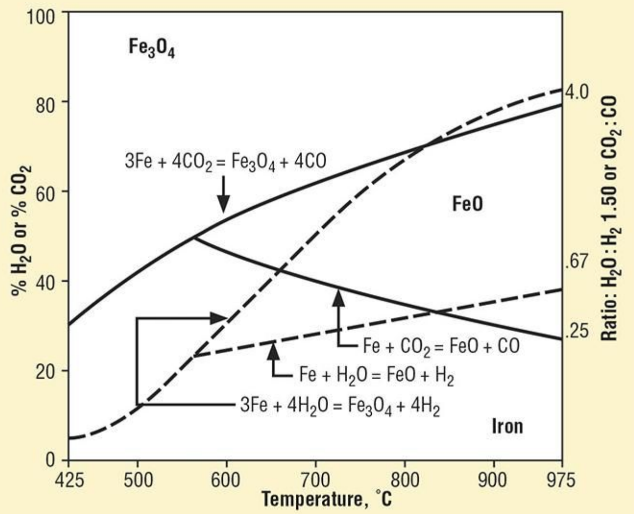
Pre-cleaning

A number of techniques are in use including several multiple-step processes. For example, parts are washed in a 50/50 solution of water and hydrochloric (alternatively hydrofluoric) acid at 150ºF (65ºC) then rinsed in SK-250 followed by an additional wash for 30 seconds in a 50/50 solution of hydrochloric (hydrofluoric) acid at room temperature. They are then rinsed in 104 detergent followed by a deionized water rinse and perchloroethylene rinse and dry. Surfaces are sometimes grit or vapor blasted (using 180-260 grit).

Decarburization/Degassing[1]

The removal of carbon from glass-to-metal sealing alloys is a prerequisite for the production of good glass-to-metal seals. On sealing glass to metal, the presence of carbon in the metal causes bubbles of CO_2 and CO to form at the glass-to-metal interface. These bubbles cause blistering and, therefore, a poor seal. To remove the carbon from the glassing operation, metal parts are usually decarburized in wet hydrogen, dissociated ammonia or nitrogen/hydrogen mixtures provided the hydrogen content is at least 10% and the dew point is carefully controlled. The chemical reaction responsible for decarburization/degassing is:

$$\text{(1)}\qquad C\ \text{(in solution)} + H_2O = CO + H_2$$



Variation of equilibrium constants vs. temperature and atmosphere compositions

	K_1	$\%CO_2$	K_2	$\%H_2O$	D.P.	K_3
650°C	3.77	4.5	0.51	4.5	+32°	1.92
700°C	0.94	2.6	0.65	3.4	24	0.70
760°C	0.35	1.2	0.83	2.0	18	0.36
815°C	0.12	0.5	1.02	1.0	7	0.13
870°C	0.05	0.2	1.22	0.5	-3	0.06
925°C	0.02	0.1	1.44	0.25	-12	0.003

$Fe + H_2O \leftrightarrow FeO + H_2$
$3Fe + 4H_2O \leftrightarrow Fe_3O_4 + 4H_2$
$Fe + CO_2 \leftrightarrow FeO + CO$
$3Fe + 4CO_2 \leftrightarrow Fe_3O_4 + 4CO$
$CO + H_2O \leftrightarrow CO_2 + H_2$

Equilibrium constants

$$K_1 = \frac{CO_2}{(CO)}{}^{-2};\ K_2 = \frac{CO x H_2O}{CO_2 x H_2};\ K_3 = \frac{H_2O}{CO x H_2}$$

Fig. 2. Equilibrium data for oxides of iron[8]

Processing temperatures in the 900-1100˚C (1650-2010˚F) range are typical up to a maximum of about 1205˚C (2200˚F). A dew point range of 20˚ ± 2˚C (68˚ ± 4˚F) is common with about 26°C (79˚F) being a reported maximum. Exposure time is usually less than one hour.

Oxidation[2,3]

Factors that influence glass-to-metal or ceramic-to-metal seals involve adherence issues, including:

1. Oxide thickness – adherence is not developed when the oxide is too thick or too thin. There is a range of oxide thickness where good adherence is developed in a reasonable time. Too thick/thin an oxide requires an unreasonable sealing time.
2. Oxidation temperature – of minor importance as long as oxidation time is sufficient to give an oxide thickness that falls in the optimum range.
3. Optimum sealing time (time to effect the seal, or glass-oxide contact time) – a function of the sealing temperature, and both sealing time and temperature are a function of oxide thickness.

The optimum range of oxide thickness depends on the type of metal and has been reported to be approximately 0.6-1.1 mg/cm². The oxide is composed of two parts an oxide scale and intergranular oxide. The residual intergranular oxide layer should be in the 2.0-6.5 μm (80 - 255 micro-inch) range for best results. Oxidation below optimum thickness will cause the glass to dissolve the oxide and contact the base metal directly. A weak bond and poor seal will result. Over-oxidation is also undesirable because the oxide itself has little shear strength and the glass may become saturated with oxide. These factors also contribute to a poor seal.

In summary, to develop good adherence in a short time, the oxide must not contain alpha Fe_2O_3 and must be thicker than the minimum stated above (Fig. 2). If the oxide contains appreciable amounts of alpha Fe_2O_3, the sealing process is not nearly so straightforward and the conversion of alpha Fe_2O_3 into Fe_3O_4 must be allowed for. Thus, formation of a stable, glass-to-metal seal oxide depends on the creation of a layer of magnetite (Fe_3O_4). A well-oxidized part will have a dark-gray appearance.

One method of oxide formation is air oxidation. Oxidation in air involves reactions that are irreversible and not controllable due in part to the fact that the relative humidity change is constantly affecting the oxygen potential.

$$2Fe + O_2 \rightarrow 2FeO \qquad (2)$$

$$4Fe + 3O_2 \rightarrow 2Fe_2O_3 \qquad (3)$$

$$3Fe + 2O_2 \rightarrow Fe_3O_4 \quad (4)$$

The result of thermal oxidation in air is almost always unsatisfactory with respect to thickness and composition uniformity.

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Considerations in Glass-to-Metal and Ceramic-to-Metal Sealing – Part Two

The second part of our discussion on sealing follows. Let's learn more.

Oxidation[2,3] (continued)

Steam Oxidation

Iron has an equilibrium constant that favors complete Fe_3O_4 formation below 1050°F (565°C) in a steam atmosphere (eq. 6). Above this temperature, a mixture of Fe_3O_4 and FeO is obtained depending on the H_2O content and the H_2O/H_2 ratio. At temperatures below 1525°F (830°C), H_2O is a stronger oxidizing medium than CO. The reactions are as follows:

$$(5)\quad Fe + H_2O = FeO + H_2$$
$$(6)\quad 3Fe + 4\,H_2O = Fe_3O_4 + 4\,H_2$$

Exothermic Gas Oxidation

Carbon dioxide produced in lean exothermic gas can be used to produce an acceptable glass-to-metal seal. Natural gas and air combusted in a specific air-to-gas ratio (typically 7:1 or 8:1) creates an atmosphere that is a mixture of N_2, CO, CO_2 and H_2. Below 1050°F (565°C) in a lean exothermic-gas atmosphere, iron has an equilibrium constant that favors complete Fe_3O_4 formation. Above this temperature, FeO is common unless the dew point is sufficiently high – in the range of +40°F (+4.5°C) as introduced into the furnace chamber. On reheating FeO + Fe to temperatures of approximately 750°F (400°C) but less than 1040°F (560°C) – in the range of mask stabilizing and frit sealing – the oxide changes to Fe_3O_4. Above a temperature of 1525°F (830°C), CO_2 is a stronger oxidizing medium than H_2O.

Table 1. Relationship between dew-point temperature and moisture content of gases[7]

Temp. °F (°C)	Volume of water ppm (%)	Weight of water grains[a]/foot³ (milligrams/liter)	Temp. °F (°C)	Volume of water ppm (%)	Weight of water grains[a]/foot³ (milligrams/liter)
-120 (-84)	241,000 (24.1)	85 (194)	20 (-7)	2,200 (0.22)	0.79 (1.8)
-110 (-79)	193,000 (19.3)	68 (155)	30 (1)	1,800 (0.18)	0.46 (1.0)
-100 (-73)	150,000 (15.0)	53 (121)	40 (4)	750 (0.075)	0.27 (0.60)
-90 (-68)	115,000 (11.5)	41 (93)	50 (10)	410 (0.041)	0.15 (0.33)
-80 (-62)	86,000 (8.6)	30 (69)	60 (16)	220 (0.022)	0.081 (0.18)
-70 (-57)	64,000 (6.4)	22 (51)	70 (21)	120 (0.012)	0.045 (0.10)
-60 (-51)	47,000 (4.7)	17 (38)	80 (27)	67 (0.0067)	0.024 (0.054)
-50 (-46)	33,000 (3.3)	12 (27)	90 (32)	34 (0.0034)	0.012 (0.027)
-40 (-40)	25,200 (2.52)	9.1 (21)	100 (38)	16 (0.0016)	0.0039 (0.013)
-30 (-34)	16,500 (1.65)	5.8 (13.3)	110 (43)	8 (0.0008)	0.0027 (0.0062)
-20 (-29)	12,000 (1.20)	4.2 (9.6)	120 (49)	4 (0.0004)	0.0013 (0.0029)
-10 (-23)	8,200 (0.82)	2.9 (6.6)	130 (54)	2 (0.0002)	0.00057 (0.0013)
0 (-18)	5,500 (0.55)	1.9 (4.4)	140 (60)	0.6 (0.00006)	0.00022 (0.00049)
10 (-12)	3,600 (0.36)	1.3 (2.9)	150 (66)	0.2 (0.00002)	0.00008 (0.00018)

Notes: (a) 7,000 grains per pound

Nitrogen/Hydrogen (Wet/Dry) Oxidation

The most common approach used today involves gases such as nitrogen or argon, a portion of which can be bubbled through water and then combined with a small percentage of hydrogen for use as a furnace atmosphere. The amount of moisture picked up by a gas is a function of the water temperature and the dwell time within the saturator.

Oxidation typically takes place from 1470–1920°F (800–1050°C) in atmospheres that include a mixture of N_2, H_2O and H_2. Water is an easily controlled oxidizing species and provides an adherent and uniform oxide on metal surfaces. Hydrogen is added to protect against air leaks by combining with the oxygen to form water. Some researchers[2] have found that for Kovar, the atmosphere which performed best was N_2–1% H_2O–0.4% H_2 run at 1830°F (1000°C) for 10 minutes. This produced an oxide scale of about 1 μm (40 micro-inch) and an oxide intergranular penetration of about 4 μm (155 micro-inch).

Seal Formation

Parts consisting of preoxidized metal bodies and pins (or leads) are assembled onto fixtures designed to hold the parts in the proper orientation. Fixtures can be machined graphite blocks or ceramic. If graphite is used, attention must be paid to the furnace atmosphere with respect to deterioration (life) of the fixtures. If ceramic, the temperature profile must be considered with respect to thermal shock.

These assemblies are then placed in a furnace operating around 1800°F (1000°C) in a nitrogen/hydrogen or lean exothermic-gas atmosphere (N_2-H_2-H_2O) setup to produce a slightly oxidizing atmosphere. This ensures a good glass seal while not over-oxidizing the surface (making the part difficult to clean). A slow cool from sealing temperature to around 1000-1200°F (540-650°C) is often used to prevent cracking of the glass due to thermal contraction.

Thermal-expansion differences between the metal alloy and the glass must be carefully matched to produce a good seal. Thermal expansion changes significantly as a function of the nickel content of the alloy. For example, a Kovar (Fe-29N-i17Co) alloy has a coefficient of thermal expansion between 77–575°F (25–300°C) of 3.0 ppm/°F (5.4 ppm/°C) while Alloy 42 (Fe-42N) has a coefficient of thermal expansion between 77–575°F (25–300°C) of 4.4 ppm/°F (8 ppm/°C).

Ceramic-to-metal seals do not have as stringent a limitation on thermal expansion because yielding of the soft metal "solder" applied during the joining process results in a reduction of strain. Nevertheless, it is advisable to use comparable thermal-expansion values wherever possible.

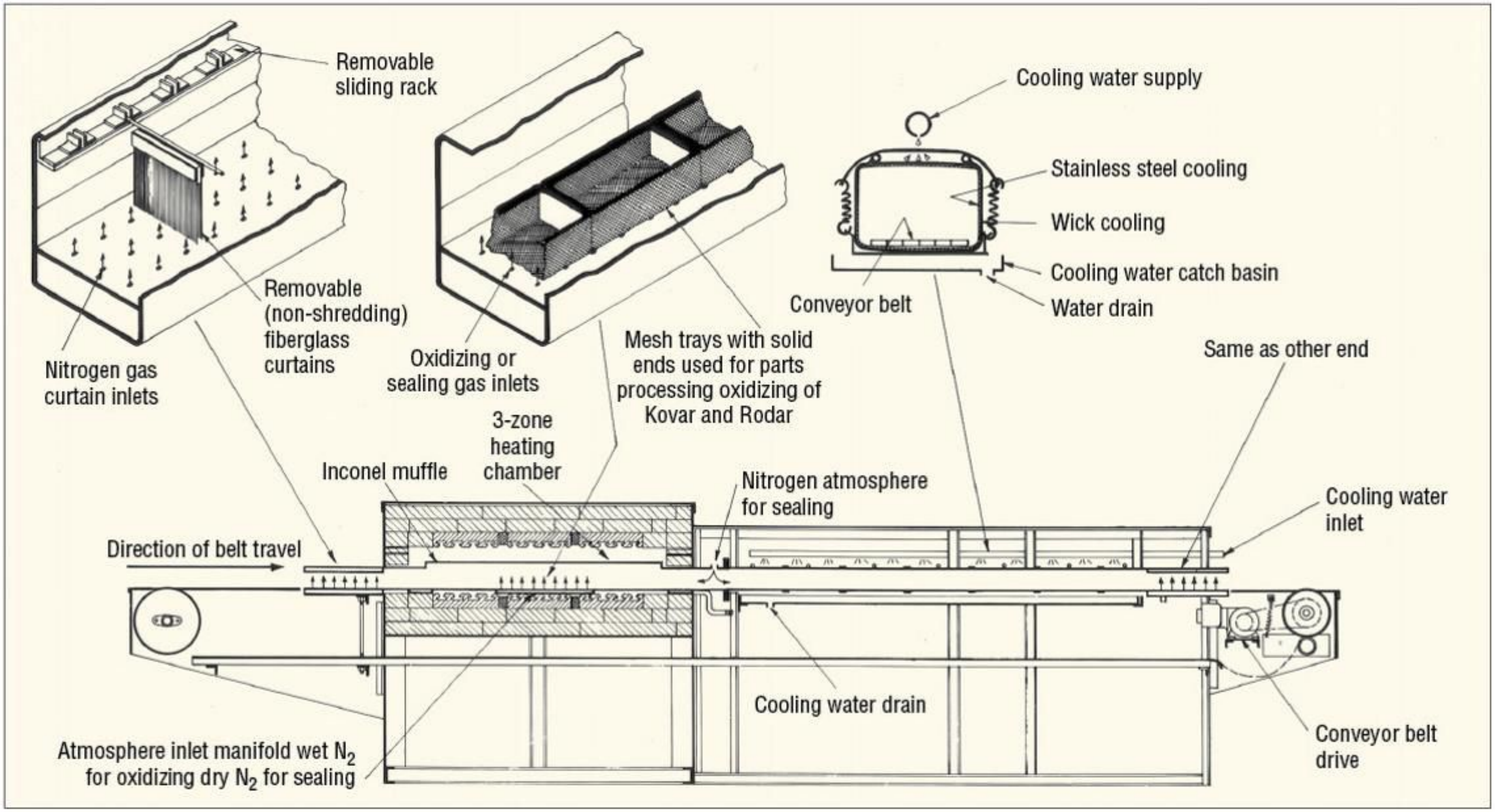

Fig. 1. Glass-to-metal processing furnace model LAC-M-030439 (Courtesy of C. I. Hayes)

Equipment

Separate furnaces are typically used for oxidation and sealing. These generally fall into two broad categories, mesh-belt conveyor furnaces (Fig. 1) or box-style furnaces. Special equipment designs have also been used in the past with great success. The secret is to avoid air infiltration (drafts, etc.) and have an extremely tight furnace. Most glass-to-metal sealing furnaces have nitrogen and fiber curtains (front and rear) with special gas inlets located either just behind the heating chamber or in the floor of the furnace to direct gas movement onto the parts.

Sealing Characteristics

Good seals are bubble free and have metal surfaces that range from light to dark gray and are hermetic (leak free @ 1 x 10^{-7} Torr). By contrast, poor seals are shiny or metallic in appearance (under-oxidized or over-worked), have poor strength, bubbles on the surface of the metal and the oxide that does exist strips off when the seal fractures. They have a tendency to leak in testing or service.

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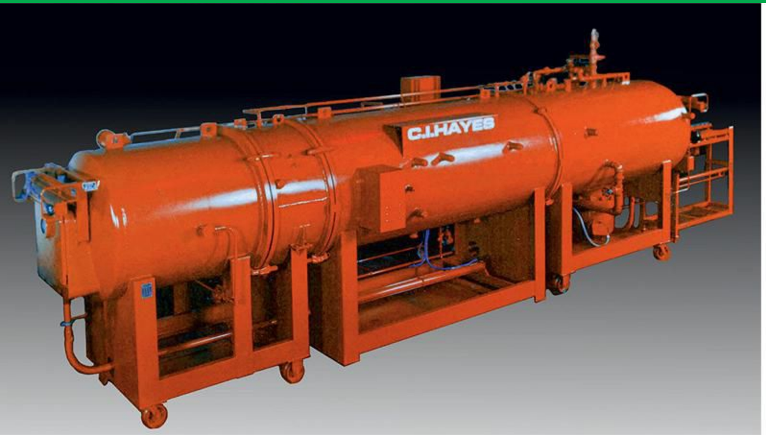

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Quest for Fire – Combustion Basics

Man has always had a primeval fascination with fire and the heat treater is no exception, particularly since he has learned to harness this energy source as a cost-effective solution to heating and processing materials. We are on a continual quest to understand what combustion is and how we can safely and efficiently use this valuable resource. Let's learn more.

The Industrial Heating Equipment Association (IHEA) defines combustion as "the rapid oxidation of a fuel resulting in the release of usable heat and the production of a visible flame." By its very nature, combustion is a complex interaction of exothermic (i.e. heat producing) chemical reactions between a fuel and an oxidant producing both heat and light. The transport of energy, mass and momentum are the physical processes involved in combustion, while the chemical processes are conduction of thermal energy, diffusion of chemical species and the flow of gases, all of which follow from the release of chemical energy in the exothermic reaction. Combustion accounts for approximately 85% of the world's energy.

For "perfect combustion," natural gas and air are combined in a ratio of 10 parts air to one part gas (10:1, pronounced "10 to 1") to produce approximately 1,000 BTU (252 kCal) of energy (Eq. 1). Perfect combustion is defined as the exact stoichiometric ratio (i.e. the balance of products and reactants) in which no excess fuel or air is produced (Fig. 1). In other words, perfect combustion is achieved when 100% of the air (or oxygen) required for combustion is provided. For each 100 cubic feet (2.8 m^3) of air input, 100 BTU (25 kCal) of heat is liberated regardless of the fuel used (hydrocarbon gases, oil, coal, biomass, etc.).

$$(1) \quad CH_4 + \text{Air } (2O_2 + 8N_2) \rightarrow CO_2 + 2H_2O + 8\,N_2$$

Deviations from perfect combustion also impact the analysis of the exhaust (flue) gas. Certain heat-treat applications require either excess fuel to, for example, provide a protective non-oxidizing atmosphere or excess air to, for example, provide an oxygen-rich atmosphere or improve low-temperature uniformity. Adding even small amounts of excess air lowers the flame temperature and the speed of heat transfer (i.e. the radiating "power" of the flame).

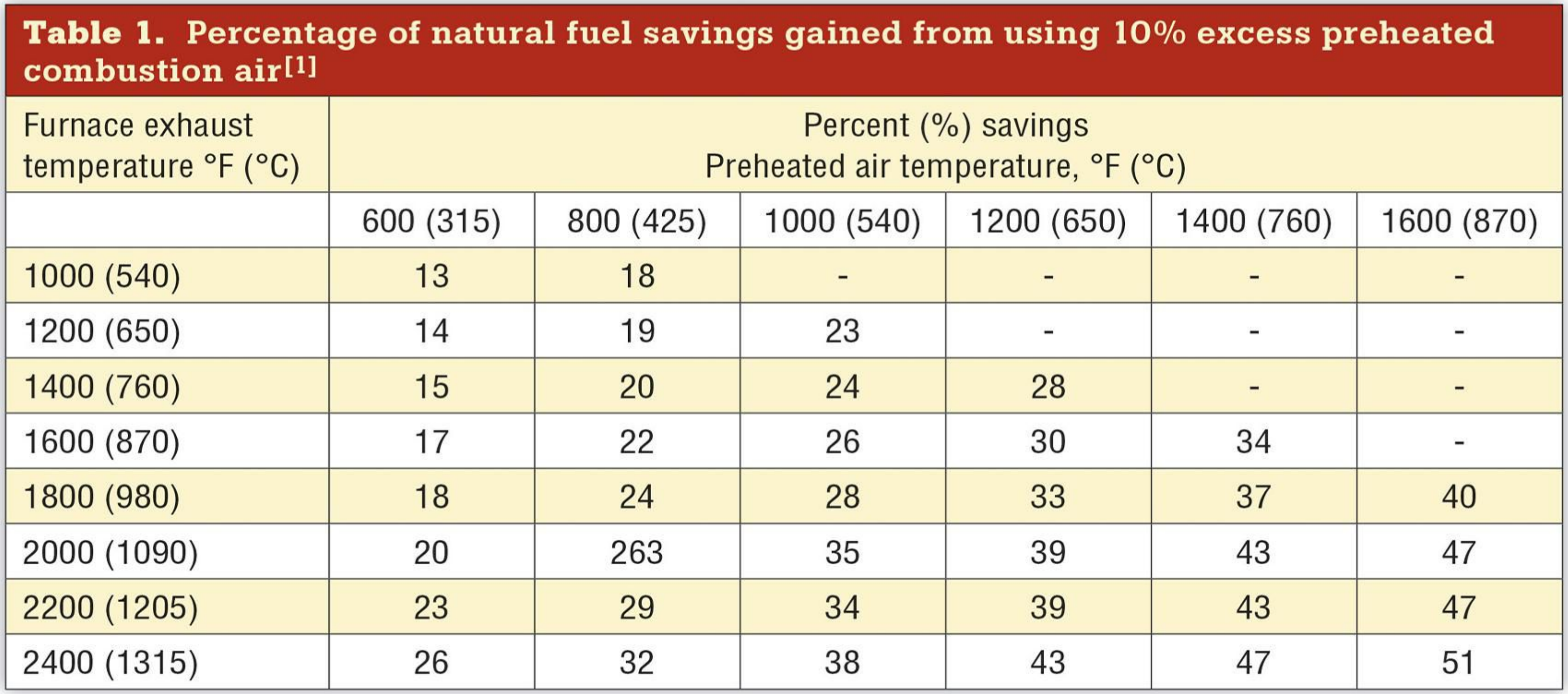


Table 1. Percentage of natural fuel savings gained from using 10% excess preheated combustion air[1]

Furnace exhaust temperature °F (°C)	Percent (%) savings Preheated air temperature, °F (°C)					
	600 (315)	800 (425)	1000 (540)	1200 (650)	1400 (760)	1600 (870)
1000 (540)	13	18	-	-	-	-
1200 (650)	14	19	23	-	-	-
1400 (760)	15	20	24	28	-	-
1600 (870)	17	22	26	30	34	-
1800 (980)	18	24	28	33	37	40
2000 (1090)	20	263	35	39	43	47
2200 (1205)	23	29	34	39	43	47
2400 (1315)	26	32	38	43	47	51

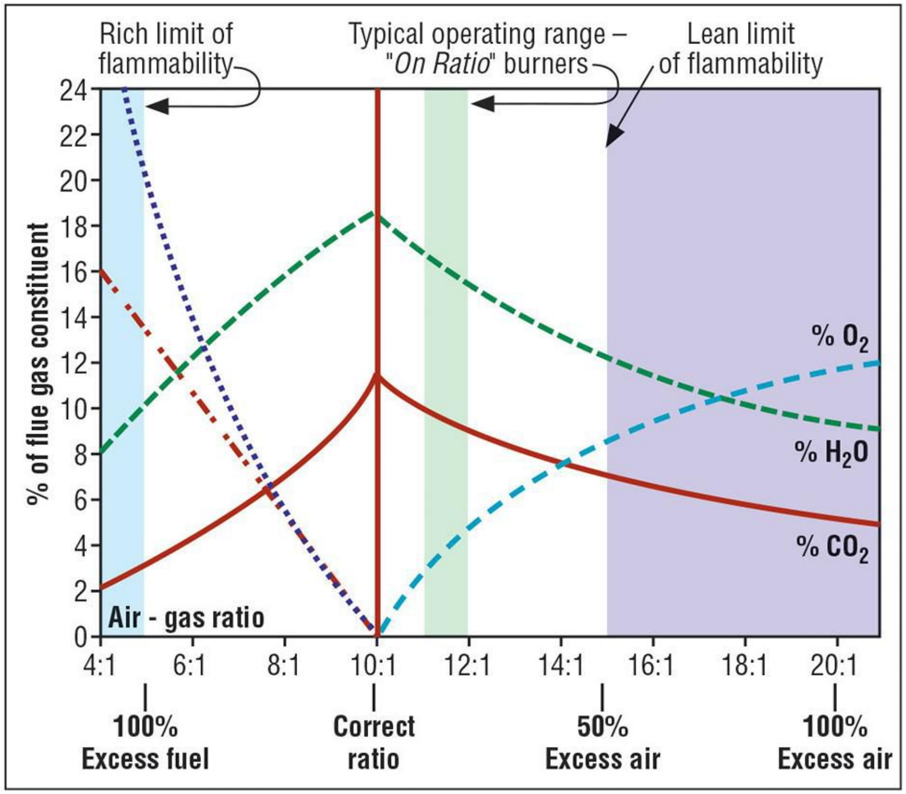


Fig. 1. Fuel-air ratios[2]

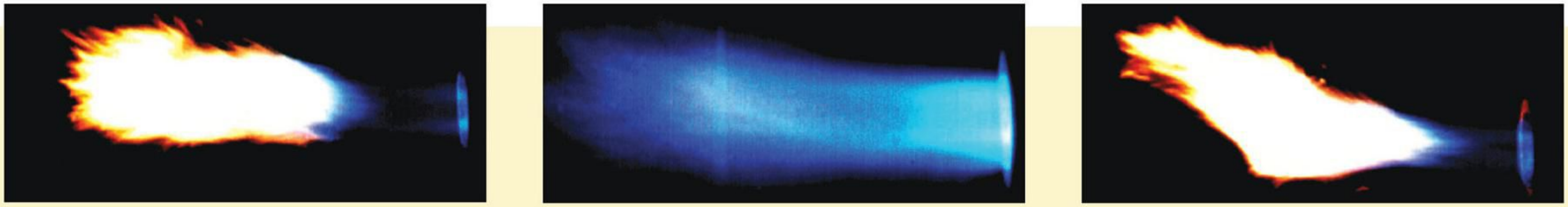
Combustion Basics

The onset of combustion begins with ignition from a source (e.g., pilot, spark igniter or Glo-plug that provides heat to at least 1200°F (650°C). Once ignition commences, requirements for good combustion (Fig 2) are: (a) the proper proportioning of fuel and air to achieve stable combustion, (b) thorough mixing of the fuel and air within the flammability limits of the fuel, (c) initial and sustained ignition of the mixture, and (d) removal of the products of combustion from the process.

The scientific subject areas most relevant to combustion are the fields of thermodynamics (e.g., stoichiometry, gas and gas-mixture properties, heat of formation and reaction, equilibrium and adiabatic flame temperature), transport phenomena (heat transfer by conduction, convection and radiation, mass transfer) and chemical kinetics/fluid dynamics (e.g., laminar and turbulent flow, inertial and viscosity effects, combustion aerodynamics).

In the practical world, a Sankey diagram (Fig. 3) can be used to represent heat loss in industrial furnaces and ovens. Calculations of each individual heat-loss component allows us to determine the exact amount of available heat – that is, the energy left over from the total heat input provided after all losses are accounted for. This is the energy “available” to perform useful work – in other words, how much energy remains to heat the work and balance (make up for) thermal losses.

The available heat can be determined (Fig. 4) from measurements of the flue-gas exhaust temperature and the percentage of excess oxygen in the exhaust. This latter fact is used to determine the amount of



On-ratio (stoichiometric) combustion (all fuel consumed, minimum exhaust volume). The flame is primarily blue near the burner tile with a yellow cone shape. The highest flame temperature (hottest flame) is produced.

Lean combustion (flue products oxidizing – free oxygen). All fuel is combusted but the flame temperature drops due to heating the excess air. Note the pale blue color of the flame and the sharper more conical shape.

Rich combustion (incomplete, air starved or fuel rich, CO and H_2 formed, creating a reducing atmosphere). Flame is predominately yellow, and the shape is less defined. The flame temperature also drops.

Fig. 2. Fuel-air ratios and resultant flame characteristics[3]

excess given the fact that 1% $O_2 \approx$ 5% excess air.

Air for combustion can be supplied to the burner in several ways. Primary air is a term used to describe air supplied and mixed with fuel prior to ignition. This is usually controlled through orifices and valves where all combustion air is mixed with the fuel and is ready to ignite as soon as it reaches the burner nozzle. Sealed-nozzle mixed burner systems depend entirely on primary air. By contrast, secondary air is supplied to the flame after it is ignited and is brought in at the burner. An example of both types is in atmospheric burners that use about 70% primary air and 30% secondary air.

The percentage of fuel savings[5] for any operating condition (preheated air, excess air, oxygen-enriched air) or combination of conditions can be determined (Eq. 2).

Classification of Heating Systems

$$\frac{\%\,\mathit{fuel\ saved}}{100} = \frac{\mathit{fuel\ used}(a) - \mathit{fuel\ used}(b)}{\mathit{fuel\ used}(a)} =$$

$$\frac{\dfrac{\mathit{required\ input}(a)}{\%\ \mathit{available}(a)} - \dfrac{\mathit{required\ input}(b)}{\%\ \mathit{available}(b)}}{\dfrac{\mathit{required\ input}(a)}{\%\ \mathit{available}(a)}} =$$

$$\frac{1/A(a) - 1/A(b)}{1/A(a)} = 1 - \frac{\%\ \mathit{available}(a)}{\%\ \mathit{available}(b)}$$

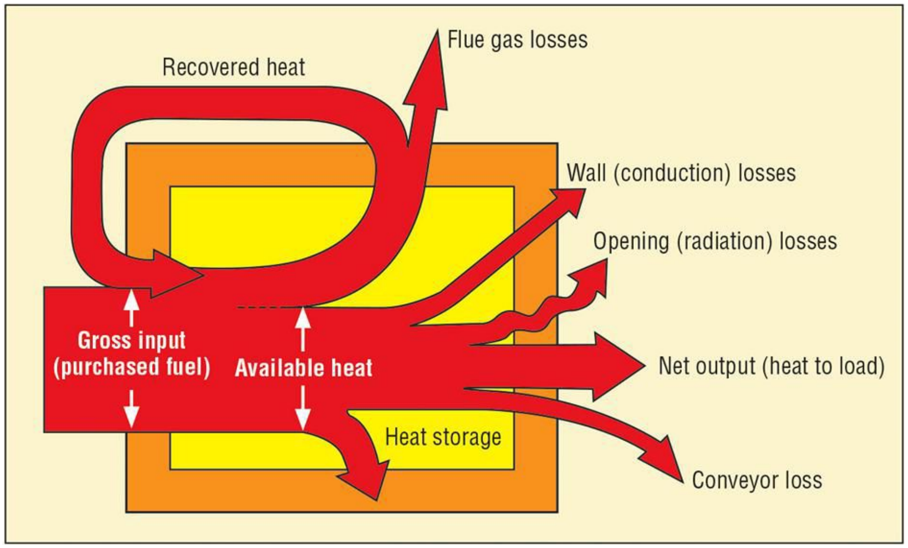


Fig. 3. Heat losses in a continuous furnace[2]

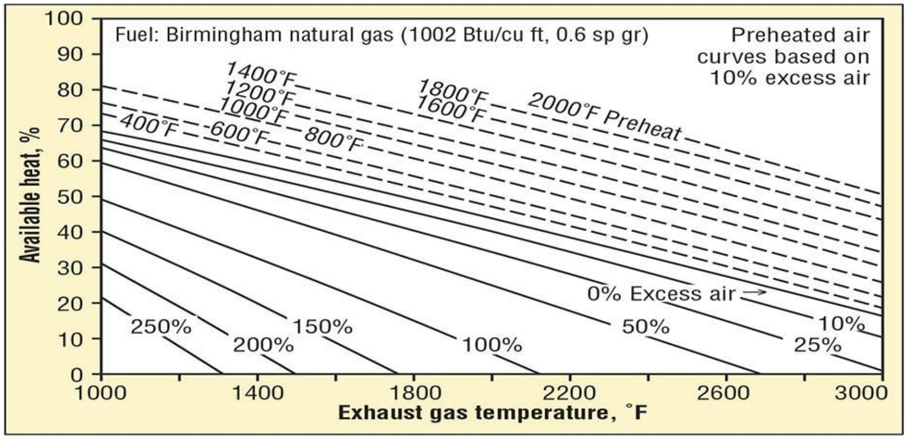


Fig. 4. Available heat input[2]

Combustion systems can be divided into two general categories: direct-fired and indirect-fired systems. In direct-fired applications, the products of combustion are exposed to the work, whereas indirect systems fire into radiant tubes or find the work protected from the flame by such items as retorts and muffles. In most direct-fired combustion systems (e.g., box furnaces), secondary air is pulled into the furnace through leaky doors, other openings and negative furnace pressure.

Recuperative systems also have provided many benefits. There are two types of air preheaters: recuperators (Fig. 5) and regenerators. Recuperators are gas-to-gas heat exchangers placed on the furnace stack. Internal tubes or plates transfer heat from the outgoing exhaust gas to the incoming combustion air while keeping the two streams from mixing. Recuperators are available in a wide variety of styles, flow capacities and temperature ranges. Regenerators include two or more separate heat-storage sections. Flue gases and combustion air take turns flowing through each regenerator, alternately heating the storage medium and then withdrawing heat from it.

Typical Payback Period = (Cost of combustion air preheating system, obtained from the supplier or contractor) ÷ (Reduction in fuel usage, Million Btu/hr × Number of operating hours per year × Cost of fuel per Million Btu).

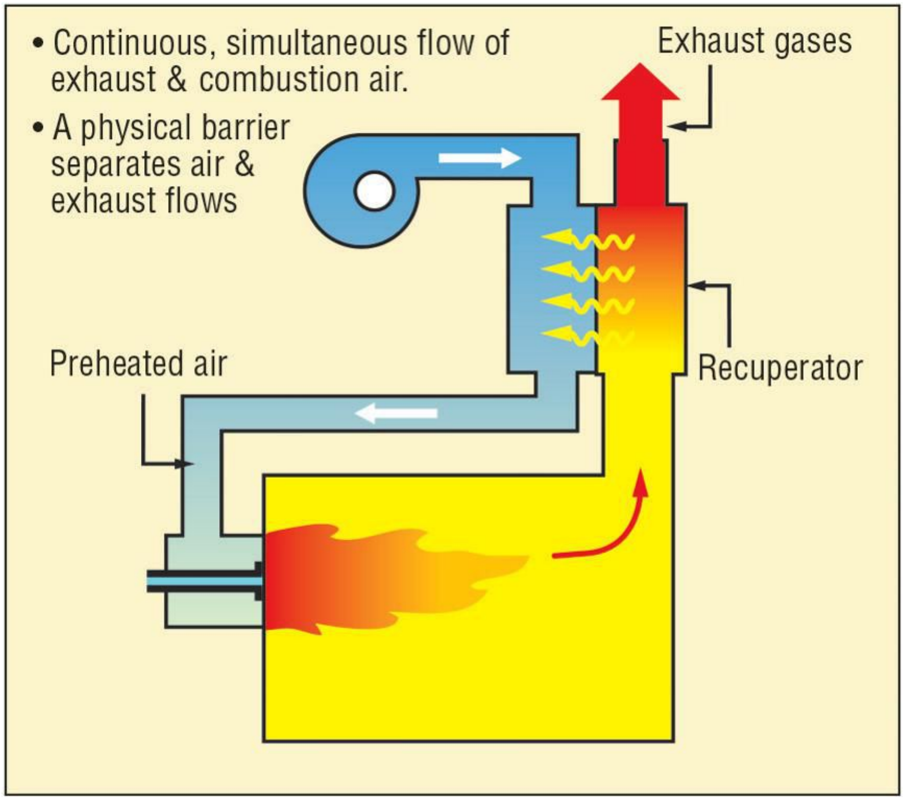


Fig. 5. Recuperative systems[2]

Good Combustion Practice

Understanding the basics of combustion will provide tangible benefits to the heat treater. Benefits include:

- Faster heat-up times and load recovery (due to higher flame temperatures and greater heat transfer)
- Greater efficiency (more available heat)
- Reduced pollution (minimum exhaust volumes, reduced fuel use)
- Cost savings (more cost competitive versus alternative energy sources).

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Electric Heating Elements Part One: Silicon Carbide

Electric heating elements are a popular choice of many heat treaters. They come in a variety of shapes, sizes and materials. One of the most common types are silicon carbide heating elements, known by several tradenames including Globar® and StarBar®. They are used extensively throughout the heat-treating industry when high temperatures, maximum power and heavy-duty cycles are required. Let's learn more.

A silicon carbide (SiC) heating element (Fig. 1) is typically an extruded tubular rod or cylinder made from high-purity grains of silicon carbide that are fused together by either a reaction-bonding process or a recrystallization process at temperatures in excess of 3900°F (2150°C). The result is a chemically stable material with a low thermal-expansion coefficient and little tendency to deform.

Recrystallization forms fine grains of silicon carbide that act as "bridges" or connection points between larger grains, thus forming conductive pathways. The number of bridges formed dictates the material's resistance – the greater the number, the lower the resistance. The secret to the creation of a good heating element is controlling this formation process within the material so as to develop a consistent electrical resistance.

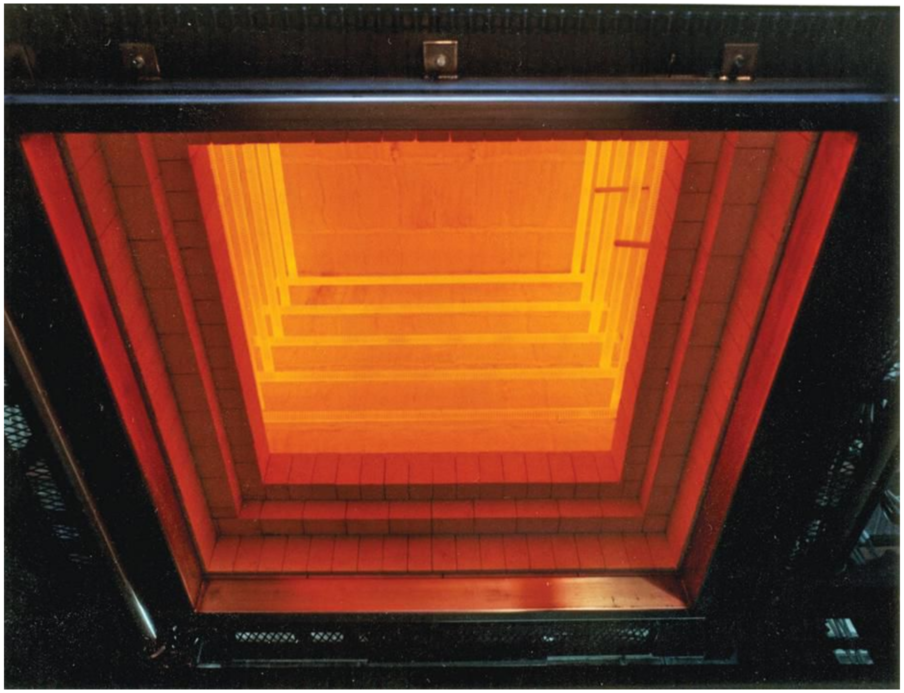

Fig. 1. Silicon carbide heating elements in operation[2] (Photograph Courtesy of Keith Company)

The factors that influence the life of a silicon carbide heating element include the type of furnace atmosphere, watt density, operating temperature, type of service (continuous or intermittent) and

Table 1[3] **The Influence of Furnace Atmosphere on Element Life**

Atmosphere Type	Recommended maximum operating temperature °F (°C)	Operating Limits watts/in^2	Effect on Element
Ammonia	2370 (1300)	25-30	Reduces silica film; forms methane from silicon carbide
Argon	Maximum	Maximum	No effect
Carbon dioxide	2730(1300)	20-25	Attacks silicon carbide
Carbon monoxide	2800 (1540)	25	Attacks silicon carbide
Endothermic Gas			
18% CO	Maximum	Maximum	No effect
20% C	2500 (1370)	25	Carbon pickup
Exothermic	Maximum	Maximum	No effect
Halogens	1300°F (700°C)	25	Attacks silicon carbide and reduces silica
Helium	Maximum	Maximum	No effect
Hydrogen			
+75°F	2370 (1300)	25-30	Reduces silica film; forms methane from silicon carbide
-60°F	2000 (1095)	25-30	
Methane	2400 (1315)	20	Hot spotting from carbon pickup
Nitrogen	2500 (1370)	20-30	Forms insulating carbon nitrides
Oxygen	2400 (1315)	25	Oxidizes silicon carbide
Sodium	2400(1315)	25	Attacks silicon carbide
Sulfur Dioxide	2400 (1315)	25	Attacks silicon carbide
Vacuum	2200 (1315)	25	Below 7 microns, vaporizes silicon carbide
Dew Point			
+60°F	2000° (1090)	20-30	Reacts with silicon carbide to form silicon hydrates
+50°F	2000 (1090)	25-35	
0°F	2500 (1380)	30-40	
-50°F	2800 (1540)	25-45	

Notes:
1. Guidelines only. Specific operating parameters (e.g., heat-up rate, frequency of turning equipment on and off, atmosphere fluxuation) can dramatically influence the values shown.
2. Temperatures are element temperatures, which are usually 100-150°F (55°-80°C) above control temperature settings.
3. In especially aggressive atmospheres, silicon carbide element life can be improved by applying special glazing and coating treatments.
4. In carbonaceous atmospheres, silicon carbide elements will pick up carbon. This process, although slow, can be noted by a gradual change in amperage.
5. In extreme cases, silicon carbide elements can be encased in metallic or ceramic tubes to protect them from the surrounding environment.

maintenance. Furnace type, design and loading play an important role as well. Silicon carbide heating elements (Fig. 2) are extremely versatile, operating, for example, in air up to 3000°F (1650°C).

Transformers used for silicon carbide heating elements have multiple secondary taps in anticipation of a change in resistance of the elements over time. Silicon carbide heating elements, being 20–30% porous, oxidize or otherwise react with the furnace atmosphere and increase in resistance during their operational life. Oxidation causes a reduction in the cross-sectional area of the bridges, resulting in greater resistance to electrical flow. The oxygen in the air reacts with the silicon carbide grain, reducing it to silica (SiO_2) as shown by the equation 1 below:

$$(1) \quad SiC + 2O_2 \rightarrow SiO_2 + CO_2$$

It is estimated that new silicon carbide bars will increase in resistance 10–15% on startup, which should be taken into consideration when considering replacing the bars. In most cases, silicon carbide heating elements fail mechanically long before they fail due to aging.

Tips for Extended Service Life

To maximize element life, be sure to do the following:

1. Handle the elements with care – Silicon carbide heating elements have low tensile strength and, therefore, are sensitive to mechanical damage from rough handling, dropping (even in the packaging) or forced bending that can occur during storage, unpacking or installation.
2. Match resistance – The purpose of matching resistance of elements is to improve their life and to improve temperature uniformity in the furnace. Silicon carbide heating elements are typically factory tested with the test amperage marked on the shipping box and/or the element. Elements can be connected in parallel (preferred since they tend to come into balance in use), series or series-parallel. Elements connected in parallel should be matched in resistance within ±20%, while elements connected in series should be matched within ±5%.
3. Choose the proper size element for the equipment – If there are any doubts about the size to use, check the design parameters with the original furnace equipment manufacturer.
4. Install carefully – Check that the terminal holes through the insulation are in alignment so that the elements slide in without striking the opposite side or are put under tension due to forcing them into position. Be sure to center the elements in the furnace chamber so that no portion of the heating section of the element is in the brickwork.
5. Pack the element with ceramic fiber to a depth of about 1 inch (25 mm) so as to avoid heat loss, but be sure that the terminal ends of horizontally mounted elements lie flat in the terminal holes and are supported by the furnace walls.
6. Use the lowest voltage that will maintain the desired furnace operating temperature. This will ensure the

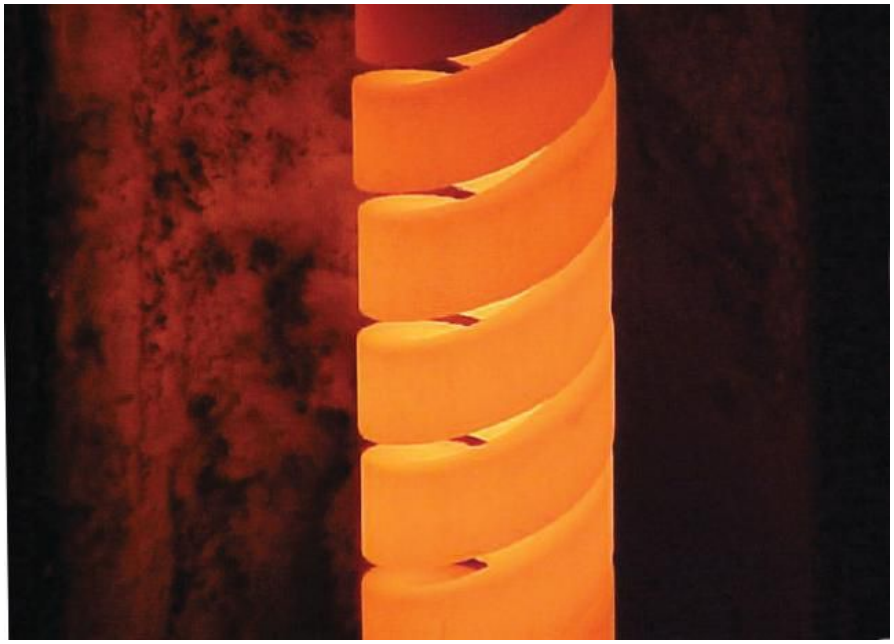

Fig. 2. Spiral-cut silicon carbide heating-element design provides increased resistance for applications up to 3000ºF (1650ºC)

lowest possible surface temperature of the element and lengthen its service life.

7. Run the correct silicon carbide element watt density for the required furnace atmosphere (Table 1).
8. Perform in-service inspections – Check the amperage as an indication that the elements are operating correctly.
9. Maintain matched-resistance circuits at all times. Don't mix old and new elements in the same circuit.
10. Be sure that the elements are loose in the terminal holes not only when the furnace is cold but also hot.

How Do You Know It's Time to Change Heating Elements?

Furnace type, design and cycling make a difference. Let's answer this question by considering a mesh-belt copper-brazing furnace with a muffle operating at 2050°F (1120°C) in a hydrogen/nitrogen (75%/25%) atmosphere. The furnace is run six days a week with a typical belt loading of 10–12 lbs/linear foot (15–18 kg/linear meter). Typical element life is expected to be in the range of 12–24+ months. Here's how to determine when its time to change elements:

1. Beginning with a change of elements, measure the amperage and voltage to the individual elements on a quarterly basis. Calculate their resistance and watt input to determine whether they are balanced or not. If a particular element shows erratic readings or large changes in resistance from one set of readings to the next, it is time to replace it. Note: Silicon carbide heating elements should be changed in sets depending on how many elements are in series with one another (sometimes in pairs, sometimes more).
2. Also, there is a relatively simple procedure that should be done to take the guesswork out of knowing when to change elements. With all new elements, lower the furnace temperature (and monitor it during this procedure) and place the power controller (SCR) in manual mode calling for 100% power. Take a digital meter with extra long leads and, CAREFULLY touching the element on either end, measure the voltage drop across the element. Note: Do not take this reading by touching the straps as they run slightly cooler and your reading won't be as accurate. Measure the current using a clamp-on ammeter around the straps and calculate resistance and watt input. Record this information for future reference. When the elements have doubled in resistance, it is time to change elements. Repeat these measurements every quarter.
3. When you see that the elements have degenerated to about 2/3 of their original resistance value, it is time to change to the next highest tap on the transformer.

4. If you are fully "tapped up" – on the highest tap setting on the transformer – and a particular zone begins to "struggle" (when it has difficulty maintaining your temperature set point), then you know it's time to change all of the elements in that zone.

Summary

The choice of heating element depends on many factors. For example, silicon carbide elements are capable of higher operating temperatures and higher watt loadings than say metallic elements. They are self-supporting and can be used in furnaces either too wide or too long to be spanned by other element types and are relatively easy to change while hot. Silicon carbide heating elements are used extensively in brazing and sintering furnaces running continuously at or above 2050°F (1120°C) and for other processes where the temperature range lies between 2375°-2725°F (1300°-1500°C).

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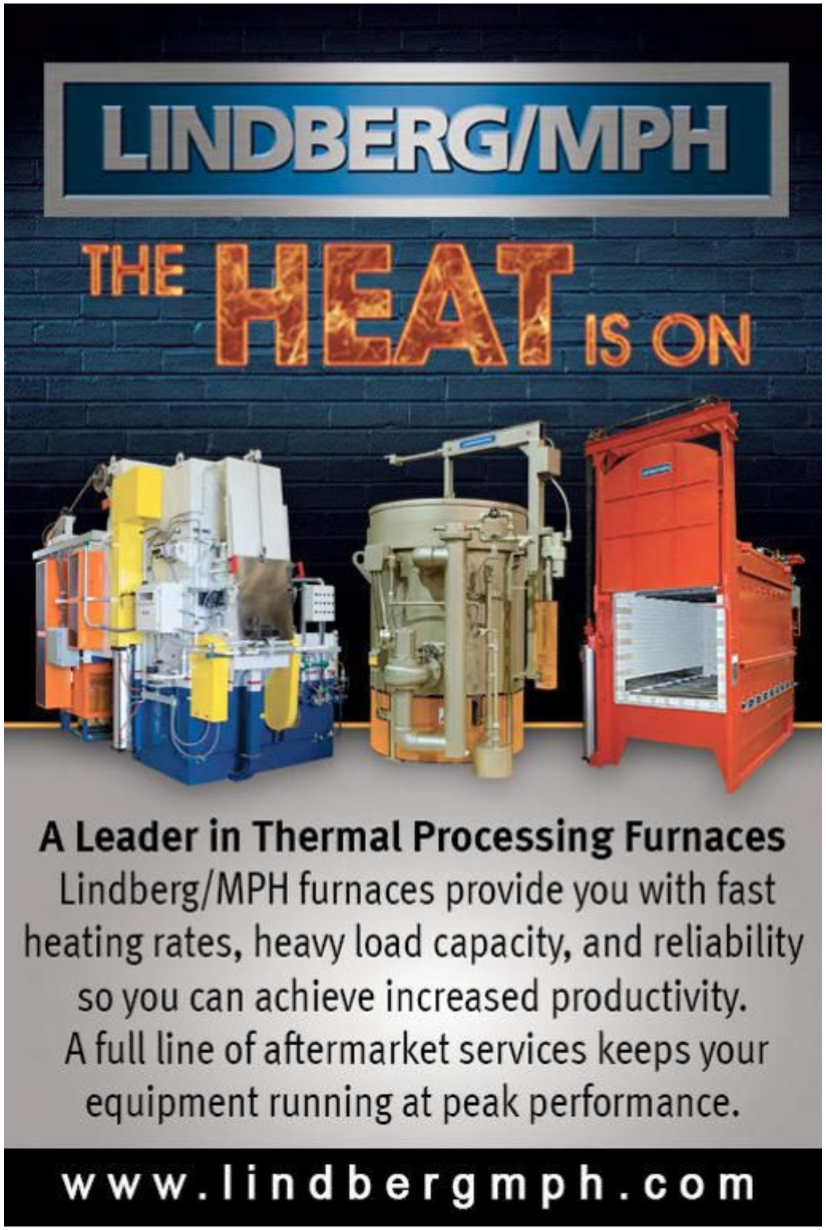

Electric Heating Elements Part Two: Metallic Elements

Electric heating elements in the form of coil, ribbon or rod (Fig. 1) made from alloys such as nickel-chromium, iron-chromium-aluminum and refractory metals are widely used throughout the heat-treating industry. They are found in both low- and high-temperature furnaces and perform well in cyclic-duty service. Let's learn more.

The temperature of a heating element and its surroundings is primarily dependent on the rate at which energy is being supplied and the rate at which it is able to transfer this energy – in the form of heat – to its surroundings. There is a critical rate of heat exchange, reflected in a design value called the watt-density, measured in watts/in^2. Ideally, the power being generated is only slightly greater than the heat-transfer rate demanded by the load. A good match means that the elements will not run too hot, will heat the load in a reasonable amount of time and will not fail prematurely. Good element life and good temperature uniformity within the workload area are the result. The elements themselves can be supported from the furnace sidewalls, suspended from the roof or laid on the furnace floor. Refractory, alloy or ceramic hooks, hangers and supports are commonplace as is placing the elements into ceramic "form" tile.

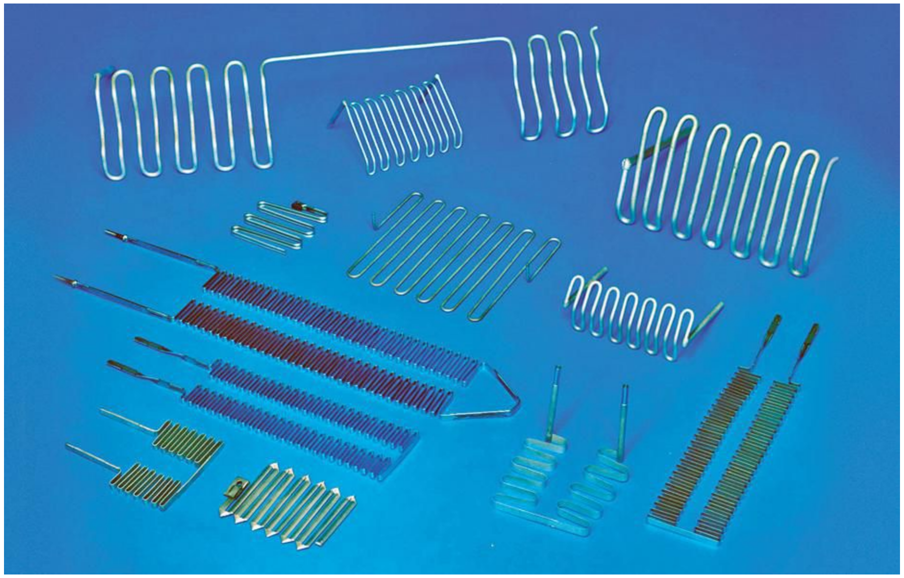

Fig. 1. Typical metallic heating-element types[1]

The type of alloy used depends on the furnace temperature rating (Table 1) and the type of atmosphere used (Table 2). The factors that influence the life of a metallic heating element include the type of furnace atmosphere, watt density, operating temperature, type of service (continuous or intermittent) and maintenance. Furnace type, design and loading play an important role as well.

Table 1. Heating element type by furnace temperature rating[2]

Heating Element Type	Chemical Composition	Maximum Chamber Temperature °F	Atmosphere Types [5]	Special Remarks
Refractory Metals				
Molybdenum	Mo	3200˚F	Vacuum, hydrogen or dissociated ammonia	High-purity alumina brick recommended in atmosphere furnaces; Used in annealing, brazing and degassing refractory metals and ceramic firing (all types)
Tantalum	Ta	3700˚F	Vacuum	Same as above
Tungsten	W	4500˚F	Vacuum	Same as above
Nickel-Iron		2200˚F	Vacuum	Annealing, brazing, degassing refractory metals, ceramic firing (all types)
Nickel-Based Alloys				
Nickel-Chromium 35 – 19	35% Ni, 18.5 - 20% Cr, bal. Fe	1700 - 1800˚F	Hydrogen, dissociated ammonia, forming gas, nitrogen/ hydrogen mixtures, nitrogen, air, endothermic (<0.5%C), exothermic	Chromel "D" or (formerly) Chromax. Applications include annealing, brazing, enameling, hardening, normalizing, sintering, soldering, stress relief and tempering
68-20	68%Ni, 20% Cr, 8.25%Fe, 1.5%Si, bal. Ni	2200˚F	Same as above	Chromel "AA," use 80-20 pins
60-16	60% Ni, 16% Cr, bal . Fe	1850˚F	Same as above	Nichrome or Chromel "C"
80-20	80% Ni, 20% Cr	2100˚F	Same as above	Nichrome V or Chromel "A"
Iron-Based Alloys				
Iron-Aluminum	22% Cr, 5.5% Al, 0.5% Co, bal. Fe	2300˚F	Air, oxidizing or dry hydrogen atmospheres, non-reducing types	Kanthal A-1. Element should be fully supported on ceramic. Applications include annealing, brazing, enameling, hardening, normalizing, sintering, soldering, stress relief and tempering
	22% Cr, 5.5% Al, 0.5% Si, 0.1%C, bal. Fe	2300˚F	Air, oxidizing or dry hydrogen atmospheres, non-reducing types	Formerly Hoskins #875.
Chromium-Aluminum	20%Cr, 5%Al, 1.5%Si, 0.05%C, bal. Ni	2200˚F	Hydrogen, dissociated ammonia, forming gas, nitrogen/hydrogen mixtures, nitrogen, air, endothermic (<0.5%C), Exothermic	Use 80-20 pins
Molybdenum disilicide	$MoSi_2$	3000˚F	Air or oxidizing atmospheres (only)	Kanthal Super

Notes: 1) Guidelines only. Specific operating parameters (heat-up rate, frequency of turning equipment on and off, atmosphere fluctuation) can dramatically influence the values shown. 2) Temperatures are maximum element temperatures, which are typically 50-100°F (10-40°C) above control temperature (process) settings. 3) When using any of these elements near their upper temperature limit, current proportioning control using saturable core reactors or silicon-controlled rectifiers is highly recommended. 4) If the basic control function is to be derived from a thermocouple located inside a retort, muffle or hood, a second high-limit thermocouple must be used and connected to a thermocouple in the element space and set not to exceed the extreme temperature limit of the element involved. 5) See Table 2 for details.

By contrast, many ovens use sheathed heaters in which the heating coil is enclosed in a metallic sheath that is packed with a magnesium oxide (MgO) insulating material. These tubular heaters are offered with a choice of sheath materials for different temperatures and environments, including steel, copper-coated steel, Incoloy®, Inconel® and stainless steel.

Tips for Extended Service Life

To maximize element life, be sure to do the following:

1. Understand that for every 1% increase in voltage, the result will be a 2% increase in power. This is especially important since most power utilities in the U.S. can fluctuate as much as ±10% of nominal voltage. If purchasing a new electric furnace, be sure to accurately measure your plant voltage, and convey this to the OEM to have them design accordingly.
2. Know the design limitation (watt density) of the heating elements. If accurate wattage is important, test the finished element design to determine the proper allowance for rise in resistance with temperature.
3. If more power is needed, increase the diameter of the element wire or reduce the length of the element.
4. Leave adequate room for expansion and contraction. If an element must be anchored between terminals, monitor it to ensure that excess warpage or creep (movement under its own weight over time) will not adversely affect the operation of the element or equipment.
5. Understand the cyclic nature of your application. Elements need adequate space to move on their hangers or supports. Do not locate elements so close to the bottom of a furnace or to a refractory shelf that expansion will cause them to rest on the refractory, potentially creating an area where heat will not dissipate from the element, allowing a hot spot to develop.
6. Install carefully. Check that the terminal holes through the insulation are in alignment so that the elements slide in without striking the opposite side or are put under tension due to forcing them into position. Be sure to center the elements in the furnace chamber so that no portion of the heating section of the element is in the brickwork.
7. Design for the proper element voltage. Do not run an element designed for 230 volts on a 460-volt supply.
8. Keep all types of contaminants and foreign substances away from the elements, including sulfur-based compounds (these form low-melting eutectics with the nickel in the heating element and result in premature element failure,

Table 2. Heating-element type as a function of furnace atmosphere type[2]

Heating Element Material	Atmosphere Type - Oxidizing	Atmosphere Type – Neutral		Atmosphere Type - Reducing					Atmosphere Type - Vacuum	
	Air O_2	Ar, He	N_2	Lean Exo	Rich Exo	Endo (>0.5%C)	D.A	H_2 (dry)	1 Torr	1 x 10^{-4} Torr
Fe-Ni-Cr	1-1750°F	1-1750°F	1-1750°F	2-1700°F	3-1600°F	4	1-1700°F	1-1700°F	4	4
80-20	1-2150°F	1-2150°F	1-2150°F	1-2150°F	2-1850°F	3*	1-2100°F	2-2050°F	2- 2000°F	2-1850°F
Al-Fe-Cr	1-2500°F	2**-2500°F	2**-1800°F	2**-2300°F	4	4	3**-2300°F	2-2450°F	3** - 2200°F	4
Inconel	5	5	5	5	5	1-1200°F	2-2100°F	2-2100°F	5	5
Ni-W	4	6	6	4	6	6-2000°F	6-2450°F	6-2450°F	6-2450°F	6-2400°F
Pt	2-2750°F	2-2750°F	2-2750°F	5	5	5	5	5	4	4
Mo	4	2-3500°F	4	4	4	4	1-3500°F	1-3500°F	1-3500°F	1-3500°F
Cb	4	2-3500°F	4	4	4	4	4	4	2-4000°F	2-4000°F
Ta	4	2-4500°F	4	4	4	4	4	4	2-4500°F	2-4500°F
W	4	2 – 5000°F	4	4	4	4	2 – 5000°F	2 – 5000°F	2 – 5000°F	1 – 5000°F
Cermets	6-2900°F	6	6	6	6	6	6	6	6	4
Silicon Carbide	1-3150°F	2-2250°F	2-2250°F	2-2500°F	2-2500°F	3*- 2000°F	2-2400°F	2-2400°F	5	5
Super Kanthal	1-3100°F	2-2700°F	2-2900°F	4	4	4	4	4	3 -2650°F	4
Graphite (Carbon)	4	2 -5500°F	2 -5500°F	5	5	5	2 -5500°F	2 -5500°F	2-4500°F	2-4500°F

Notes: 1) Guidelines only. Specific operating parameters (heat-up rate, frequency of turning equipment on and off, atmosphere fluctuation) can dramatically influence the values shown. 2) Rating: 1 = good, 2 = fair, 3 = poor, 4 = bad, not recommended, 5 = not used, 6 = experimental. 3) Asterisks: * = Carbon deposits, arcing, ** = pre-oxidized, periodic re-oxidation

phosphorus or oil. Avoid contaminants such as excess cleaning compounds that may build up on the surface of an element over time, creating an insulating layer. If melting has occurred inside a ceramic support plate or form tile, replace it.

9. Welded joints between element sections are best. Pressed or pressure joints can be used but must be thoroughly tested.
10. Be sure that the elements are properly secured to the terminals, and periodically check that the connections remain tight (this must be done with the power off).

Common Problems

"Green rot," a type of corrosion phenomenon that takes place in heating elements made of 80-20 alloy, occurs in the temperature range between 1600-1900°F (870-1040°C). This attack is a result of preferential oxidation of the chromium by the furnace atmosphere when it is oxidizing to chromium and reducing to nickel. Atmospheres that are oxidizing or reducing to both chromium and nickel do not exhibit this type of attack.

Another common problem with 80-20 and 35-19 electric heating elements occurs if they are exposed to a high carbon potential (>0.50%C) atmosphere. Changes to their electrical characteristics and/or failure by mechanical collapse (softening) or melting can occur, especially at temperatures above 2000°F (1090°C).

Eutectic melting, most notably in nickel-based alloys in the presence of sulfur (>25 grains per 100 cubic feet of atmosphere), can be catastrophic to electric heating elements. The Ni-S eutectic is 1175°F (635°C).

Summary

There are uses for metallic heating elements throughout the heat-treating industry including open element rod, ribbon and wire designs, embedded or ceramic/fiber-supported coils, band heaters,

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ceramic segment heaters, cast-in aluminum heaters, sheathed heaters and cartridge heaters to name a few. The secret to the successful application of each design is to understand its limitations and how it is being applied in a given application. Used properly, each type of metallic heating element will provide exceptional service performance and life.

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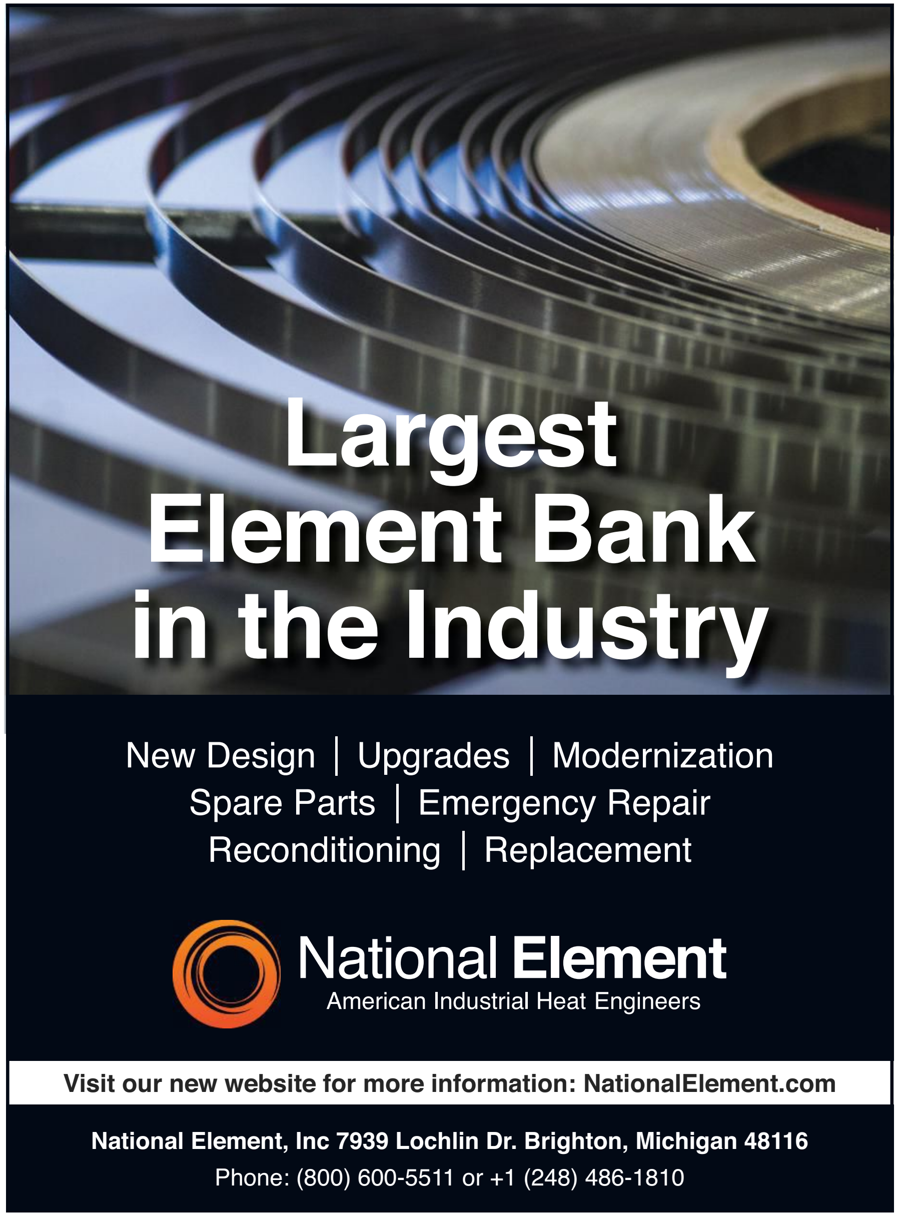

The Ubiquitous O-Ring

O-rings are an integral part of any successful vacuum system. Wherever detachable components (e.g., valves, pumps, etc.) are used, O-rings are necessary. But not all O-rings are created equal. Let's learn more.

The choice of an O-ring is dependent on two factors: the end-use application and the vacuum/pressure range over which it is intended to operate. A typical O-ring is an elastomer – a polymer material with the property of elasticity – having a Durometer "hardness" in the range of 65–80 Shore (i.e. about the hardness of an automotive tire tread or soft skateboard wheel).

The secret to their success is their ability to adapt to the unevenness of mating surfaces. The O-ring must be smooth, crack or scratch-free and properly lubricated. A vacuum system in which the construction materials are chosen carefully, welded or brazed joints are sound, static and dynamic seals are designed properly, and all materials and components are designed to withstand bake-out temperatures can operate with a leakage of less than 10^{-10} cubic centimeters/second.

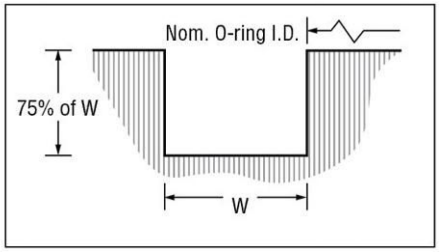

Fig. 1. O-ring groove dimensions for horizontal position

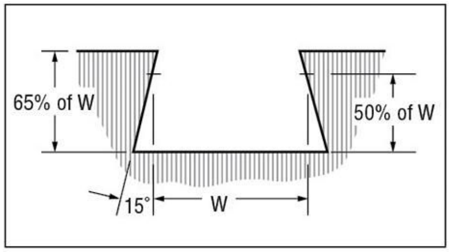

Fig. 2. O-ring groove dimensions for vertical position

Application Uses

In general, the vapor pressure of most organic materials is inversely proportional to the temperature. It is, therefore, very important that O-rings be kept cool, especially when the vacuum system is being baked out. The best of the materials must be held below 250˚F (120˚C), and for minimum outgassing an even lower temperature is desirable. In fact, if the temperature is held below 65˚F (18˚C), it is possible to use carefully constructed O-ring joints at pressure down to as low as 10^{-9} Torr.

In most applications, O-rings are intended to be static seals. A static seal is accomplished by plastically deforming an elastic material (e.g., Buna-N, Viton® Neoprene®, silicone or Teflon®) into the non-uniform surface of a mating flange, thus reducing the

Table 1. Operating-temperature limits for O-ring sealing materials

Material	Pressure Limit psi (N/mm)	Maximum Usage Temperature °F (°C)
Neoprene	145 (1)	225 (105)
Buna N	145 (1)	250 (120)
Teflon	145 (1)	400 (200)
Viton	145 (1)	450 (230)
Indium	1015 (7)	212 (100)
Aluminum	4,350-29,000 (30-200)	392 (200)
Copper	21,750-87,000 (150-600)	842 (450)
Gold	14,500-72,500 (100-500)	1472 (800)

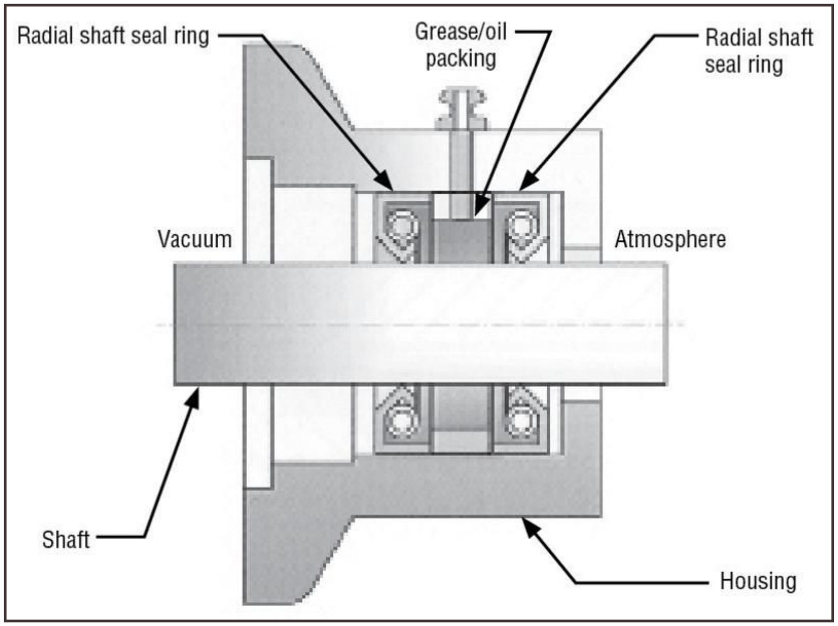

Fig. 3. Rotating shaft seal[1]

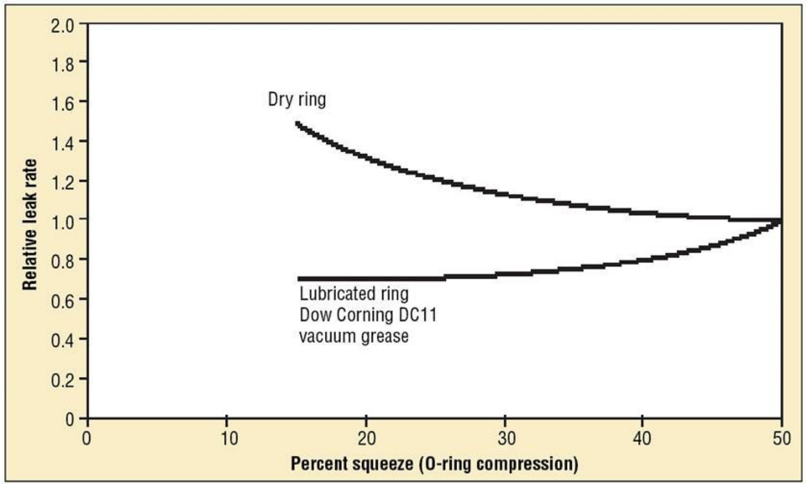

Fig. 4. Effect of lubrication as a function of O-ring compression on leak rate

leakage to an application-specific level. With proper precautions and tight tolerances, O-rings can be used in dynamic (moving) sealing applications as well.

In most cases, O-rings are placed in grooves and pressed between flanges, with one flat flange and one grooved flange being typical. The grooves must have close tolerances, a finish to at least 32 RMS and follow these rules:

- O-ring compression – the ratio of width to height – should be a maximum of 15-20% of the O-ring thicknesses of 0.196-0.393 inches (5-10 mm) and 30% for thicknesses under 0.125 inches (3 mm).
- The O-ring should fill 80-90% of the groove.
- For horizontal flanges, 25% compression on the O-ring is highly desirable (Fig. 1).
- For vertical flanges, an undercut groove is beneficial to hold the O-rings firmly in place (Fig. 2).

O-rings can be stretched up to about 5% of their length but no more than 10%.

Types of Seals

Flat seals should be avoided wherever possible because it is difficult to achieve the pressure required for the sealing material to cover all of the surfaces evenly.

Fig. 5. Typical O-ring failures[3]

Elastomer seals having a trapezoidal configuration are used for valve seals and for the covers and doors of large vacuum chambers. Deformation of the seals is typically kept within desired limits by attaching spacers in applications such as large chamber doors where high surface loading is possible.

Radial shaft seals (Fig. 3), or cap seals, are used where sealing of a rotating member is required. Care should be taken to ensure that only shaft sealing rings with a fully rubber-coated metal ring are used. With the shaft in motion, the leak rate will be significantly higher. Cap seals should only be used for manually rotated feed-through components.

Metal seals perform better in high-temperature applications (Table 1). These are made of materials such as copper, copper-nickel, aluminum, indium and even silver and gold (usually in the form of wire seals). Care must be taken to ensure that specific contact forces are maintained.

Common Fallacies

Here are some of the most common misconceptions when working with O-rings:

1. One of the most common beliefs is that the vacuum grease applied to the O-ring surface is responsible for actual sealing and that the more grease present, the better (and longer) the seal will last. In reality, the layer of vacuum grease is intended only to act as a lubricant to seat properly under the applied compression forces (Fig. 4) and should be a very thin layer, so much so that when you move your finger along the O-ring surface it will slide or glide unhindered with no appreciable amount of grease buildup.
2. O-rings do not need to be re-greased after every run. An O-ring should be wiped down using a clean rubber glove. Running your fingers over the surface of the O-ring is an excellent way to detect minute particles of dirt or grit and reveal nicks or areas that should be cleaned and re-greased. Thoroughly

cleaning the surface of the O-ring with alcohol, methyl-ethyl-keytone (MEK) or acetone before re-greasing (or applying vacuum grease initially) is critical.

3. The shelf life of a typical O-ring is forever. In reality, O-rings have a shelf life of only about six months.
4. O-rings cannot be spliced. Yes, they can as long as the part line is in the plane of the cross section. They must be joined together by gluing the material with a proper adhesive.

O-ring Failures[3]

O-ring seals often fail prematurely in applications because of either improper design or material selection. From the end-user's point of view, a seal can fail in three general ways:

- Leaking
- Contamination
- Change in appearance

Contributing factors are pressure-/vacuum-induced stress and thermally induced stress. Elevated temperatures may cause seal degradation, swelling or outgassing. Pressure or vacuum environments (or altering between the two) can cause outgassing and weight loss.

O-ring failures can be classified into the following general categories:

1. Abrasion (Fig. 5a) – The seal or parts of the seal exhibit a flat surface parallel to the direction or motion. Loose particles and scrapes are often found on the seal surface. Contributing factors include rough sealing surfaces, excessive temperature, process environment containing abrasive particles, dynamic motion and poor elastomer surface finish.
2. Flattening or over-compression (Fig. 5b) – The seal exhibits a flat-sided cross section with the flat sides corresponding to the mating seal surfaces. Contributing factors include excessive compression, excessive temperature, incompletely cured elastomer, elastomers with high compression set and excessive volume swell.
3. Degradation – The seal exhibits blisters, cracks, pits, voids or pockmarks on its surface. Absorption of gas occurs at high pressure and the subsequent rapid decrease in pressure. The absorbed gas blisters and ruptures the elastomer surface as the pressure is rapidly removed. Contributing factors include rapid pressure changes, low-modulus/hardness elastomers and incompatibility with the pressure/vacuum or thermal environment.
4. Extrusion (Fig. 5c) – The seal develops ragged edges (generally on the low-pressure side) that

appear tattered. Contributing factors include excessive clearances, excessive pressure, low-modulus/hardness elastomers, excessive gland fill, irregular clearance gaps, sharp gland edges and improper sizing.

5. Installation damage (Fig. 5d) – The seal or parts of the seal may exhibit small cuts, nicks or gashes. Contributing factors include poor techniques, improper tools, sharp edges on glands or components, improper sizing of elastomer, low-modulus/hardness elastomer and elastomer surface contamination.
6. Over compression (Fig. 5e) – The seal exhibits parallel flat surfaces (corresponding to the contact areas) and may develop circumferential splits within the flattened surfaces. Contributing factor includes improper design (failure to account for thermal volume changes or excessive compression).
7. Thermal Degradation – The seal may exhibit radial cracks located on the highest temperature surfaces. In addition, certain elastomers may exhibit signs of softening (a shiny surface) as a result of excessive temperatures. Contributing factors include elastomer thermal properties, excessive temperature excursions or cycling.
8. Plasma Degradation (Fig. 5f) – The seal often exhibits discoloration, as well as powdered residue on the surface and possible erosion of elastomer in the exposed areas. Contributing factors include chemical reactivity of the plasma, ion bombardment (sputtering), electron bombardment (heating), improper gland design and incompatible seal material.
9. Other – Spiral failure in which the seal exhibits cuts or marks that spiral around the circumference. Contributing factors include difficult or tight installation (static), slow reciprocating speed, low-modulus/hardness elastomer, irregular O-ring surface finish (including excessive parting line), excessive gland width, irregular or rough gland surface finish and inadequate lubrication.

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The Molecule Counters Part One: Vacuum Gauges

Counting molecules is a job for vacuum gauges. Depending on the type of vacuum systems and the required operating vacuum level, different vacuum gauges are required, often in combination with one another, to accurately determine and/or control the vacuum level of the chamber at any given moment in time. Let's learn more.

What is a Vacuum Gauge?

A vacuum gauge is an instrument for measuring pressures below atmospheric pressure. There are many types, each designed for a specific function. Some of the more common types of vacuum gauges are shown below, listed in order of descending pressure range:

Manometer – A relatively simple device that usually consists of a tube or column filled with a liquid. The pressure is found by measuring the column height or the difference in heights of several columns.

Thermal conductivity gauge – These devices operate on the principle that heat transported by gas molecules can be related to gas pressure. A heat source causes changes in surface temperature (or in the heating power required to maintain constant temperature), and this is related to the pressure of the system. Various types of thermal-conductivity gauges are distinguished according to the method of indicating the surface temperature (that is, the way in which the wire temperature is measured). The most common types are Pirani gauges (typical range: 1 Torr to 10^{-5} Torr) and thermocouple gauges (typical range: 1 Torr to 10^{-3} Torr).

Knudsen (radiometer) gauge (typically accurate to 10^{-6} Torr) – These devices measure pressure in terms of the net rate of transfer of momentum by molecules between two surfaces maintained at different temperatures and separated by a distance smaller than the mean free path of the gas molecule.

McLeod gauge (typically accurate to 10^{-6} Torr) – The principle of operation involves measuring the pressure of a gas by measuring its volume twice, once at the unknown low pressure and again at a higher reference pressure.

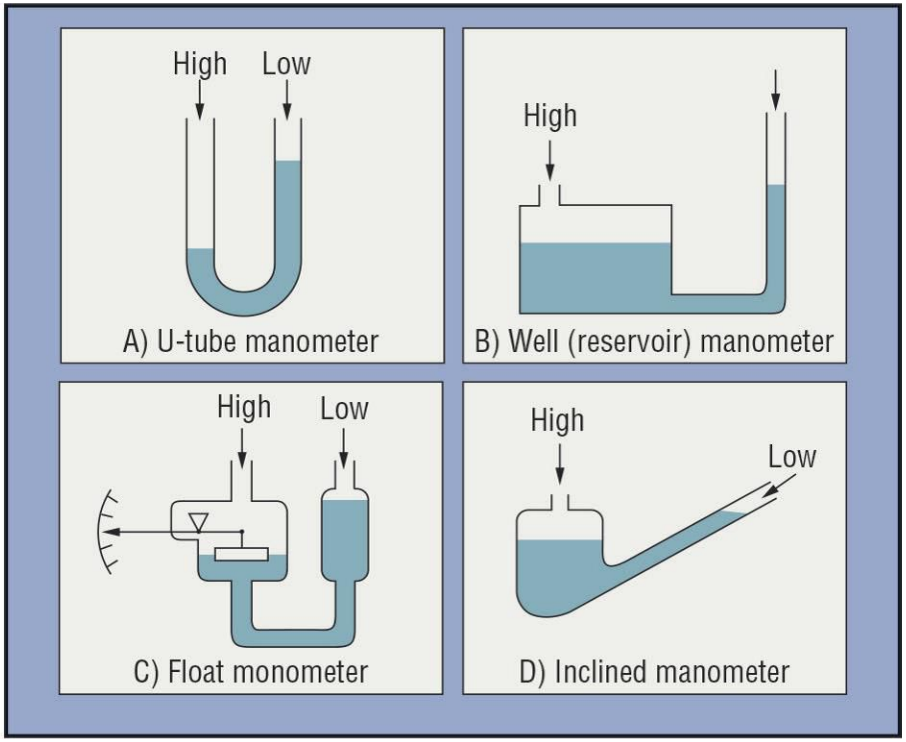

Fig. 1. Types of manometers[1]

Ionization gauge (typically accurate to 10^{-9} Torr and beyond) – These devices have a means of ionizing the gas molecules and a means of correlating the number and type of ions produced with the pressure of the gas. Various types of ionization gauges are distinguished according to the method of producing the ionization. The common types are hot-cathode gauge (typical range: 1×10^{-2} to 1×10^{-10} Torr) and cold-cathode gauge (typical range: 1×10^{-2} to 1×10^{-11} Torr).

A Little History

Engineers first became interested in vacuum measurements in the 1600s, when they noted the inability of pumps to raise water more than about 30 feet (9 m). The Duke of Tuscany in Italy commissioned Galileo to investigate the "problem." Galileo, among others, devised a number of experiments to investigate the properties of air. After Galileo's death in 1642, Evangelista Torricelli continued the work that included vacuum-related investigations and the invention of the mercury barometer. He discovered that the atmosphere exerts a force of 14.7 psi (101.3 kPa) and that, inside a fully evacuated tube, the pressure was enough to raise a column of mercury to a height of 29.9 inches (760 mm). The height of a column of mercury is therefore a direct measure of the atmospheric pressure. The value of 1/760th of an atmosphere is called a Torr, in honor of Torricelli. Today, manometers (Fig. 1) come in a variety of forms to suit particular application needs.

Pressure is simply defined as a force per unit area, and the most accurate way to measure air pressure is to balance a column of liquid of known

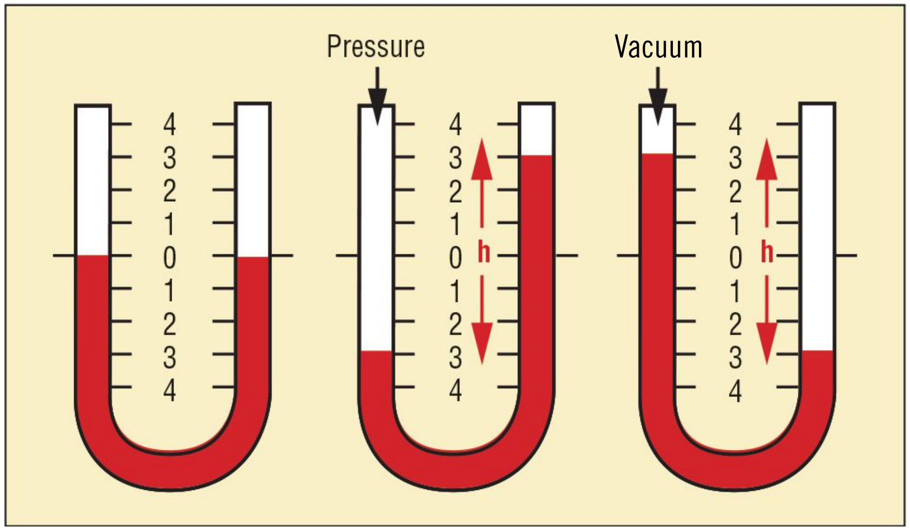

Fig. 2. Principles of the manometer[2]

weight against it and measure the height of the liquid column so balanced. The units of measure commonly used in the U.S. are inches of mercury (Hg), using mercury as the fluid, and inches of water column (W.C.), using water or oil as the fluid.

The U-tube manometer (Fig. 1A) is the most common type of manometer today because the difference in height between the two columns is always a true indication of the pressure regardless of variations in the internal diameter of the tube. With both ends of the tube open, the liquid is at the same height in each leg. When positive pressure is applied to one leg (Fig. 2), the liquid is forced down in that leg and up in the other. The difference in height (h), which is the sum of the readings above and below zero, indicates the pressure. When a vacuum is applied to one leg (Fig. 2), the liquid rises in that leg and falls in the other. The difference in height, "h," which is the sum of the readings above and below zero, indicates the amount (or degree) of vacuum.

Pressure Ranges

The correct choice of gauge depends on knowledge of the working principles of the gauge, the range of pressures it can measure and its accuracy over the required range. These factors have been determined by experience (Fig. 3), and there is a vacuum gauge for every pressure range.

For low vacuum ranges (higher pressures) between atmospheric and 10 Torr, Bourdon tubes, bellows, active strain gauges and capacitance sensors are all suitable measurement devices.

For mid-range vacuum requirements – those in the 10^{-2} to 10^{-3} Torr range – there are several choices including the capacitance manometer, a good choice for more accurate measurements, and the hot cathode ion gauge.

For intermediate vacuum applications (between 10^{-2} and 10^{-4} Torr), capacitance manometers are

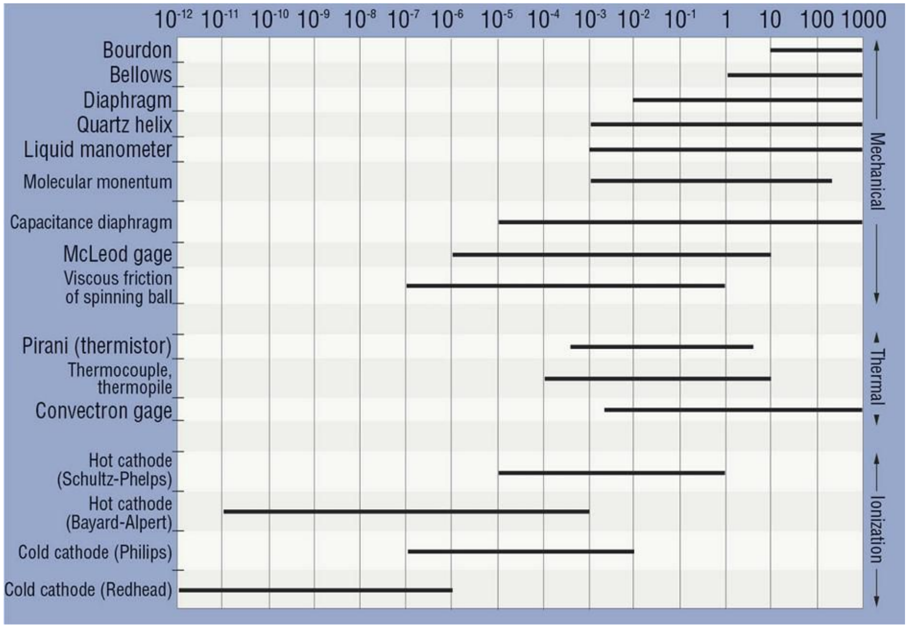

Fig. 3. Vacuum gauge measurement ranges[1]

the best in terms of performance but are also the most expensive. The lowest-priced gauge is the thermocouple type, but its error is the greatest. Digital Pirani gauges represent a good compromise solution with accuracy between that of capacitance and thermocouple sensors.

For ultrahigh-vacuum service ($\geq 10^{-6}$ Torr), either cold cathode or Bayard-Alpert hot cathode gauges are used. There is some concern over accuracy and/or stability, and both require frequent calibration.

Dictionary of Vacuum Gauge Terms[5, 6]

Absolute pressure – Pressure measured above the zero value of a perfect vacuum designated psia (pounds per square inch absolute).

Atmospheric pressure – The pressure exerted by a mercury column 760 mm high at 0°C under a standard acceleration of gravity (980.665 cm/sec^2) – 14.7 psi at sea level.

Boyle's Law – One of the gas laws, Boyle's law states that pressure and volume in a gas are inversely proportional (assuming constant temperature and mass).

Gauge pressure – Pressure measured at atmospheric pressure as a reference point. Gauge pressure is designated psig (pounds per square inch gauge).

Millimeter of mercury (mm Hg) – A unit of pressure defined as that pressure that will support a column of mercury one millimeter high.

Pressure – The force per unit area a gas exerts. Common units are Torr, millibar, microns, psia or millimeters of mercury.

Torr – A unit of pressure defined as 1/760th of an atmosphere.

Vacuum – A space filled with gas at a pressure less than atmospheric pressure. Vacuums are classified as rough (760 Torr to 1 Torr), low (1 Torr to 10^{-3} Torr), high (10^{-3} to 10^{-6} Torr), very high (10^{-6} to 10^{-9} Torr) and ultrahigh (10^{-9} Torr and above).

Part Two will talk about the types of vacuum gauges in more detail.

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The Molecule Counters Part Two: More About Vacuum Gauges

Four, five, six…

Counting molecules is a job for vacuum gauges, and it's now time to understand the differences between these devices and when to use them. Let's learn more.

Recall first that the vacuum level in a vessel is determined by the pressure differential between the evacuated volume and the surrounding atmosphere (Table 1). The two basic reference points in all these measurements are standard atmospheric pressure (760 Torr) and perfect vacuum (0 Torr), so calculating changes in volume in vacuum systems requires conversions to negative pressure (psig) or absolute pressure (psia).

Mechanical Gauge Designs

Mechanical gauges measure pressure or vacuum by making use of the mechanical deformation of tubes or diaphragms when exposed to a difference in pressure. For this reason, they are classified as differential pressure gauges. Typically, one side of the element is exposed to a reference vacuum, and the instrument measures the mechanical deformation that occurs when an unknown vacuum pressure is exposed to the other side.

Table 1. Comparison of vacuum and pressure levels

Percent of absolute vacuum	Inches of mercury (mm Hg)	Pressure psig (mbar)
10	-3.0 (-76.2)	-1.47 (-101.3)
15	-4.5 (-114.3)	-2.21 (-152.4)
20	-6.0 (-152.4)	-2.95 (-202.7)
25	-7.5 (-190.5)	-3.68 (-253.7)
30	-9.0 (-228.6)	-4.42 (-304.1)
35	-10.5 (-266.7)	-5.15 (-355.1)
40	-12.0 (-304.8)	-5.89 (-405.4)
45	-13.5 (-342.9)	-6.63 (-456.4)
50	-15.0 (-381.0)	-7.36 (-507.4)
55	-16.5 (-419.1)	-8.10 (-558.5)
60	-18.0 (-457.2)	-8.84 (-609.5)
65	-19.5 (-495.3)	-9.57 (-659.8)
70	-21.0 (-533.4)	-10.31 (-710.8)
75	-22.5 (-571.5)	-11.05 (-761.9)
80	-24.0 (-609.6)	-11.78 (-812.2)
85	-25.5 (-647.7)	-12.52 (-863.2)
90	-27.0 (-685.8)	-13.26 (-914.2)
95	-28.5 (-723.9)	-13.99 (-964.6)
100	-30.0 (-762.0)	-14.70 (-1013.5)

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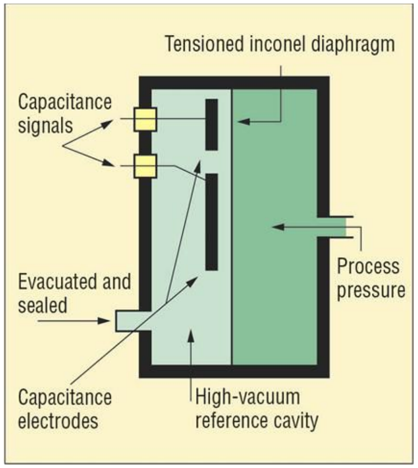

Fig. 1. Principle of operation of a capacitance vacuum manometer[1]

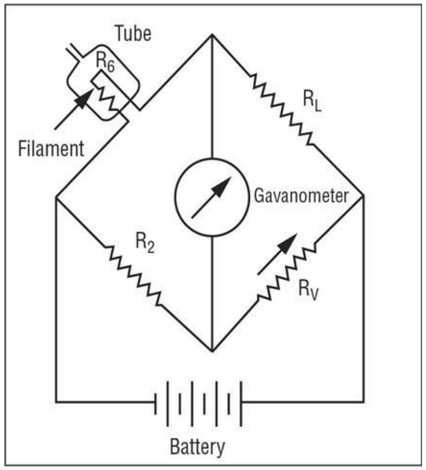

Fig. 2. Pirani gauge Wheatstone-bridge network[5]

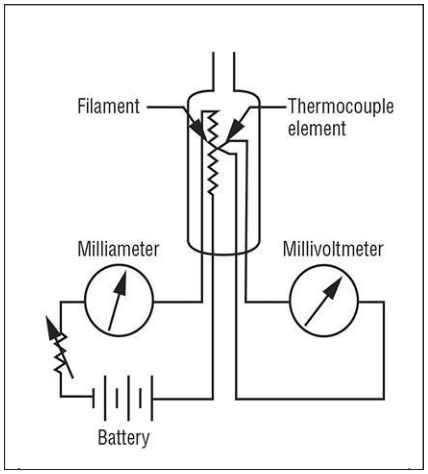

Fig. 3. Thermocouple gauge[5]

Fig. 4. Typical thermocouple gauge[3]

Bourdon Gauges

These gauges work on the principle of the Bourdon tube, which consists of a tube with an elliptical cross section formed in an arc. The tube is rigidly fixed at one end and closed at the other end. When the pressure in the tube increases, the radius of the arc increases (in other words, the tube tries to straighten itself out). When the pressure decreases, the radius decreases (thus, the free end of the tube moves in response to a change in pressure). A system of mechanical linkages attached to the free end moves a pointer over a calibrated scale.

A quartz Bourdon tube uses a quartz helix element, and instead of moving linkages, the deformation rotates a mirror. When used for vacuum detection, two quartz Bourdon elements are formed into a helix. The reference side contains a sealed vacuum and the measurement side is connected to the unknown process vacuum. The pressure difference between the two sides causes an angular deflection that is detected optically. The optical readout has a high resolution, about one part in 100,000. Advantages of this sensor are its precision and the corrosion resistance of quartz. Its main limitation is high price.

Manometer

The basic manometer consists of a reservoir filled with a liquid. When detecting vacuums, the top of the

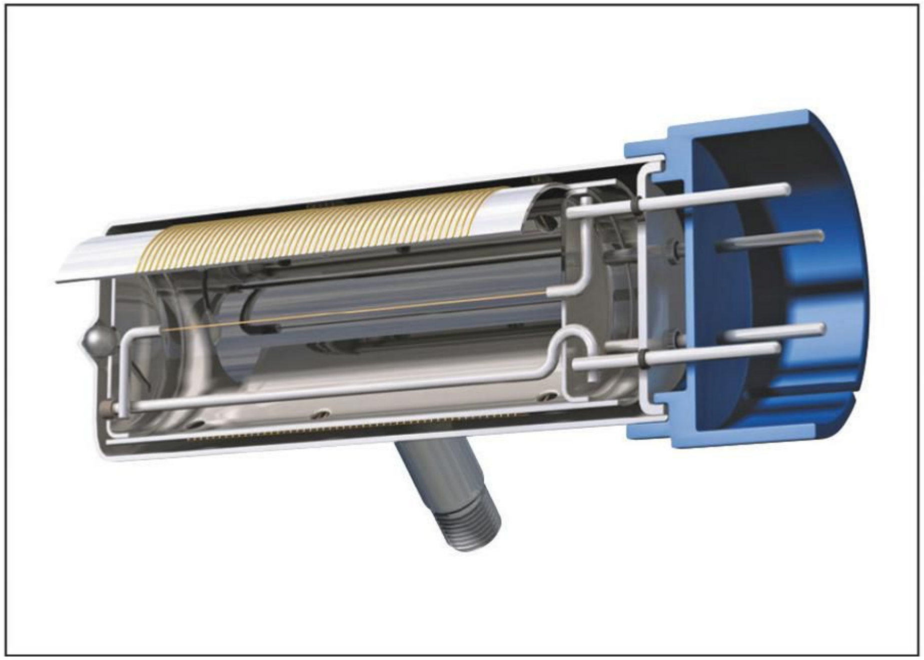

Fig. 5. Typical conduction gauge[2] Courtesy of Brooks Automation, Inc.

column is evacuated and sealed. A relatively small change in vacuum pressure will cause a relatively large movement of the liquid. Manometers are simple, low cost and can detect vacuums down to about 10^{-3} Torr. The accuracy of the gauge is determined by how closely the difference in height of the two arms can be measured. Today, digital-readout manometers have greatly improved accuracy. The sensitivity of the gauge depends on the density of the fluid used.

Capacitance Vacuum Manometer

A capacitance sensor operates by measuring the change in electrical capacitance that results from the movement of a sensing diaphragm relative to some fixed capacitance electrodes (Fig 1). The higher the process vacuum, the farther it will pull the measuring diaphragm away from the fixed capacitance plates. In some designs, the diaphragm is also allowed to move. In others, a variable dc voltage is applied to keep the sensor’s Wheatstone bridge in a balanced condition.

Fig. 6. Typical conduction gauge[3]

The amount of voltage required is directly related to the pressure.

The great advantage of a capacitance gauge is its ability to detect extremely small diaphragm movements. Accuracy is typically 0.25 to 0.5% of the reading. Thin diaphragms can measure down to 10^{-5} Torr, while thicker diaphragms can measure in the low vacuum to atmospheric range. To cover a wide vacuum range, one can connect two or more capacitance sensing heads into a multi-range package.

McLeod Gauges

The McLeod gauge uses the principle of Boyle's law ($P_1V_1 = P_2V_2$; that is, if the temperature is held constant, the increase in pressure is exactly proportional to the decrease in volume) to amplify and measure pressures that are too small to be measured with a manometer. To do this, a sample of gas from the system is isolated and reduced in volume by a known amount. This reduction in volume causes a proportional increase in the pressure of the gas. Originally invented in 1878, the McLeod gauge is still used today, mainly for calibrating other gauges (although other techniques are making this obsolete). McLeod gauges can cover vacuum ranges between 1 and 10^{-6} Torr.

Molecular Momentum Gauges

This vacuum gauge is operated with a rotor that spins at a constant speed. Gas molecules in the process sample come in contact with the rotor and are propelled into the restrained cylinder. The force of impact drives the cylinder to a distance proportional to the energy transferred, which is a measure of the number of gas molecules in that space. The full scale of the instrument depends on the gas being measured. The detector has to be calibrated for each application.

Viscous Friction Gauges

At high-vacuum levels, viscosity and friction both depend on pressure. This instrument measures vacuum down to 10^{-7} Torr by detecting the deceleration caused by molecular friction on a ball that is spinning in a magnetic field. Vacuum is determined by measuring the length of time it takes for the ball to drop from 425 to 405 revolutions per second after drive power is turned off. The higher the vacuum, the lower the friction and, therefore, the more time it will take. This design is accurate to 1.5% of indicated reading, is resistant to corrosion and can operate at temperatures up to 7500˚F (4150˚C).

Thermal Gauge Designs

Below 1 Torr, a change in the pressure of a gas will cause a change in its thermal conductivity (the ability of a gas to conduct heat). If an element heated by a constant power source is placed in a gas, the resulting surface temperature of the element will be a function of the surrounding vacuum. Because the sensor is an electrically heated wire, thermal vacuum sensors are often called hot-wire gauges. Because the characteristics of all gases are different, the response of a thermal-conductivity gauge will vary for each gas. To read accurately, the gauge must be calibrated for the gas being measured.

Fig. 7. Hot-cathode gauge[3]

Fig. 8. Bayard-Alpert ionization gauge[3]

Pirani Gauges

The Pirani gauge utilizes the change in electrical resistance of a wire with temperature. A sensor wire is heated electrically, and the pressure of the gas is determined by measuring the current needed to keep the wire at a constant temperature by use of a Wheatstone bridge network (Fig. 2). The Pirani gauge is linear in the 10^{-2} to 10^{-4} Torr range. Above these pressures, output is roughly logarithmic. Pirani gauges are inexpensive, convenient and reasonably accurate (within 2% at the calibration point and 10% over their operating range).

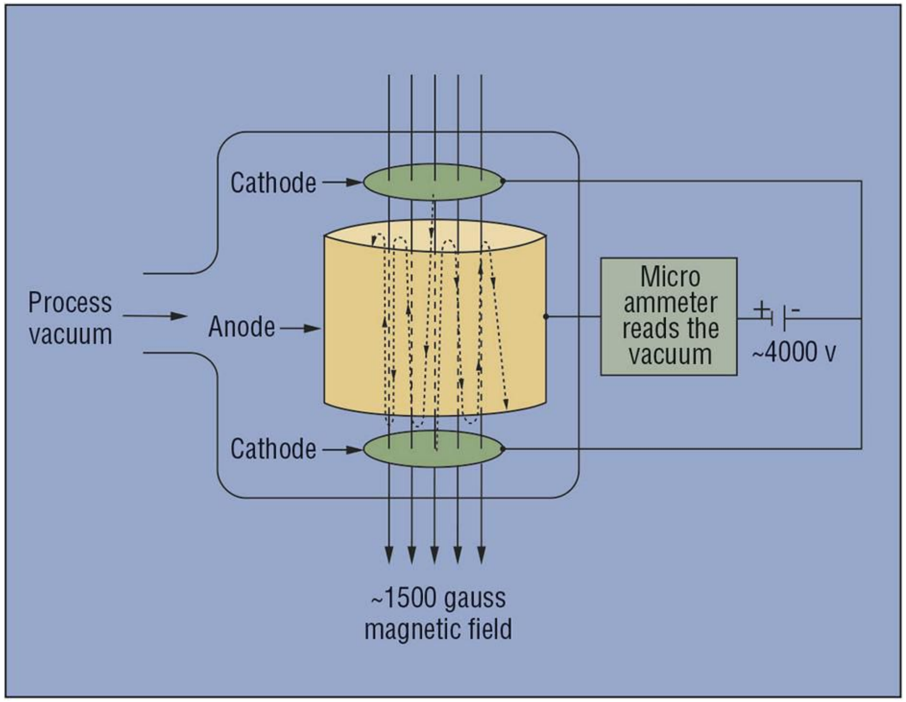

Fig. 9. Cold-cathode gauge[1]

Thermocouple Gauges

The thermocouple gauge relates the temperature of a filament in the process gas to its vacuum pressure (Fig. 3, 4). The filament is heated by a constant current of 20-200 mA dc, and the thermocouple generates an output of about 20 mV dc. The heater-wire temperature increases as pressure is reduced.

Typical thermocouple gauges measure 10^{-3} to 2 Torr. This range can be increased by use of a gauge controller with a digital/analog converter and digital processing. Using an industry-standard thermocouple sensor, such a gauge controller can extend the range of a thermocouple sensor to cover from 10^{-3} to 1,000 Torr, thereby giving it the same range as a convection-type Pirani gauge but at a lower price.

Convection Gauges

Similar to the Pirani gauge, this sensor uses a temperature-compensated, gold-plated tungsten wire to detect the cooling effects of both conduction and convection and thereby extends the sensing range (Fig. 5). At higher vacuum, response depends on the thermal conductivity of the gas, while at lower vacuums it depends on convective cooling by the gas molecules. Measurement range is from 10^{-3} to 1,000 Torr. With the exception of its expanded range, features and limitations of this sensor are the same as those of Pirani and most thermocouple gauges.

The convection gauge (Fig. 6) measures absolute pressures by determining the heat loss from a fine wire filament maintained at a constant temperature. The response of the sensor depends on the gas type. A pair of thermocouples is mounted at a fixed distance from each other. One thermocouple is heated to a constant temperature by a variable-current power supply.

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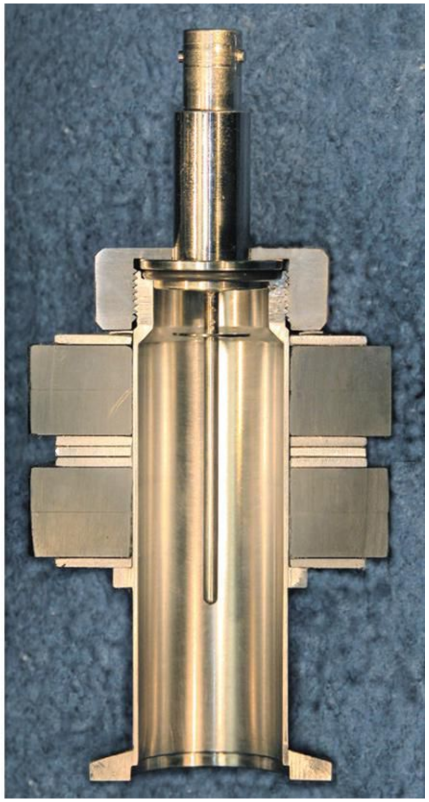

Fig. 10. (left) Cold-cathode gauge[3] Fig. 11. (right) Cross-sectional view of a cold-cathode gauge[3]

Power is pulsed, and the temperature is measured between heating pulses. The second thermocouple measures convection effects and also compensates for ambient temperature. This sensor must be mounted vertically.

Combined Gauges

To get around the range limitations of certain sensors, gauge manufacturers have devised a means for electronically linking multiple sensor heads. For example, one manufacturer offers a wide-range vacuum gauge that incorporates two pressure sensors in one housing: a fast-response diaphragm manometer for measurements between 1,500 Torr and 2 Torr and a Pirani gauge for measuring between 2 Torr and 10^{-3} Torr. The gauge controller automatically switches between the two sensors.

Ionization Gauge Designs

When fast-moving electrons pass through a gas, they can knock some of the outer electrons off of the gas molecules. The remaining part of the molecule (called an ion) then has a positive charge. The process is called ionization by bombardment. For a constant current of electrons at a given gas velocity, the rate at which these positive ions are formed is proportional to the concentration of the gas molecules (assuming the temperature remains constant). The ionization efficiency varies with the kind of gas, so these gauges must be calibrated for the gas for which they will be used. Two types are available: hot cathode and cold cathode.

Refined by Bayard-Alpert in 1950, the hot filament in the hot-cathode gauge emits electrons into the vacuum, where they collide with gas molecules to create ions (Figs. 7, 8). These positively charged ions are accelerated toward a collector, where they create a current in a conventional ion-gauge detector circuit. The amount of current formed is proportional to the gas density or pressure. Most hot-cathode sensors measure vacuum in the range of 10^{-2} to 10^{-10} Torr.

Newer instruments extend this range significantly by using a modulated electron beam, synchronously detected to give two values for ion current. At pressures below 10^{-3} Torr, there is little difference in the two values. At higher pressures, the ratio between the two readings increases monotonically, allowing the gauge to measure vacuums up to 1 torr.

Because most high-vacuum systems were made of glass in 1950, it made sense to enclose the electrode structure in glass. Today, however, a modern vacuum system may be made entirely of metal. One argument in favor of this is that glass decomposes during routine degassing, producing spurious sodium ions and other forms of contamination. Nevertheless, glass gauges remain the most popular choice for hot-cathode sensors.

Cold-Cathode Gauge

This gauge (also called a Philips gauge or Penning gauge) is based on the glow discharge that occurs in gas at low pressures in the presence of a magnetic field. Electrons that originate in one of the cathodes in the gauge do not go directly to the anode (because of the magnetic field). Instead, they travel back and forth in helical paths between the cathodes multiple times before striking the anode. The increased path length provides a high probability of ionization (even at low gas pressures where a glow discharge normally does not occur). The total discharge current (negative and positive ions) is used to measure the pressure.

The major difference between hot- and cold-cathode sensors is in their methods of electron production. In a cold-cathode device, electrons are drawn from the electrode surface by a high potential field. In the Phillips design (Fig. 9 - 11), a magnetic field around the tube deflects the electrons, causing them to spiral as they move across the magnetic field to the anode. This spiraling increases the opportunity for them to encounter and ionize the molecules. Typical measuring range is from 10^{-10} to 10^{-2} Torr. The main advantages of cold-cathode devices are that there are no filaments to burn out, they are unaffected by the inrush of air, and they are relatively insensitive to vibration.

Summary

There you have it – everything you wanted to know about vacuum gauges. The secret to success is having accuracy and repeatability so as to ensure that the process being run stays under control.

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How to Move Molecules in Vacuum Systems Part One: Mechanical and Booster Pumps

In order to create a vacuum within a closed container, or vessel, we need to remove the molecules of air and other gases that reside inside by means of a pump. The vacuum vessel (Fig. 1) and pumps (mechanical, booster, diffusion, holding) together with the associated piping manifolds valves, gauges and traps comprise a typical vacuum system. Let's learn more.

Mechanical Pumps

To reach the various vacuum levels, different vacuum pumping systems are required. The foundation of any of these systems is the positive displacement mechanical, or roughing, pump. The roughing pump – so called because it is used to produce a "rough" vacuum – is used in the initial pumpdown from atmospheric pressure to around 2 x 10^{-2} Torr, depending on the type of pump.

The internal components of the mechanical pump (Fig.2) help us understand its operation. Basically, it is an eccentric cylinder driven about an axis by an electric motor. During operation, the rotor turns with the shaft, which causes the piston to sweep the volume between it and the stator. The piston does not turn in this case, but the vane-like extension on the piston (called the slide, or slide valve) moves up and down in an oscillating seal (called the slide pin or slide-valve pin).

At the start of a rotation, the ported slide valve is open. As the rotation occurs, the slide valve closes, trapping a given volume of gas. This volume is compressed as the revolution continues. Near the end of the revolution, the pressure is above atmospheric, and the gas discharges through a spring-loaded poppet valve. On the completion of the revolution, the slide valve opens, and another increment of gas is admitted.

A vacuum pump will remove a number of molecules with each rotation. How many molecules will depend largely on the actual pump displacement, rotational speed and vacuum-system pressure. Each time molecules are removed, the remaining molecules spread out in the vacuum chamber to occupy the available volume. This repeats (molecules are removed by the pump) the pressure reduces and there are less and less molecules to expand into the pump inlet with each rotation.

Mechanical pumps can be single or dual stage. A single-stage design will achieve a pressure of about 1 x10^{-2} Torr, while a dual-stage pump is capable of reaching pressures around 1 x 10^{-3} Torr. A two-

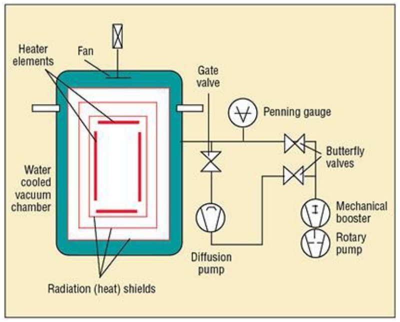

Fig. 1. Typical vacuum system[1]

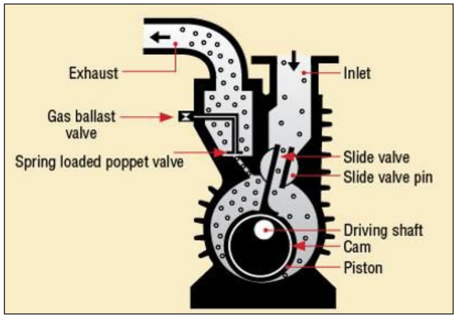

Fig. 2. Mechanical pump operation[4]

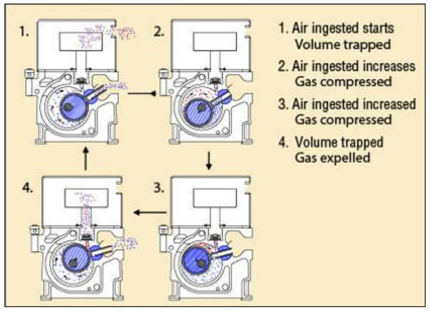

Fig. 3. Booster pump operation[1]

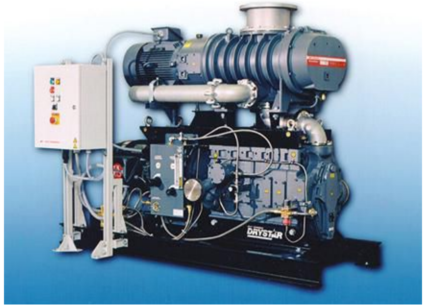

Fig. 4. Typical mechanical pump and booster combination (Courtesy of Edwards Vacuum)

stage, or compound, pump has two pumping chambers connected in series. The exhaust of the first stage is coupled to the inlet of the second stage.

Lower pressure, less molecules and more speed in the same volume results in less pumping efficiency. Why mechanical pumps start off with high efficiency and fall of at these pressure ranges can be explained as follows. Consider one cubic foot of volume at atmospheric pressure (760 Torr). If we were to put this volume of gas in a container that was twice as large, the pressure would be exactly half, or 380 Torr. If we double the volume, we halve the pressure. Thus, doubling the volume again to 4 cubic feet results in a pressure of 190 Torr. So, to evacuate a chamber to 1 x 10^{-3} Torr theoretically requires that we remove a volume of 760,000 cubic feet. In everyday operation, a mechanical roughing pump will have great difficulty achieving this ultimate pressure (lowest attainable pressure) since its efficiency begins to fall off at 1 x10^{-1} to 8 x 10^{-2} Torr.

An alternative to "wet" mechanical pumps – those that use mechanical pump oil – are the so-called "dry" mechanical pumps. These pumps are used in applications where pumping efficiency and process contamination concerns are important issues. They have positive environmental impact due to reduced oil consumption and minimal disposal issues, and they operate with less noise and vibration.

Dry pumps operate on the compressor principle. As the two rotors rotate, gas is drawn in through an inlet slot aligned with the cavity in one of the rotors. Further rotation closes the inlet while the lobes, or claws, compress the trapped volume of gas until the cavity in the second rotor exposes the outlet or exhaust slot. A small volume of gas remains trapped and is carried over into the next pumping cycle. These designs produce high compression ratios and operate at high efficiency.

Booster Pumps

Enter the booster pump, or blower, a different type of mechanical pump that is placed in series with the roughing pump. It is designed to "cut in" at around 700 Torr and provide higher speeds in the pressure range of 100 Torr to 1 x 10^{-3} Torr. In this intermediate pressure range, the roughing pump is losing efficiency while the diffusion (vapor) pump is just starting to gain efficiency.

The operation of the booster (Fig.3) is as follows. Two impellers are mounted on parallel shafts and rotate in opposite directions. They are geared together so that the correct relative position of each impeller to the other can be maintained. The impellers do not touch each other, and no sealing fluid is used. Any back leakage is small compared to the total speed of the pump in its useful range.

During operation, gas from the inlet side is trapped between the impeller and housing. No compression takes place as this gas is moved from the inlet to the discharge port. When the leading lobe of the impeller passes the discharge port, gas from the discharge area (at higher pressure) enters, but it is swept away by the trailing lobe.

Mechanical booster pumps have a useful compression ratio of 10:1. Therefore, they must be backed by a mechanical roughing pump in order to reach their maximum efficiency (Fig. 4). The mechanical booster pump is highly efficient in reducing the time required to evacuate a large or "gassy" system to the operating pressure at which the diffusion pump is efficient.

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How to Move Molecules in Vacuum Systems Part Two: Diffusion Pumps and Troubleshooting Tips

Vacuum pumps have been called the heart of a vacuum system. Let's look at how we can reach low vacuum levels using diffusion pumps. And we need to know how all pumps should be maintained to keep the vacuum system running trouble free. Let's learn more.

Diffusion Pumps

The diffusion pump (Fig. 1) is a type of vapor pump (since it pumps vapors), and it is used to help achieve even lower system pressures. The diffusion pump is capable of pumping gas with full efficiency at inlet pressures not exceeding 2x10^{-2} Torr and discharge (or foreline) pressures not exceeding 5x10^{-1} Torr. The diffusion pump cannot operate independently. It requires a separate pump to reduce the chamber pressure to or below the diffusion pump's maximum intake pressure before it will operate. Also, while operating, a separate or holding pump is required to maintain the discharge pressure below the maximum tolerable pressure.

The operation of the diffusion pump is as follows. The inlet of the pump is attached directly to the vessel, and a mechanical pump is attached to the outlet. The pressure of the entire system is reduced to about 5x10^{-2} Torr. At this point the diffusion-pump heater is turned on, heating a fluid in the boiler portion of the pump. The rise in pressure forces the vapors up the chimney of the pump, where it is directed out spray nozzles into the surrounding area of lower pressure. The nozzles deflect the vapor as a jet downward and outward to the walls (where the vapor condenses).

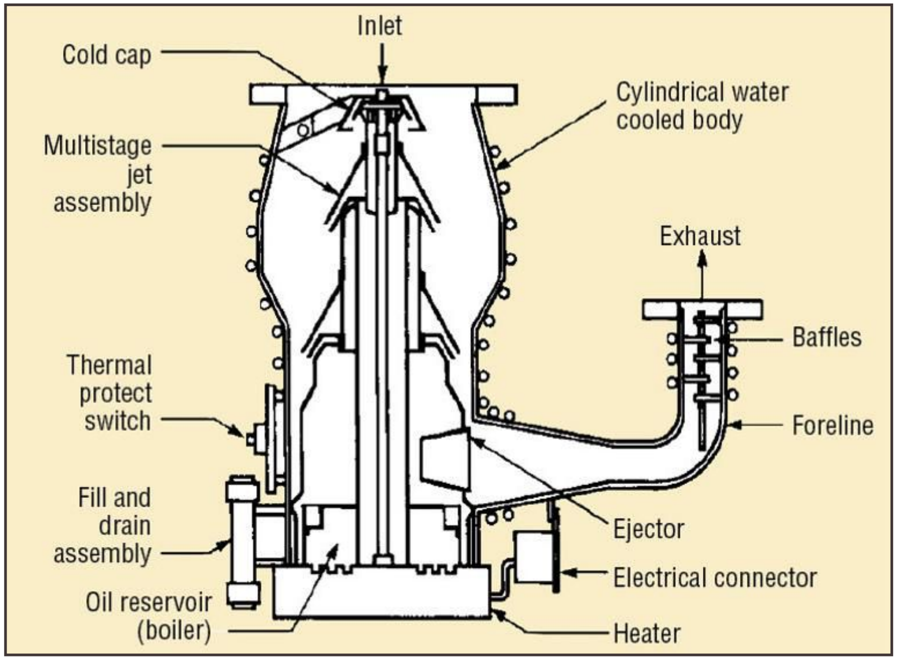

Fig. 1. Diffusion pump operation [2]

Table 1. Typical properties of certain fluids used in diffusion pumps (ultra-high-vacuum applications) [3]

Fluid Property	Polyphenyl Ether	Silicone	Hydrocarbon
Vapor Pressure, Torr (25°C)	4×10^{-10}	2×10^{-8}	5×10^{-6}
Molecular Weight	446	484	420
Density (25°C)	1.20	1.07	0.87
Flashpoint (°C)	288	221	243
Boiling Point (1.3 mbar, °C)	29	223	220
Viscosity, cSt (25°C)	1000	40	135
Viscosity, cSt (100°C)	12.0	4.3	7.0
Surface Tension (dynes/cm)	49.9	30.5	30.5
Refractive Index (25°C, 589 nm)	1.67	1.56	1.48
Thermal Stability	Excellent	Good	Poor
Oxidation Resistance	Excellent	Excellent	Poor/Fair
Chemical Resistance	Excellent	Good	Poor
Radiation Resistance	Excellent	Good	Fair

Gas molecules from the vessel enter the pump throat and diffuse through the less-dense fringe at the edge of the vapor stream. When a gas molecule has penetrated into the high-density core of the stream, the probability of its being knocked backward toward the inlet is less likely than the probability of its being carried along the vapor stream toward the outlet. Thus the predominant direction of molecular travel is away from the inlet and toward the outlet. In a multistage pump, the gas molecules are directed toward the next nozzle, where the action is repeated. Several succeeding stages will compress the low-pressure gas at the inlet to a higher pressure at the outlet, where it is removed to atmosphere by the mechanical pump.

The movement of molecules from an area of low pressure to an area of higher pressure will only continue as long as the region of higher pressure (or forepressure) does not exceed a critical limit. Consequently, it is necessary for a diffusion pump to be “backed” by a mechanical pump. In practice, the backing pump has two or three times the minimum capacity required.

Oils based on silicones, hydrocarbons, esters, perfluorals and polyphenyl ethers can be used as diffusion-pump fluids being vaporized in the range of 190 - 280˚C (375 - 535˚F). Each fluid has specific properties (Table 1). Mercury is no longer used in vacuum pumping systems due in large part to its toxicity. The choice of the pump fluid depends on the required application (vacuum level) of the pumping system.

Although diffusion pumps have been replaced in some applications by more advanced designs – cryogenic or turbomolecular pumps – they are still widely used due to their reliability, simple design and operation without noise or vibration. They are also relatively inexpensive to operate and maintain.

Evacuation Effects

In general, the effects of evacuating a vessel can be summarized as follows[4]:

A. The effects of evacuating a vessel from 760 Torr (atmospheric pressure) to 1 Torr are:
  1. Removing (high relative humidity) air
    a. Water vapor condensation (due to cooling effect associated with a sudden drop in pressure).
    b. “Fog” develops (a cloud “swirls” around with a turbulence that is characteristic of a gas flow at high pressure and high flow rate).
  2. (Slow) Change in the composition of the gas remaining
    a. Initially, air is the major component of the gas (certain other contaminants such as oils, grease and water exist on cold surfaces such as vessel walls).

   b. Eventually, almost all of the air is pumped out. The grease and water will continue to evaporate, and their partial pressure will constitute a much larger portion of the total pressure. This is called outgassing.

B. The effects of evacuating a vessel from 1 Torr to $1x10^{-4}$ Torr are:
  1. The ability of the gases remaining in the vessel to conduct heat begins to decrease rapidly.
  2. A change in the electrical characteristics of the gas begins (voltage to start a discharge decreases).

C. The effect of evacuation from $1x10^{-4}$ Torr to $1x10^{-6}$ Torr is:
  1. Decreasing molecular density
     a. Molecules collide with the sides of the vessel as often as they will with each other.
     b. There is an increase in sliding friction.

Pump Problems

The most common problems experienced with the various pumping systems can be summarized as:

- Contamination of the oil (mechanical pump)
- Gas leaking into the pump (mechanical pump)
- Solid particles (mechanical pump)
- Exposure of the hot pump fluid to the atmosphere (diffusion pump)
- Interruption of cooling fluid (diffusion pump)
- Power failure (diffusion pump)
- High forepressure (diffusion pump)

Troubleshooting Guide (Diagnosis of Problems)

Of the various mechanical-pump problems that can arise, contamination of the oil in the pump is the most common. Vapors present in the gas being pumped may condense and mix with the oil. Moisture (water vapor) especially, if not removed, will flash to vapor, tie up a large portion of the pump's capacity and create a significant loss in pumping efficiency, resulting in either extremely long pumpdown times, failure to achieve a low vacuum level or both. In addition, the oil may break down chemically, forming a sludge, which causes numerous short- and long-term problems with pump operation. In order to rid the oil of water and other liquid condensates, a gas ballast is used. A ballast valve on the pump can be opened – manually or automatically – to admit air (or another gas) into the pump, disrupting its operating efficiency. The result is a reduction in the compression necessary to exhaust the gases and, correspondingly, a decrease in the amount of vapor that condenses. The use of a gas ballast increases the amount of oil carried out in the exhaust.

Other common problems with mechanical pumps that also require routine maintenance and inspection include:

- Loose or slipping belts
- Improper oil level (too low or too high)
- Stuck discharge valve
- Clogged oil lines or valves
- Damaged discharge valve
- Ingestion of foreign contaminants (metal fines, metal chips, etc.)
- Excessive vibration (pipe connection or floor mounting)

Of the various diffusion-pump problems, exposure of the hot pump oil to the atmosphere or interruption of the coolant flow is of the most concern. Accidentally introducing air when the diffusion pump is at too high a temperature almost inevitably leads to a pump malfunction or failure, and this often requires expensive and lengthy repairs (most often at the manufacturer). Severe cracking (breakdown) of the oil and oxidation will occur depending on the type of oil. These lead to excessive backpressure, and the products of the oil breakdown will deposit on the jet structure blocking the openings or, in the area of the oil heater, burning it out. Overheating due to inadequate coolant flow also decomposes the oil and can cause excessive backstreaming into the vacuum furnace chamber.

Other common problems with diffusion pumps include power failures and excessively high foreline pressures.

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Tips for Selecting Induction Heating Equipment

Induction heating equipment is one of those areas that many of us need to know more about. We need to know when and how to apply this technology, if it is the right one for the job and how to purchase it if we need to do so. Let's learn more.

The following factors typically influence equipment design:

- Material
- Prior microstructure
- Part geometry
- Austenitizing temperature
- Production rate
- Power requirements, kW*
- Frequency selection, kHz*
- Pattern/profile (shape of heating area)
- Coil design*
- Process-development requirements
- Application-specific criteria (e.g., water versus polymer)
- Method of loading and unloading the workpiece (e.g., manual or robotic)
- Stock removal after heat treatment
- Type of tempering (furnace/oven versus induction)

* Typically selected by vendor based on information provided.

Key process parameters for induction heating include:

- Type (single-shot or scanning)
- Power level
- Frequency
- Part position (rotation)
- Quench flow and pressure
- Quench temperature
- Quench time
- Quench concentration (if polymer).

Questions to Ask

The following questions (and their answers) will help educate the buyer:

- How many kilowatts do we need to do the job?
- What frequency is needed for the case depth required?
- How does the part get placed into and removed out of the machine?
- How many different coil sizes (and types) will we need to cover our range of parts?
- How does the control system work? Are the

Fig. 1. Near-perfect tuning match

computer screens user-friendly, and do they have the type of information you need?

- Do we need an optical pyrometer as part of the control system? What are the effects of overheating and underheating, and how can we reject parts based on temperature criteria?
- What, if any, temperature differential will there be, from one end of the part to the other, from center to surface?
- What input voltage do we have in the plant?
- What type of plant cooling system is needed? If existing, is there unused capacity?
- How much will the machine cost to operate per unit of time? Are there demand charges for electricity (on-peak or off-peak)?
- How much will it cost to install?
- What maintenance needs to be done on the equipment? How frequently does maintenance need to be performed?
- How much (floor) space and headroom is needed and in what configuration?
- Do we need ventilation or an air exhaust system?
- Should we wash the parts prior to induction heat treating?

In addition, here are some specific items to focus on.

Power Supply

Power, expressed in kilowatts (kW), refers to the induction power-supply size. A power supply must be sized to heat a given mass or given surface area to a specific temperature within a specified time. A general rule of thumb is that the surface area (exposed to the coil) used to determine the power level is 6-12 kW/inch^2. The prior part microstructure (annealed, normalized, quenched and tempered) will influence

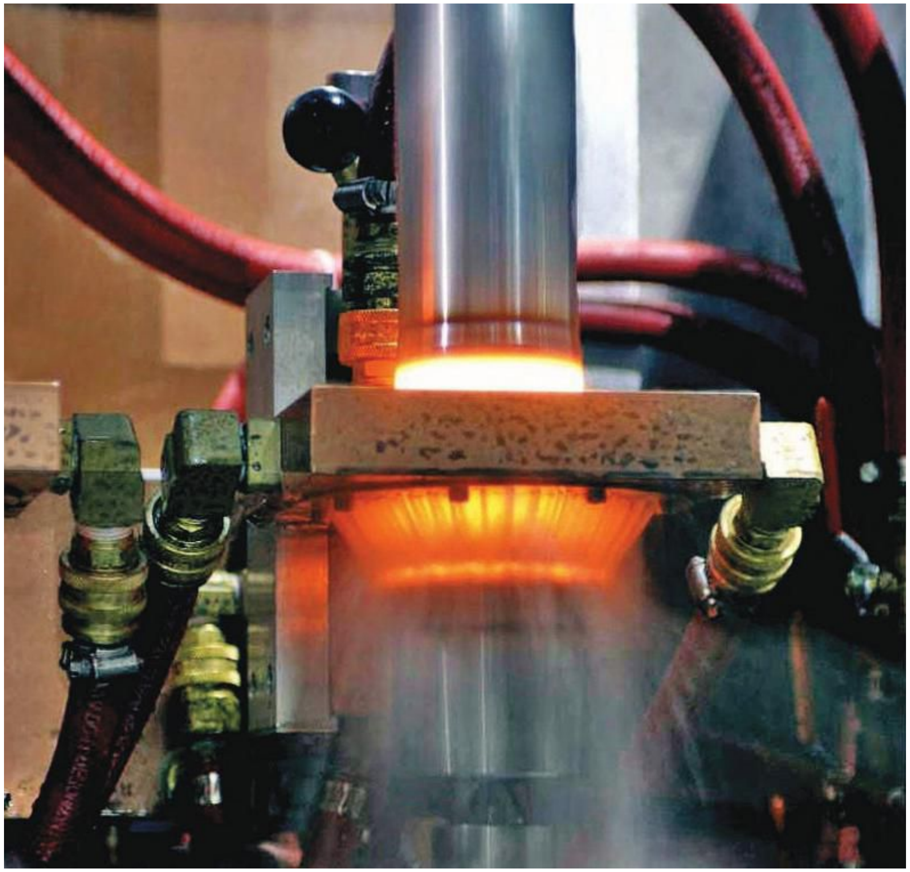

Fig. 2. Steering component heated with a machined single-turn coil

the power density (kW/inch^2) required. For example, a quench-and-tempered microstructure is optimum for most induction applications.

More power is not necessarily better. Matching the power and frequency (Fig. 1) is the key. While more power lets you heat faster, it may also melt the surface or produce a deeper pattern (there is also more danger of through hardening), and it is harder to control grain growth.

Frequency

High frequency in the form of alternating current is passed through the coil to create a magnetic field producing eddy currents. These are generated within the metal under the surface, and the resistance to this current flow is the principal source for heating of the metal.

The following guideline for "relative" depth of penetration (depth of hardening or case depth) as a function of frequency might be useful. This information is application-specific and dependent on both power density and heat time but is considered typical of what is found in the industry:

- @ 450 kHz the case depth developed is (approximately): 0.030-0.040 inch
- @ 100 kHz the case depth developed is (approximately): 0.050-0.080 inch
- @ 30 kHz the case depth developed is (approximately): 0.080-0.120 inch
- @ 10 kHz the case depth developed is (approximately): 0.090–0.200 inch

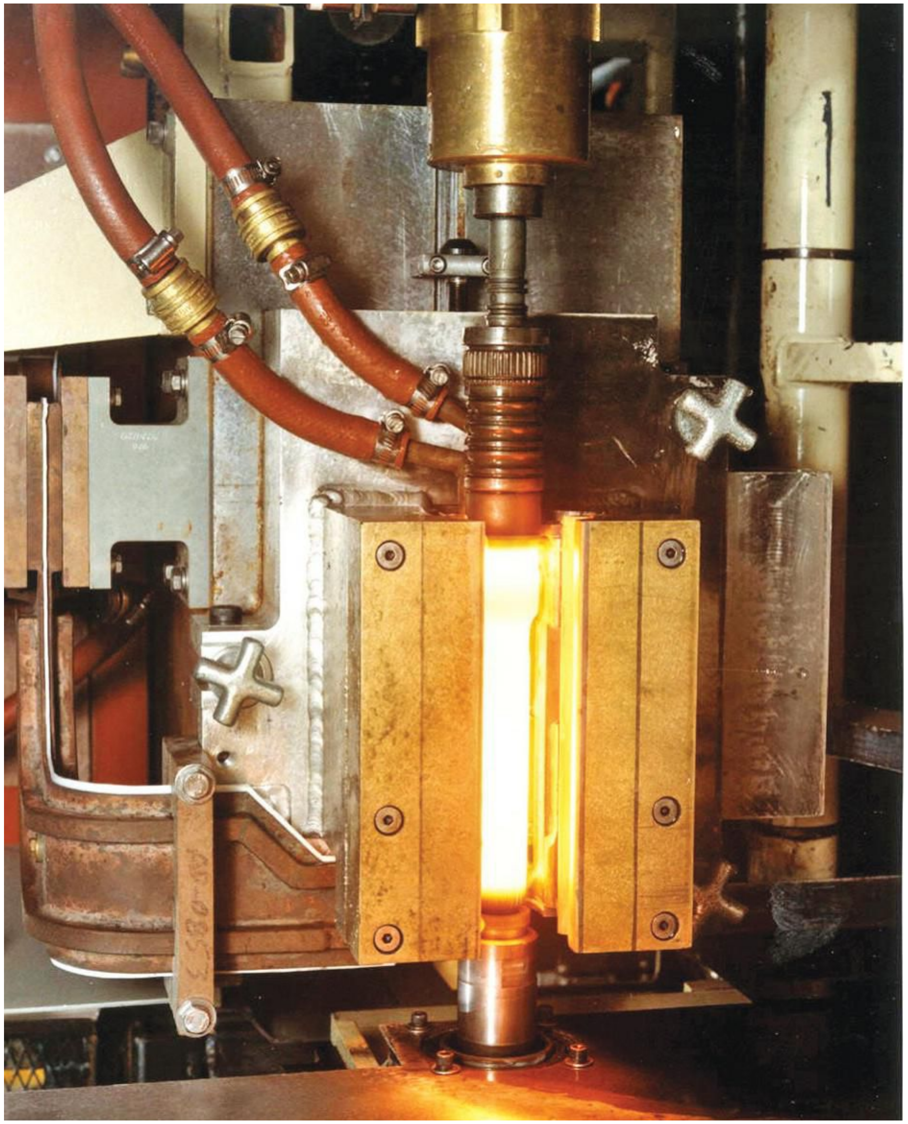

Fig. 3. Single-shot coil

The depth of current penetration (hardened depth) is a function of part diameter and the resistivity of the material. There is an optimum depth of current penetration range, which is what each manufacturer strives to provide.

Higher frequency has a distinct advantage when you have marginal prior part microstructures (annealed or normalized). Higher frequency allows the concentration of more energy near the surface of the part, avoiding long heating times or requirements for higher power density (more costly in terms of $/kW).

Tuning

Tuning (power-supply load matching) is a very important consideration to achieve the desired case-depth profiles and for overall power-system efficiency. This is done by adjusting the tap of a variable-ratio transformer or by adding/subtracting a portion of a capacitor to achieve the desired frequency and a balanced load match between amperes, volts and kW. These are typically viewed on a meter panel on the power supply.

The goal is to have a system that requires a minimal amount of tuning, which becomes more important as power levels approach the maximum nameplate rating of the power supply. For consistency of heating, the power supply should be tuned so that it is not running to its limit.

Computer-controlled and recipe-selected automatic capacitor contactors can be used to load match when many different workpiece sizes are heat treated on the same piece of equipment. Many power-supply manufacturers design a tuning window that allows you to make some changes to the part size being run. When only one part is dedicated to a system, no tuning is required after initial setup and testing.

Coils

All workpieces have their own function, shape and properties. Coils (inductors) are water-cooled copper tubing or machined from solid copper blocks. They are designed and built specifically to meet the metallurgical requirements and production rates of the workpiece. Coils require periodic maintenance (repair) and must be suitable for the applications whether it be scanning, static heating or single-shot heating.

Scanning inductors (Fig. 2) may be single-turn or multi-turn design. The required number of turns is determined by the ability to load match (tune) the coil to the output transformer in the power supply or to match specific process requirements. Too wide a heating zone affects the end of the pattern's transition zone. Scanning allows use of a smaller kW power supply and is easily adapted for a manual or an automated process for running a variety of different part diameters or lengths.

Single-shot inductors (Fig. 3) also knowns as static inductors are designed). They usually have very short heat times and have a fixture to position the part and rotate while heating. Quenching can occur in the coil, or the part can be indexed to a separate quench ring or a submerged tank with agitation.

Induction systems that need different coils to heat treat various workpieces use quick-disconnects so that no hand tools are required to change the coil. Systems heat treating just one part size with one coil can utilize bolt-on connections and hose clamps (requiring tools to change).

Controls

Control packages should be based on functionality, need and budget constraints. Factors such as how often one changes inductors, the number of shifts of operation, the skill level of the operator, the complexity of the workpiece (or pattern) and product end-use application all play a part when selecting what types of controls are best. Controls can be as simple as the power-supply kW output rotary pot with a timer. An ideal control package would utilize a Windows-based-software operator computer with a color screen that has recipe storage and can hold hundreds of workpiece process requirements.

A computer-based control system can store data such as dwell times, quench pressure, flow and temperature levels for a particular workpiece, kW at the coil and scan speed. Maintenance checklists and drawings or even a video of how to change the coil can be stored on hard drives.

Future Needs

A key ingredient of any successful purchase is flexibility and adaptability to new innovations. The following list will help you determine if you need this feature now or decide if it will be incorporated on the machine in the future. It will also help you figure out how much will it cost?

- Automatic adjustment of the power-supply frequency
- Automatic inductor-changing carousels
- Automatic recipe development
- Computer calculation of the power levels, scan speeds and dwell times necessary to process the first sample for subsequent metallurgical evaluation based on user inputs (diameter of the part to be processed, required case depth, type of material, face and I.D. dimensions of inductor)
- Automatic depth and hardness testing in the heat-treat machine
- Faster real-time processing recipe data
- Simpler and less-expensive controls
- One coil fits all size parts.

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Oil Quenching Part One: How to Interpret Cooling Curves

Cooling curves, cooling-rate curves and oil analyses are provided by suppliers to help heat treaters better control an important process variable – quenching. Unfortunately, in many cases these reports are not well understood or, worse yet, filed to be used merely as ISO documents. It's time to pull them out of the file cabinet and learn how to interpret them so we know what it is we are being told. Let's learn more.

The goal of quenching is to produce the desired hardness, strength and toughness in the part while minimizing distortion and residual stress. Uniformity of heat extraction by the quench medium is critical, and mechanical, physical and metallurgical properties developed are directly related to the material and the cooling rates within the part.

Cooling curves and cooling-rate curves (Fig. 1) not only tell us how the oil behaves through the three stages of quenching but also characterize the quenching behavior. In other words, these types of cooling curves measure our "quenching power" and tell us how the oil was performing on the day of the test. When looked at over time and quantified by part test data, they become a powerful predictive tool.

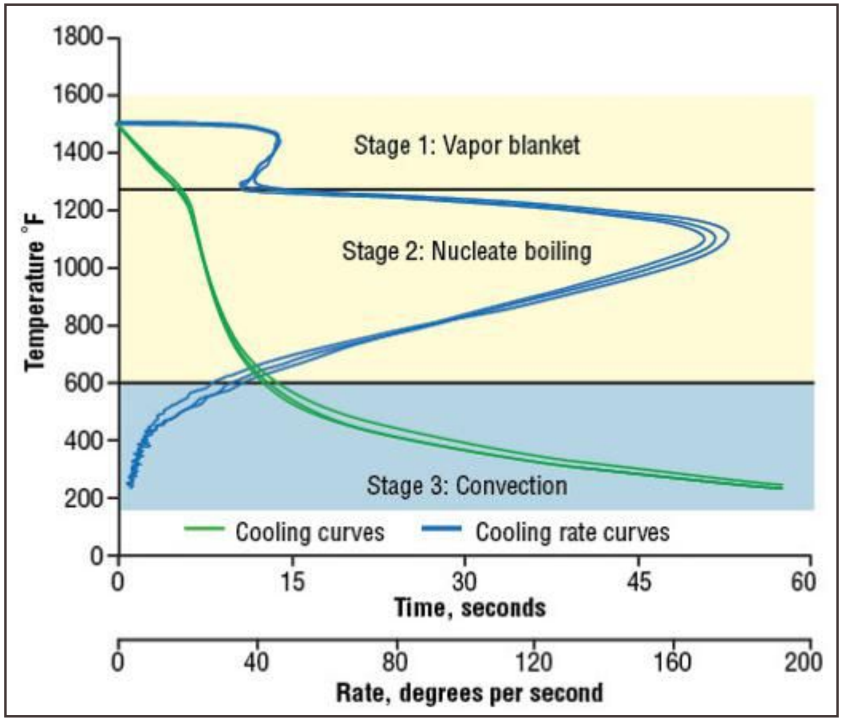

Fig. 1. Typical cooling curves and cooling-rate curves for new oils

The basic shape of a cooling curve is set by the quenchant formulation. Over time, we see shifts in the cooling curves, and as we build up a history of how the oil is changing, we can tell how different conditions – contamination, oxidation, drag-out, agitation – affect quench performance (Fig. 2).

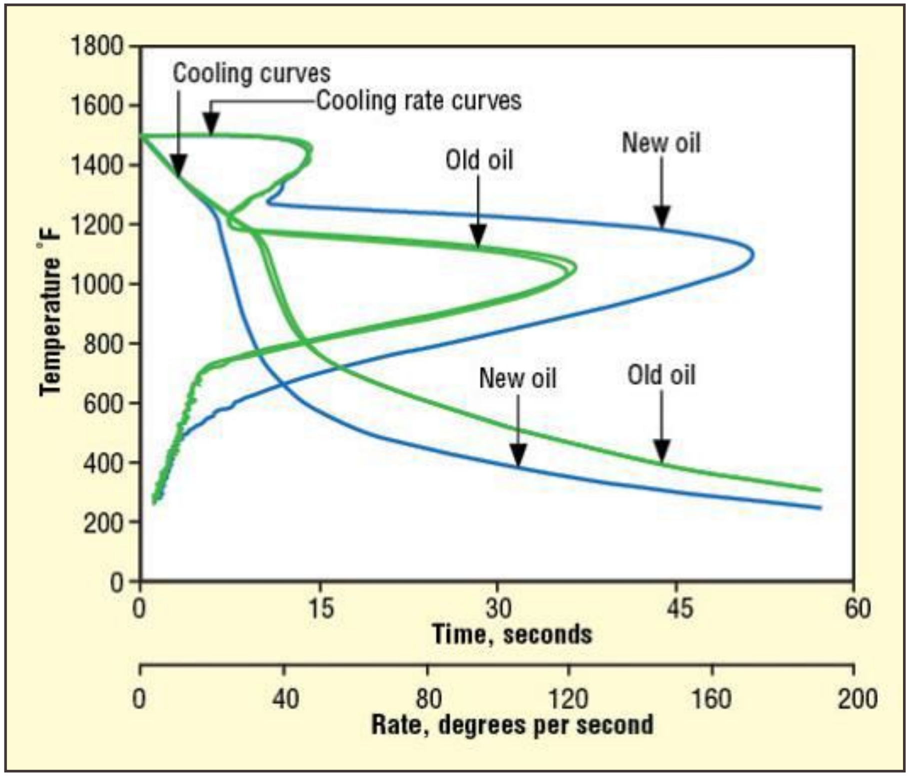

Fig. 2. Typical cooling curves and cooling-rate curves for new and used oils

Cooling curves are typically generated by immersing a heated thermal probe into an unagitated, heated quenchant and generating time/temperature data as the probe cools. Standard test procedures are based on ASTM D-6200 (Standard Test Method for Determination of Cooling Characteristics of Quench Oils by Cooling Curve Analysis) and ISO-9950 (Industrial Quenching Oils – Determination of Cooling Characteristics – Nickel Alloy Probe Test Method). These methods allow comparison of new oil results from supplier to supplier and from plant to plant worldwide.

Three Stages of Quenching

When quenching into any liquid, there are three distinct stages of cooling. Stage 1 is called the vapor-blanket stage, and it occurs immediately upon quenching. It is characterized by the Leidenfrost phenomenon – the formation of an unbroken film or vapor layer that surrounds and insulates the part. It forms because the supply of heat from the surface of the part exceeds the amount of heat that can be carried away by the cooling medium. In this stage, the cooling rate is a function of conduction through the vapor envelope. The cooling rate is slow since the vapor layer prevents the quenchant from contacting the metal surface.

As the temperature of the parts slowly drops, the vapor blanket becomes unstable and collapses, allowing the oil to come into contact with the metal surface. Stage 2 is known as the nucleate-boiling stage and is characterized by violent bubble boiling as heat is rapidly removed from the part surface. The maximum cooling rate (°F/s) is found during this stage along with the highest instantaneous value of the heat-transfer coefficient. Maximum part distortion also occurs during this stage due to differentials in uniform heat extraction from the various part surfaces.

Table 1 Classification of quench oils

Quench Oil Type	Cooling Rate [1] °F/sec (°C/s)	GM Quenchometer [2] Nickel Ball Test Seconds	Typical Operating Temperature Range ˚F (°C)
Fast	> 165 (74)	8 – 10	90 – 200 (32 – 95)
Medium	125 – 165 (52 - 74)	11 – 14	200 – 300 (95 - 150)
Slow	90 – 125 (32 – 52)	15 – 20	300 – 450 (150 – 230)
Marquench	90 – 125 (32 – 52)	18 – 25	300 – 450 (150 – 230)

[1] Per ASTM D 6200-01/2007
[2] Per ASTM D 3520-04 (Standard Test Method for Quenching Time of Heat-Treating Fluids - Magnetic Quenchometer Method)

Table 2 Maximum cooling-rate (°F/s) history

Description	1st Quarter (2006)	2nd Quarter (2006)	3rd Quarter (2006)	4th Quarter (2006)	1st Quarter (2007)
New Oil	174.5	175.8	168.7	168.7	171.9
Used Oil (IQ Furnace)	130.4	133.2	114.2	119.7	122.2
Used Oil (Open Tank)	167.9	169.3	163.2	164.7	167.8

Table 3 Characteristic temperature (°F) history

Description	1st Quarter (2006)	2nd Quarter (2006)	3rd Quarter (2006)	4th Quarter (2006)	1st Quarter (2007)
New Oil	1240	1242	1256	1260	1245
Used Oil (IQ Furnace)	1171	1171	1121	1144	1157
Used Oil (Open Tank)	1268	1270	1274	1281	1283

Table 4 Cooling-rate (°F/s) history at 600°F

Description	1st Quarter (2006)	2nd Quarter (2006)	3rd Quarter (2006)	4th Quarter (2006)	1st Quarter (2007)
New Oil	37.3	34.9	35.7	32.5	26.7
Used Oil (IQ Furnace)	14.2	13.9	14.0	14.1	15.0
Used Oil (Open Tank)	13.4	13.6	15.8	13.8	13.1

Table 5 H-value history

Description	1st Quarter (2006)	2nd Quarter (2006)	3rd Quarter (2006)	4th Quarter (2006)	1st Quarter (2007)
New Oil	0.37	0.37	0.38	0.37	0.36
Used Oil (IQ Furnace)	0.27	0.27	0.24	0.25	0.26
Used Oil (Open Tank)	0.34	0.34	0.35	0.34	0.35

Finally, when the part temperature has dropped below the boiling point of the quenching oil, Stage 3 – the convective heat-transfer stage – begins. This is the slowest of the three stages.

Quenching Speed and Other Useful Data

Quench oils are often classified according to their maximum cooling rate (Table 1). This data is especially useful when reported over time (Table 2). Other data, however, may be of equal or greater benefit to the heat treater such as: the characteristic temperature (Table 3), that is, the transformation point between the first and second stage of cooling – an indication of cooling "efficiency" for a given quench oil; the cooling rate measured at some temperature below the characteristic temperature (Table 4) where boiling ceases – an indication of cracking potential; and H-values (Table 5) – a measure of quench severity.

In the example shown, quarterly checks of cooling-curve data from both new and used quench-oil samples revealed that while the quench oil in an integral-quench furnace had cooling characteristics similar to the previous quarter's tests, it was significantly slower than a new oil or oil installed in another (open) quench tank. While the new oil and open-tank samples fit the category of a "fast" quenchant, this particular IQ furnace falls in the category of a "slow" oil. Quenching performance will be different for similar materials, parts and loads quenched in these two tanks. Adding accelerator can move the cooling curve of the oil in the IQ furnace closer to that of the new oil.

How to Benefit the Most from Cooling-Curve Data

Cooling curves are normally determined under controlled conditions in the laboratory as opposed to monitoring quench tanks in real time. Also, the choice of cooling probe further limits our ability to interpret the results because when quenching parts in the heat-treat shop, the quenching power of the oil and the heat-transfer characteristics vary not only over the surface of an individual part but within the workload as well. Thus, for example, the transition between the vapor phase and the nucleate-boiling phase can take place at different times at different points on the various part surfaces throughout the load. Even oxidation of the part surface is known to change the cooling performance. It is, therefore, critical for the heat treater to monitor part hardness, distortion and other properties to understand how his particular quench tank performs as his quench oil ages and changes.

Next time: Part Two: What Your Quench Oil Analysis Is Telling You

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Oil Quenching Part Two: What Is Your Quench-Oil Analysis Telling You?

For many heat treaters, the use of oil gives the best value based on a number of factors including[1]:

- Overall performance (cooling rate or quench severity)
- Economics/cost (initial investment, maintenance, upkeep, life)
- Minimization of distortion (quench-system performance)
- Variability (controllable cooling rates)
- Environmental concerns (recycling, waste disposal)

One additional factor – performance over time – should be added to the list since we are always looking for ways to extend the life of our quenchants without sacrificing their performance. Let's learn more.

Relationship of Physical Properties of Quenching Oils to Performance

Quench oil should be routinely analyzed (quarterly, or monthly if heavily used) to determine its performance characteristics. The testing laboratory or oil supplier's report (Table 1) should be carefully scrutinized as it contains information about the physical property characteristics of the oil. Oil analysis uses standard test methods (Table 2), but in order to gain deeper insights into the meaning of the test results, as opposed to just comparing them with previous results, we need to understand what each category is telling us.

Viscosity

Quenching performance is highly dependent on the viscosity of the oil. In general, viscosity increases as the oil degrades. Degradation can be in the form of oxidation, thermal breakdown or the presence of various contaminants. Oil viscosity changes with time, and the formation of sludge or varnish accelerates the process. Samples should be taken and analyzed for contaminants and a historical record of viscosity variation kept and plotted against a process-control parameter such as part hardness.

Water Content

One of the concerns regarding oil quenching is the presence of water in the quench oil. It is dangerous since, on quenching, water will form steam with a resulting volume expansion of approximately seven times. As the steam bubble rises out of the quench tank, its surface is coated with oil. As it exits from the furnace (usually under extremely high pressure), it is ignited at the burnoff, resulting in a huge evolution of flame.

Water detectors or other in-process monitoring devices with sensitivity in the range of 0.2-0.3% should be provided on all quench tanks. They should be properly maintained and tested daily. Some manufacturers believe that as little as 0.1% may cause dramatic changes in quenching and part surface contamination.

In addition to a fire hazard, a water concentration of 0.05% has been reported to cause soft spots, uneven hardness and staining. When water-contaminated oil is heated, a crackling sound may be heard. This is the basis of a qualitative field test for the presence of water in quench oil. Sources of water include water-to-oil heat exchangers, water-cooled seals, plate coils or water-cooled bearings. The most common laboratory tests for water contamination are either a Karl Fisher analysis (ASTM D1744) or distillation.

Flash Point

The flash point is the lowest temperature where oil vapors will ignite but will not continue to burn when exposed to a spark or flame. The flash point is the maximum safe operating temperature of the oil, and changes in the flash point indicate contamination of the quench bath. There are two types of flash-point values that may be determined – closed-cup or open-cup. In the closed-cup measurement, the liquid and vapor are heated in a closed system. Traces of low-boiling contaminants may concentrate in the vapor phase, resulting in a relatively low value. When conducting the open-cup flash point, the relatively low boiling by-products are lost during heating and have less impact on the final value. The most common open-cup flash-point procedure is the "Cleveland Open Cup" procedure described in ASTM D92. As a general rule, oil should be operated no higher than 150°F (65°C) below the flash point or about 100°F (38°C) below the flash point on quenching a full load.

Oxidation

This variable may also be monitored and is especially important in tanks running marquenching oil or oils being run above their recommended operating range. Oxidation results from the buildup of organic acids and is detected by infrared spectroscopy. It is measured by several methods, including: precipitation number, total acid number, sludge content and viscosity. The cooling curve will change, increasing in speed for cold

Table 1. Typical Quench Oil Report*

Sample ID	02-190	01-1299	01-983	01-615
Date sampled	2/5/02	11/13/01	8/21/01	5/29/01
Water, % <0.1	0.001	0.006	0.018	0.015
Visc. @100˚F (SUS) 75-100	92	91.6	91.3	90.6
Flash point, ˚F >335	350	350	350	350
Sludge, % <0.20	0.01	0.01	0.02	0.01
Precipitation No. <0.15	0.01	0.01	0.01	0.01
GMQS @ 80˚F (sec) 7-10	8.9	8.9	8.6	8.8
Problems reported	None	None	None	None

oil and decreasing in speed for hot/marquenching oils. Nitrogen blanketing of the oil is one way to reduce both oil oxidation and sludge formation.

Precipitation Number

The precipitation number is an indication of the tendency to form sludge. Sludge is one of the biggest problems encountered in quench oils, and high precipitation numbers also indicate a propensity to stain parts. Although other analyses may indicate that the quench oil is performing within specification, the presence of sludge may still be sufficient to cause nonuniform heat transfer, increased thermal gradients, and result in cracking and distortion. Sludge may also plug filters and foul heat-exchanger surfaces (the loss of heat-exchanger efficiency may cause overheating, excessive foaming and possible fires).

Sludge formation is caused by oxidation and polymerization of the quench oil and by localized overheating ("frying") of the oil. The relative amount of sludge present in quench oil may be quantified and reported as a "precipitation number." The precipitation number is determined using ASTM D91. The relative propensity of sludge formation of new and used oil may be compared providing an estimate of remaining life.

Neutralization Number or Total Acid Number (TAN)

As oil degrades, it forms acidic by-products. The amount of these by-products may be determined by chemical analysis. The most common method is the neutralization number. The neutralization number is determined by establishing the net acidity against a known standard base such as potassium hydroxide (KOH). This is known as the "total acid number" (TAN) and is reported as milligrams of KOH per gram of sample (mg/g).

The TAN is an indication of the level of oxidation. As the TAN increases, the vapor phase becomes less stable and the maximum cooling rate increases while distortion, cracking and staining tendencies increase. Both precipitation number and total acid number are controlled by filtration.

Table 2. Quench-Oil Test Standards

Test	ASTM Method
Viscosity	D 445
Flash point	D 92
Fire point	D 92
Water	D 95/D 6304
Neutralization number	D 974
Ash	D 484
Conradson carbon residue	D 189
Precipitation number	D 91
Sludge	D 91
Specific Gravity	D 287
Quenching speed (GMQS)	D 3520
Quenching speed (Cooling curve)	D 6200

Quench Speed

Quench speed (see "Part One: How to Interpret Cooling Curves") is an important measure of the oil's ability to achieve its performance properties. It can be determined by two methods, GM Quench-O-Meter (GMQS) and cooling curves. Probe surface condition and the condition of oil are factors that can influence results. Data should always be referenced back to new quench oil.

Accelerator Performance

Accelerants are often added to quench oils to return their performance characteristics close to those of new oils and to extend oil life. In general, it is not a good idea to mix an accelerator package from one supplier with oil from another. Induction coupled plasma (ICP) spectroscopy is one of the most common methods for the analysis of quench-oil additives. When additives (such as metal salts) are used as quench-rate accelerators, their effectiveness can be lost over time by both drag-out and degradation. Their effectiveness can be quantified by performing ICP spectroscopy – a direct analysis for metal ions – and compensating measures can be taken such as the addition of a specific percentage of new accelerator.

Summary

No matter what quenchant is used or how confident we are in its performance, routine testing is important. Our ability to interpret test results is equally important so that we can make informed judgments as to how we best control our heat-treating processes.

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Secrets of Effective Hot Oil Quenching

Martempering and marquenching are terms often associated with hot oil quenching. Formally, martempering (full martempering, true martempering) is a term applied when an austenitized work piece is quenched into a medium whose temperature is essentially maintained in a bath just above the martensite start (M_s) temperature of the steel and held in that medium until its temperature is uniform throughout – but not long enough to permit bainite (or pearlite) formation – and then allowed to cool in air (Fig 1a). When the martempering process is applied to carburized material, the controlling martensite start temperature is that of the case and as such this process variation is called marquenching.[1]

A modified form of these processes is called hot oil quenching (modified marquenching) and takes place just below the martensite start temperature (Fig. 1b). In many steels the required critical cooling rate is such that faster quenching than that possible by marquenching or martempering is necessary to obtain full hardness. It has also been found effective in reducing quench stresses and improving part dimensional stability.

But how is hot oil quenching best accomplished in the real world? Let's learn more.

What is Hot Oil?

Quenching into oil above 212°F (100°C) has traditionally been referred to as "hot oil" quenching. Oil temperatures in the 195°F - 450°F (90°C - 230°C) range have been used with both ends of the spectrum normally reserved for special applications. Hot oil temperatures in the 220°F - 375°F (105°C - 190°C) range are common in the heat-treating industry and take place in either batch or continuous atmosphere or vacuum-sealed quench furnaces.

Part Distortion

Distortion arising from heat treatment can be classified as two types: size distortion due to volume changes resulting from phase transformations (which are generally predictable) and shape change or warpage (which is generally unpredictable). The latter takes many forms including out-of-round, out-of-flat, bending, bowing, bucking, taper, dishing, closing-in of bores and can arise either during heating or

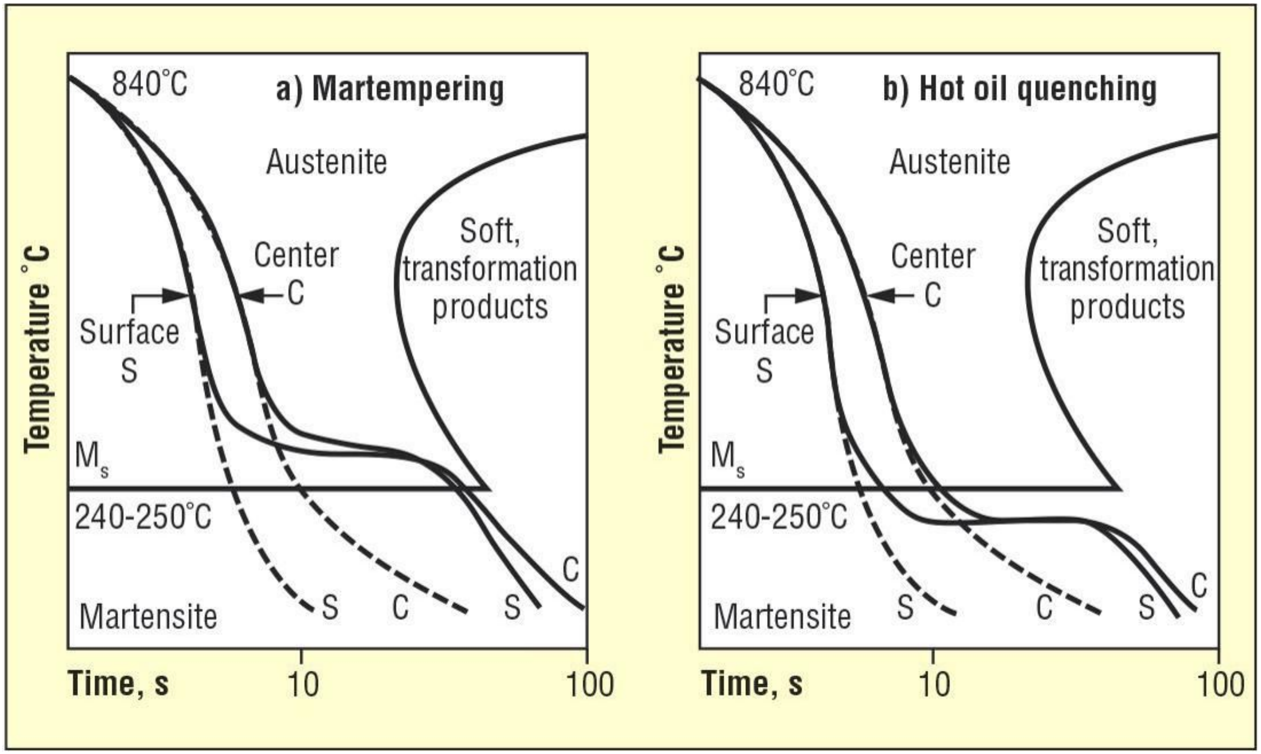

Fig. 1[2] Comparison on Martempering (a) and Hot Oil Quenching (b)

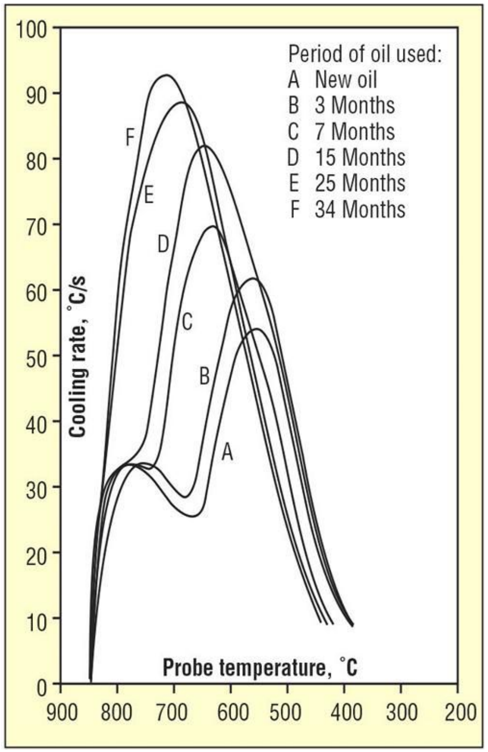

Fig. 2 [2] Influence of Hot Oil Age on Cooling Rate/Temperature Profile austenitization through the relief of internal stresses from prior operations; by sagging or creep due to inadequate part support during heating; mechanical damage; non-uniform heating; or during quenching, the result of an imbalance of the internal residual stresses generated. A number of key factors influence these changes including:

- Steel composition and hardenability
- Component part geometry
- Mechanical handling
- Types of quenchant;
- Temperature of quenchant
- Condition of quenchant
- Circulation of the quenchant

In the real world we find that cooling rates vary widely based on the type, temperature, condition, and circulation of the quenchant, which changes daily. Due to the fact that cooling rarely (if ever) occurs uniformly on the component part, we find that different quenchants produce different time durations throughout the three stages of liquid quenching (vapor phase, boiling phase, convective phase) and have a direct bearing on distortion.

Operating temperature can have a dramatic influence on distortion; hence oils have been developed for martempering and marquenching applications to minimize residual stresses by promoting uniform transformation. Hot oils are generally applied to high precision engineering components such as thin-section bearing races, and transmission gears and shafts requiring critical dimensional control. The condition of the quenchant, which for hot oils involves oxidation, contamination, and degradation, dramatically influences results since viscosity increases. It is not uncommon to filter or centrifuge hot oil baths daily and to replace the oil every 12 to 18 months. With hot oil, the cooling rate in the convective phase is increased significantly with agitation, so excessive agitation is to be avoided to counter distortion. The speed and direction of oil flow over the workpiece can also determine the nature of the distortion.

Oil Selection

The choice of oil depends on the need and is dependent on such factors as:

- Hardenability of the alloy
- Critical cooling rate and M_s and M_f temperatures as determined from the transformation (TTT) diagram of the steel
- Part austenitizing temperature in relation to oil temperature
- Part and load geometry

- Part cleanliness requirements
- Distortion expectation (allowable dimensional changes)
- Safety

For example, leaner alloyed parts usually require lower viscosity martempering oil often with speed enhancers added. Higher alloyed parts usually require higher viscosity oils and certain additives allow higher oil temperatures where distortion control is maximized. Thin distortion prone components are best martempered or quenched into the slowest speed martempering oil, which will produce the required metallurgical properties.

Hints for Extending Oil Life

The hotter the oil's temperature, the better the distortion control but the faster the oil's degradation. Also, the higher the austenitizing temperature from which a part is quenched, the more damage to the oil and the faster the oil will deteriorate. Other common concerns are oxidation and slug/contamination buildup. These can be minimized to a degree by the addition of antioxidants and with the use of a protective atmosphere cover (such as a nitrogen blanket) over the oil during heatup and operation. Oil without antioxidant additives will give the brightest and most consistent part surface appearance but will oxidize rapidly, then discolor the work. Antioxidant additives will normally produce a consistent surface finish while extending the oil's useful life. Fresh or make-up oil can be added to further reduce oil degradation. A hidden danger is heating marquench oil up too rapidly, which can also degrade the oil (low velocity burners or low watt release resistance heaters should be used).

It is important to note that oil capacity is not always an assurance of success. For example, parts run in continuous furnaces discharging parts into quench chutes may see problems with low hardness or staining due to breakdown (fractionation) of the oil in a small localized area, lack of proper heat extraction or poor oil circulation in the quench chute.

Cooling systems should be sized to handle the heat extraction and should be free of copper and other materials known to be catalysts for oxidation of oil products.

Effect of Oil Changes Over Time

As the oil ages, its heat extraction capabilities will change, influencing the end product gradually over time. In one study,[1] during the life of a hot oil, the cooling characteristics changed progressively (Fig. 2). As seen, the vapor blanket phase disappeared and

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the maximum quench rate increased (and occurred at higher temperature) resulting in a slowing down of the quench rate at lower temperature.

Finally, when changing to marquenching oils, it is important to recognize that the design of the quench tank may have to be changed or optimized for their use. Previous quenchants, sludge and water should be removed from the tank prior to introduction of the hot oil. Remember that the oil will expand and its viscosity change during heating. Protective gas atmosphere over the oil should always be used during initial heatup, and exposure to air must be minimized at all times.

Summary

Martempering oils, when applied properly, can improve part distortion control and minimize rejects due to such issues as part cracking. Martempering oils can replace other quenchants, but testing should be performed to balance the various factors discussed and to optimize their performance and life. Reduction of part production cost should be possible where the use of oil has advantages over other quenchants. Safety issues are little different than that with any other oil.

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Fundamentals of Press Quenching

Heat treaters know gears and bearing races are especially prone to dimensional changes during hardening and quenching, which can cause a number of problems during post-heat treatment manufacturing operations. Typically, additional stock allowances are needed to compensate for distortion so that parts can be machined to the proper finished dimensions. The objective of press quenching is to hold parts round and flat while they are being cooled, thereby reducing, though typically not eliminating, distortion. Let's learn more.

Hardened or case-hardened gears or bearing races are either free or press quenched. The latter involves moving them individually out of the furnace by manual or robotic means into a quench press (Fig. 1). The transfer from the furnace to the quench press, with oil or polymer flowing, must be relatively fast - typically under 10 seconds. A high volume of quenchant, uniformly directed to all internal and external surfaces of the parts, is needed. Control of out-of-roundness to within 0.005" (0.127 mm) and out-of-flatness to within 0.001" (0.025 mm) is common from most industrial presses.

Critical Considerations

Consideration and regulation of the following press parameters has been found to help control distortion:

1. Transfer time from furnace to press (consistent)
2. Manipulator contact (area, duration)
3. Part positioning on the die
4. Die design
5. Die pressure (clamping force, expander force) applied to hold the component
6. Quenchant temperature
7. Direction of quenchant flow
8. Quenchant pressure
9. Quantity of quenchant
10. Location of points of contact on the component
11. Duration of quenching (at various flow rates)
12. Flow paths to and through the lower die for the quenchant to reach top and bottom simultaneously
13. Die set maintenance and repair
14. "Pulsing" feature (optional)

It is especially noteworthy to mention that old, worn or damaged tooling can be responsible for as much, if not

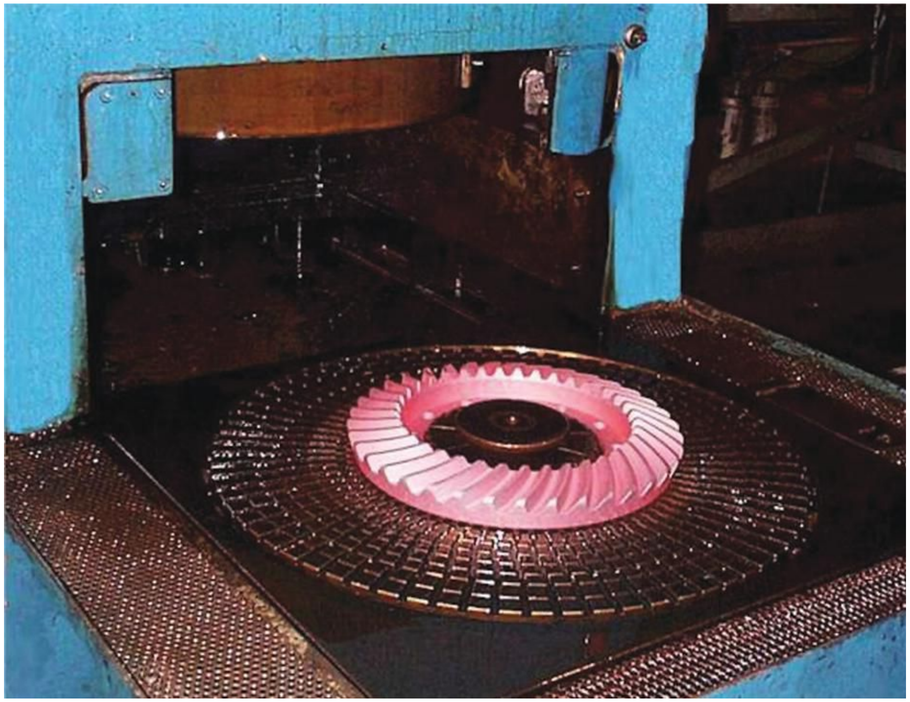

Fig. 1. Typical press quench operation

more, distortion than part design or geometry. Quench dies should be routinely inspected for damage or wear and repaired or replaced as necessary. Also important is to check final dimensions after repair since one of the hidden dangers is that of altered or restricted quenchant flow.

The "pulse" feature is a popular option on presses as it allows a part to expand and contract normally while still controlling shape. Without it, stresses are induced because the part is not allowed to contract and expand. Pulsing reduces the friction caused by constant pressure and clamping on the race as it contracts during cooling. This friction promotes stresses that result in eccentricity and out-of-flatness. Properly applied, the pulsing technique finds the die in contact with the part throughout, but the pressure is released and re-applied every two seconds during the entire quench cycle. The expander pressure is normally not pulsed

How the Press Works

A typical press-quench system (Fig. 2) will operate as follows. The component to be quenched is removed from the furnace (usually a continuous pusher or a rotary hearth furnace) and placed onto the lower die in the out position. Initiation of the automatic cycle moves the loaded lower die assembly into the center section of the machine. When this is fully advanced, the upper ram assembly and dies descend, with an expander centering the part just prior to the inner and outer dies locating on their respective pressure points. When the expander and dies are properly located, the ram holding them is latched in the down position and pressure is applied to all three. The inner die, outer die and expander usually have completely independent pressure controls, ranging from 0-24,000 lbf (0-107 kN) on the dies and 0-11,000 lbf (0-49 kN) on

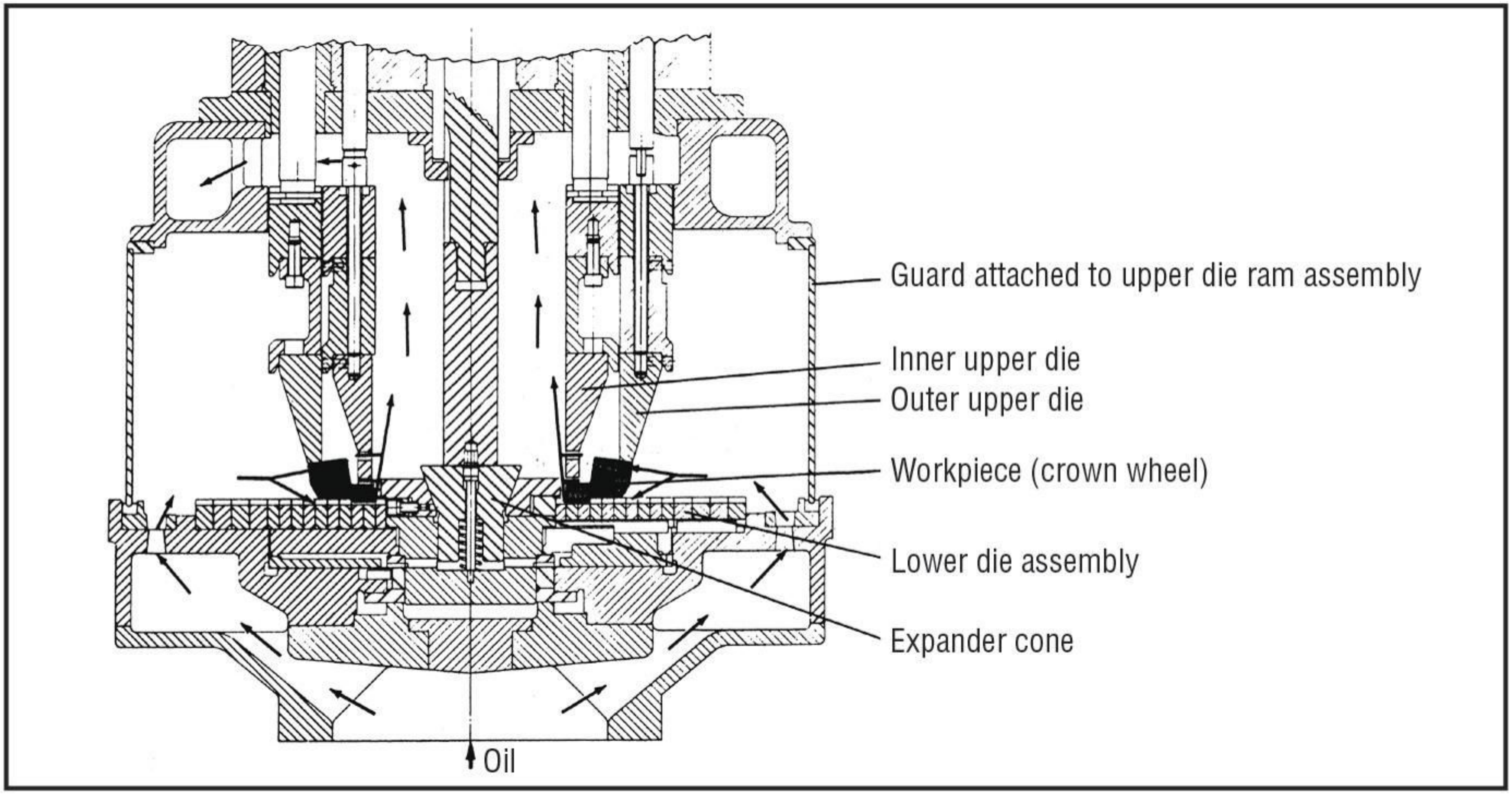

Fig. 2. Quenchant circulation in a typical press quench [1]

the expander. These are often regulated by hydraulic valves and monitored via pressure gauges. When used in a bore, the expander cone pushes out against the segmental lower die in order to hold the bore round and to size. The inner and outer dies help keep the component flat.

A guard usually completely encloses the upper dies, forms a quenching chamber and is attached to the upper ram moving up or down with it. When the upper ram latches, the quenchant pump starts supplying the chamber with fluid. The volume of quench fluid is normally controlled by a series of switches. In a typical press, each switch controls a solenoid valve supplying different flow volumes, often in the range of 50 gal/min (190 l/min), 100 gal/min (380 l/min) and 200 gal/min (758 l/min). Any combination may be used, up to a maximum of say 300 gal/min (1135 l/min). With all switches off, a minimal flow usually around 10 gal/min (40 l/min) is still maintained.

A circulation path within the press is created as the quenchant is pumped into the quench chamber through apertures around the outside diameter or through holes in the lower die. Quenchant fills the chamber around the component and flows out at the top. The elongated exit apertures may be fully open or closed to restrict flow, depending upon requirements. Timers used in conjunction with the flow-selector switches provide control of the duration of flow as well as volume, allowing a variety of flow/duration combinations.

Quality Assurance Considerations

In addition to the actual press-quenching operation itself, there are several ways to assist in maintaining the parts as distortion free as possible. Component design and manufacturing methods are the most critical in minimizing distortion, but other factors are also important. In engineering, the design should consider distortion from the initial concept through all phases of production processing, including material considerations (type and source). For example, the forging process should be designed so that material flow minimizes stress patterns. Forgings should be normalized above the austenitizing temperature, and after machining, stress relief at subcritical temperatures is needed.

Part Design

It is important in part design to avoid excessive or abrupt section changes that promote unequal heating and cooling rates. Fixturing and part support at temperature should also be considered during the design phase as creep from poor support is another major cause of distortion. The machinery employed in pressure quenching can normally hold twice the machining tolerances required of one area in its relationship to another. For example, if on a single plane the relationship between two surfaces is ± 0.002" (0.051 mm), then after quenching the best one can expect to attain is ± 0.004" (0.102 mm).

Part Sampling

In press quenching it is always a good idea to run a sample lot of parts prior to general production. In this way, the degree and nature of the dimensional changes can be observed. Possible changes in manufacturing operations can then be determined. Sampling also enables one to tell if specialized tooling is needed in conjunction with the press-quench machine for size control. Finally, metallurgical checks will allow determination of the optimized part microstructure as a function of setup parameters.

Record Keeping

Press-quench die sets and associated components (plugs, expanders, rolls, etc.) should be marked in a suitable manner to identify them for use with certain part numbers. The press-quench setup data should be stored in a computer database or kept as a written record, including:

1. Die numbers
2. Plug numbers
3. Quench time
4. Flow rates
5. Ram and expander pressure
6. Pulse (on/off)
7. Other pertinent data

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A Review of Gas Quenching from the Perspective of the Heat Transfer Coefficient

What is Pressure Quenching? The description most often used to define high pressure gas quenching is "accelerating the rate (speed) of quenching by densification and cooling of gas."[1] One of the many reasons for the intense interest in this quenching technique is related to improved part distortion and higher core hardness. A critical concern in using this technology is to avoid sacrifice of metallurgical, mechanical or physical properties, that is retain the ability to transform a material to a microstructure that is similar, identical or superior to that of a known quenching medium (e.g. oil or salt). There is a wealth of published (and often confusing) empirical data on the subject and more being generated daily. To sift through this mass of information, an understanding of the basic principles of the technology is needed. Let's learn more.

A Theoretical Understanding of the Heat Transfer Coefficient

A key difference between quenching in a liquid and quenching in a gas lies in the different mechanisms involved in their heat transfer characteristics. Most liquids (Fig. 1) such as water or oil have distinct boiling points, and thus different heat transfer mechanisms (and rates) at various temperature stages (e.g. oil has three distinct heat transfer phases: vapor blanket or "film" boiling, nucleate or "bubble" boiling and convection). For gaseous media (Fig. 2) heat transfer takes place by convection only.

There is a relationship (Equation 1) that describes the convection heat transfer (q') in terms of the heat flux (or heat flow) through the surface of a part that is proportional to the difference in temperature of the part surface (T_s) and the temperature of the quench gas (T_g) at any given moment in time.[2] This proportionality value is the heat transfer coefficient, α ("alpha").

$$q' = \alpha\,(T_s - T_g) \qquad \text{(Eq. 1)}$$

In convective heat transfer, a complex formula (Equation 2) describes the heat transfer coefficient (α) as dependent on the gas type (as described by its heat capacity (C_p), its heat conductivity (λ), and dynamic viscosity (η) of the gas), and on a geometric factor (d), which represents the area of free passage of the gas for a specific load arrangement. The physical laws that illustrate how velocity (v) and pressure (ρ) influence the heat transfer coefficient are given by:

$$\alpha = C\, v^{0.7}\, \rho^{0.7}\, d^{-0.3}\, \eta^{-0.39}\, c_p^{0.31}\, \lambda^{0.69} \qquad \text{(Eq. 2)}$$

where C is a constant[3].

Thus the greatest influence on the gas-cooling rate is the magnitude of the convective heat transfer coefficient (α), which in gas quenching is dependent on velocity and pressure as well as gas temperature. In other words, for a given geometrical arrangement (e.g. transverse gas flow over a cylindrical body) and a given gas type (e.g. nitrogen or helium), the heat transfer coefficient is directly proportional to the product of gas velocity and pressure raised to an overall (system dependent fluid dynamics constant) power, which is generally between 0.5 to 0.8.[4]

A Practical Understanding of the Heat Transfer Coefficient

Now that we have a theoretical understanding of the heat transfer coefficient, let's understand its importance in respect to heat-treating applications.

Liquid quenchants such as water, polymer or oil have the characteristic that during their nucleate boiling phase, extremely high heat

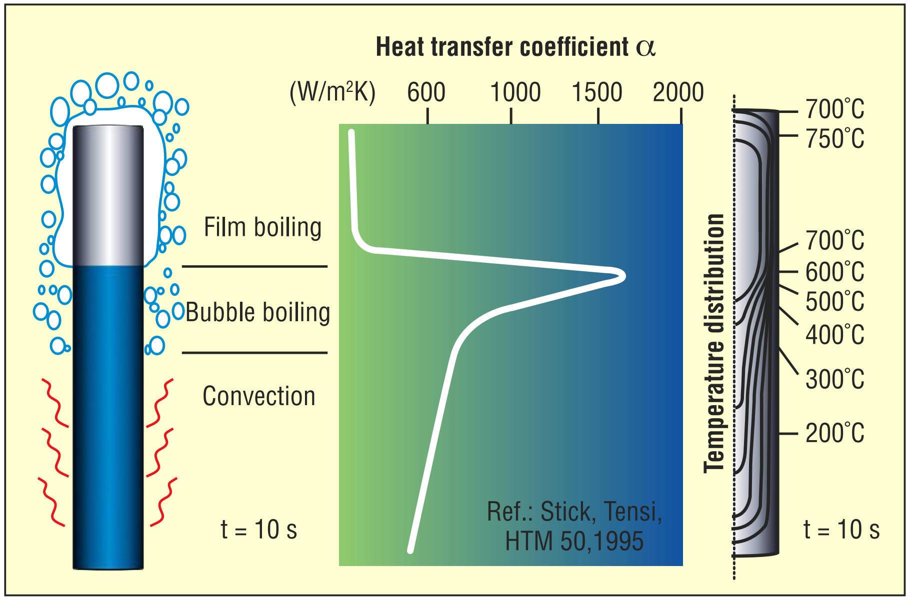

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Fig. 1. Heat transfer in liquid quenching

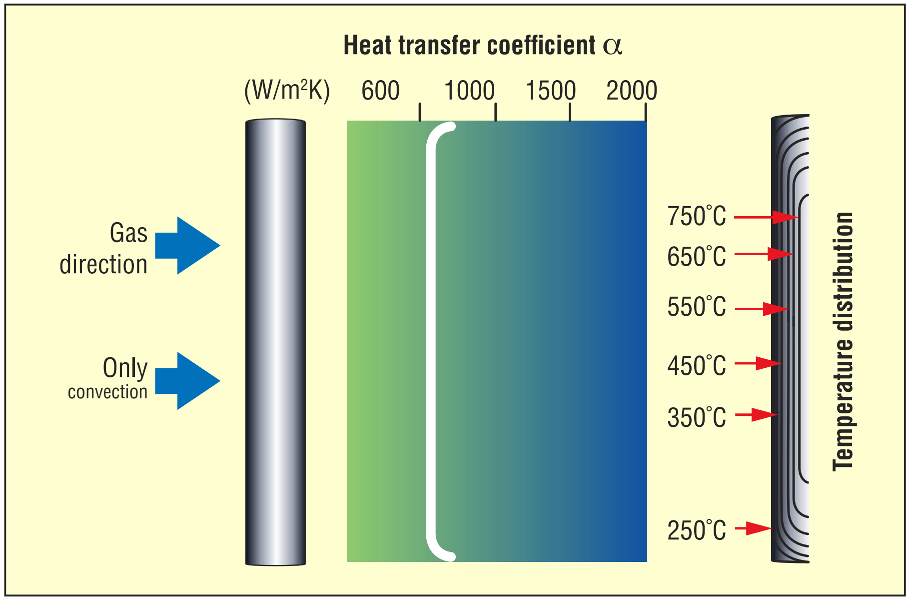


Fig. 2. Heat transfer in gas quenching

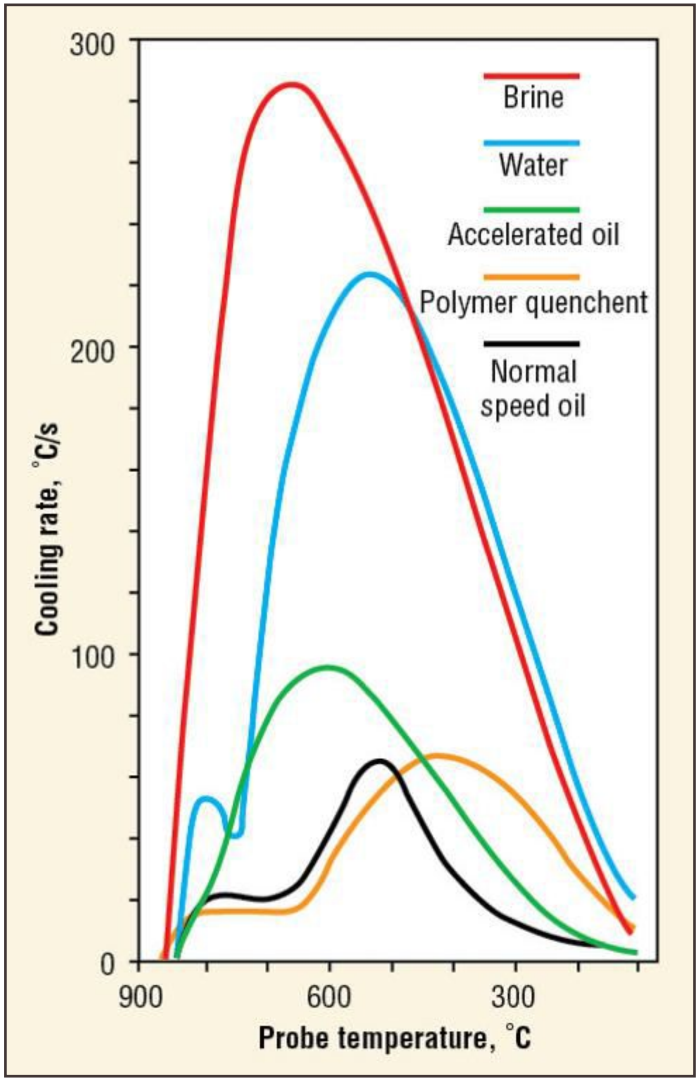


Fig. 3. Heat transfer coefficient in liquid quenchants

transfer coefficients can be achieved (Fig. 3). This is a distinct advantage in the temperature range where pearlitic transformation occurs and one not possessed by gas quenching. However, with the breakdown of the vapor phase at the onset of boiling, the so-called Leidenfrost phenomenon occurs and the result is a totally non-uniform heat transfer rate on different surfaces of the different parts and is dependent on a variety of variables and factors. This uneven transitory step creates huge temperature differentials and is the major factor in distortion when quenching in these media.

Although the maximum quenching “power” may be described by the instantaneous value of the heat transfer coefficient, the average heat transfer coefficient (Table 1) provides a better relative comparison of the various quenching media since it represents the value of the heat transfer coefficient over the entire range of cooling (from the start to the end of quenching).

Gas quenching avoids the Leidenfrost phenomena and therefore has an inherent capability to produce smaller temperature differences in a part during quenching, and thus less dimensional variation. However, as can be seen, its relative ability to successfully transform the microstructure of a part may be limited (Fig. 4).

Table 1. Average heat transfer coefficient values

Quench Media	Average value	Instantaneous value
Brine or caustic	3500 - 4500	> 15000
Water	3000 - 3900	> 12000
Oil, highly agitated	2000 - 2500	4000 - 6000
Polymmer	1500 - 2000	3000 - 4500
Oil, agitated	1500 - 1750	3000 - 4000
Oil, still	1000 - 1500	
Gas, high pressure	300 - 1000	1000 - 2000
Salt	400- 500	
Air	100 -300	

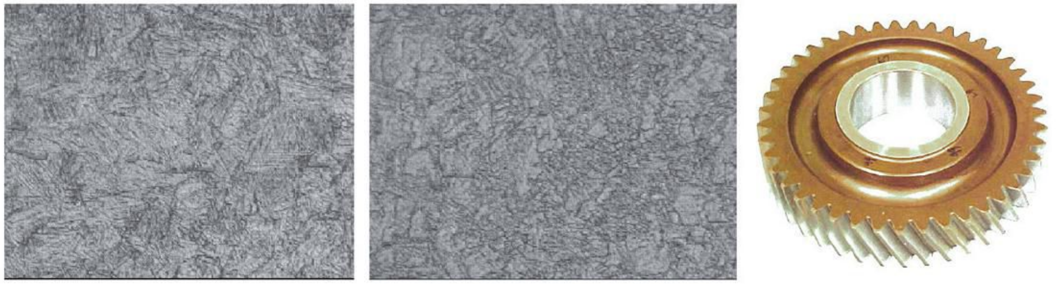
Factors That Influence Quenching Performance

The calculation of the heat transfer coefficient helps us to determine how quickly a workload will cool. There are, however, other factors (Fig. 5) that significantly influence the cooling rate and overall time involved. These are generally classified as "external" factors and are sometimes included in the formula for the heat transfer coefficient (Equation 2), as the multiplier f^a. The exponential value (a) for this factor is often equipment dependent and in the case of single chamber vacuum furnaces has been found to be in the range of 0.25 to 1.55.[7]

An understanding of the influence of these factors might be seen in a comparison of cooling rates between a single chamber vacuum furnace (hot chamber) and a cold ("blank body") chamber (Fig. 6). However, this test is reported to be somewhat misleading[9] in that it involved the use of ISO-9950 test probe (a small test specimen, 12.5 mm diameter × 60 mm long was used, which was developed for laboratory testing of quench oils). In fact, the ISO-9950 standard is entitled: Industrial Quenching Oils—Determination of Cooling Characteristics—Nickel Alloy Probe Test Method.

There are at least two main concerns why using this small specimen in HPGQ applications would be of concern:

1. A small mass cools much quicker than a typical part and as such fails to represent the entire quenching process for most components.
2. The small volume, having the thermocouple at its geometric center, fails to register a difference in cooling relevant to its position (vertical or horizontal) or to the gas flow direction (top to bottom, bottom to top, or alternating).



Microstructure is martensite
Core hardness = 44 HRC

Microstructure is primarily martensite with transformation products (bainite and ferrite) present
Core hardness = 28 HRC

Transmission gear
26 lbs (12 kg)/piece
AISI 8822H

Fig. 4. Microstructural variation in gas quenching

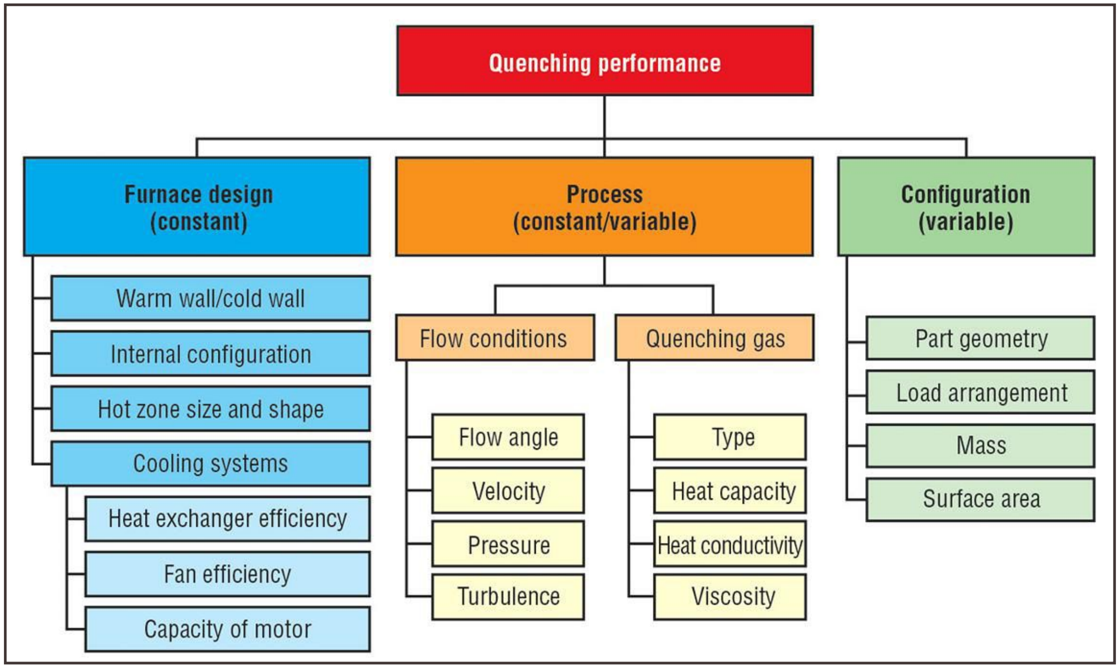


Fig. 5. Variables in high-pressure gas quenching performance[8]

For these reasons, a significant research focus has been on probes designed to measure the heat transfer coefficient within the workload during the gas quenching. These are available from a number of manufacturers.

Advantages and Disadvantages of High Pressure Gas Quenching

Properly applied, gas quenching has several recognized advantages,[2] which include:

- Safety
- Overall environmental impact
- Reduction of secondary manufacturing operations
- Optimization of dimensional variation
- Controllable cooling rates
- Overall economics

Of course, there are disadvantages,[2] which must be factored into any consideration to use this technology. These include:

- Cooling rate limitations (i.e. quench severity)
- Reversed application of heat transfer rates (i.e. slow cooling rates in the pearlitic transformation range and high cooling rates in the martensitic transformation range)
- Regulations and (pressure vessel) codes
- High noise levels

Recent Developments

Work is being done to characterize steel hardenability by a number of researchers including those in Europe. In high-pressure gas quenching applications, the ability to change the quench behavior of gases by varying individual quench parameters (e.g. gas type and composition, pressure; circulation pattern and velocity) play an important role in achieving improved hardenability in high-pressure quench applications. One example is research in the area of development of the gas quench equivalent of the Jominy test.[11]

Also, there are a number of probes and other devices designed to measure the heat transfer coefficient within the workload during the gas quenching.[12] The measurement and recording of the quenching intensity (and heat extraction dynamics) in HPGQ applications has been solved by using these types of probes. These types of sensors have thermocouples at precisely defined positions, enabling the calculation of the flow of thermal energy and thus the thermal heat transfer coefficient from the temperature measurement data during gas quenching (they are usually instrumented with three thermocouples—two of them near the surface of a cylindrical body, which enables the probe to distinguish different heat transfer characteristics in both the vertical and horizontal positions and one

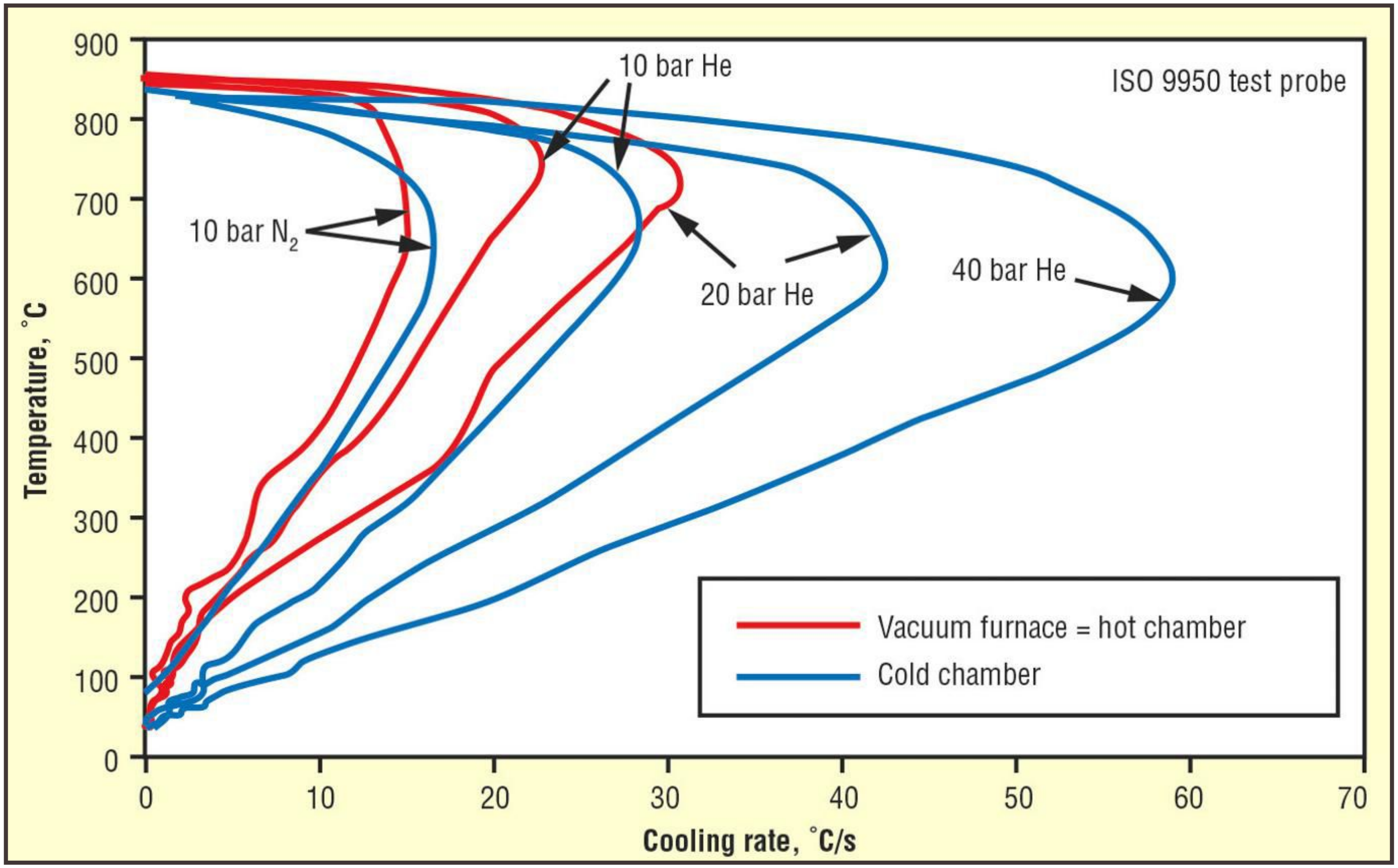


Fig. 6. Comparison of cooling rates in different quench chambers[10]

in the center. Thus, the influence of the "external" factors talked about above can be quantified. The measured quenching characteristics can be compared and data feed into a computer-based system for the prediction of hardenability, as well as determining the required quenching parameters (gas pressure, fan speed, gas type, water flow to the heat exchanger, etc.) for a specific workload during the entire quenching process. This enables the quench intensity of the gas to be precisely determined.

Another new area is that of controlled heat extraction (CHE) technology.[13] This area explores the possibility of automatically following a predetermined heat extraction (temperature-time) cycle during high-pressure gas quenching. The influence of the heat extraction dynamics on hardness distribution after quenching is being actively studied.

1. HPGQ process lasts much longer than quenching in liquid quenchants (assuming comparable dimensions and masses of the workpiece).
2. Influential quenchant parameters (e.g. medium temperature, pressure and agitation rate) cannot be changed during the quenching process when using liquid quenchants, but they can be automatically changed during high-pressure gas quenching. This versatility offers new possibilities to influence the heat extraction dynamics during the quenching process itself. Furthermore, with the temporary addition of sprayed cryogenic liquid (e.g. nitrogen), an instantaneously drop in the cooling gas temperature occurs with a substantial increase in the quenching intensity. This can have a drastic influence on hardness distribution throughout the part cross-section as well as on residual stresses and distortion after quenching.

Summary

High-pressure gas quenching has the inherent capability of limiting dimensional variation within a part and within a workload. The key is understanding the factors that influence gas quenching and holding constant as many of these variables as possible to achieve a balance between the speed of gas quenching and the uniformity (i.e. uniform heat extraction) of gas quenching. In this way, both repeatability (quality) and performance (productivity) will be achieved with an optimized microstructure.

High-pressure gas quenching technology has earned its rightful place as a valuable weapon in the arsenal of the heat treater. It should be applied in those applications where its advantages outweigh its disadvantages and, as with all technologies, the heat treater should have as complete an understanding as possible of the performance requirements of the product so as to evaluate the quench capability and application end-use.

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Understanding the Benefits of Austempering

Austempering is a unique process which both heat treaters and parts designers should understand; especially the factors that influence both process parameters and part properties. Let's learn more.

What is Austempering

Austempering is essentially an arrested quench process designed to produce a bainitic microstructure (Fig. 1) having properties that combine high hardness with toughness, resulting in a resistance to brittle fatigue. Austempering involves an isothermal transformation at a temperature below that of pearlite formation and above that of martensite formation.

Advantages include higher ductility at high hardness, increased strength and ductility at a given hardness, increased toughness (over 40 HRC), greater fatigue life and less distortion and cracking (especially in higher carbon steels). Limitations include section size and carbon level (for a given steel).

Material and Process Parameters

Several factors must be considered when specifying a steel for austempering. The following (Table 1) shows the influence of carbon and manganese content on maximum section size.[1]

Effect of chemistry on maximum section size

Steel grade	Max. section, in. (mm)	Ms temp., °F (°C)	%C	%Mn	Max. hardness, HRC
1050	0.125 (3.18)	655 (346)	0.48/0.55	0.60/0.90	48-50
1065	0.187 (4.75)	525 (274)	0.60/0.70	0.60/0.90	50-54
1084	0.218 (5.54)	395 (202)	0.80/0.93	0.60/0.90	55-57

For example, a properly austempered 1062 steel (0.63% C, 1.08% Mn) achieves a typical bainitic structure with hardness of 52.5 HRC. By comparison, a 1060 steel (0.62%C, 0.87% Mn) can have a mixed microstructure (martensite, bainite and intermediate transformation products) with the same hardness value.

Other factors to consider include part design parameters; post heat treatment operations; heat treat equipment; and process parameters such as time, temperature, furnace atmosphere and quench variables (bath chemistry, temperature, immersion time, and rate of heat transfer to the parts from the quench media).

Part thickness is an important variable in austempering. As hardenability of the material increases, section size can also be increased. High hardness and high ductility levels are limited by the carbon level in the steel (Table 2).[1]

Austempering can produce better mechanical properties than conventional quench and temper if the hardness is greater than 40 HRC (Table 3).[1]

Optimum hardness range for high toughness

Carbon content, %	Hardness, HRC
0.40	40-48
0.50	40-52
0.60	40-54
0.70	40-56
0.80	40-58

For a hardness lower than 40 HRC, distortion and other factors become the prime consideration. For example, impact strength can be increased by as much as 300%. Some materials and part shapes are more adaptable to one type of equipment than another (salt-to-salt or atmosphere-to-salt). The factors mentioned above usually are specified by the part designer.

Transformation temperature for various hardness/strength levels

Hardness, HRC	Tensile strength, ksi (MPa)	Approx. transformation temperature, °F (°C)
40	180 (1,240)	750 (400)
45	210 (1,450)	650 (345)
50	240 (1,655)	600 (315)
55	280 (1,930)	520 (270)
58	>300 (>2,070)	400 (200)

Austenitizing conditions that influence final properties or part functionality are heating method, temperature and time at temperature. Generally, published austenitizing temperature ranges are used. Where high hardness and ductility are required, it is desirable to get all carbides into solution during austenitizing. Where wear resistance or minimum distortion are required, the goal is to dissolve only enough carbides to meet the hardness and obtain a bainitic microstructure, and to austenitize at the lowest possible temperature and quench into the highest possible bath temperature.

Heating parts in a salt bath results in some decarburization. In this case, carbon correction should be made prior to austenitizing.

Fig. 1. Austempered AISI 1055

Salt-bath austenitizing is best suited for minimum distortion, especially of long, slender parts. Austenitizing in a furnace under protective atmosphere (e.g., endothermic) allows controlling carbon content in the part using conventional techniques (oxygen probe, infrared, dew point), and is better suited for small, symmetrical parts that can be hopper fed, and which if treated in a salt bath are prone to carrying austenitizing salts into the quench. Austenitizing in a controlled atmosphere typically allows using lower hardenability steels having 5-10% thicker sections than salt bath processing.

Salt Quenching

The desired strength (hardness) level governs the salt quench-bath temperature. Immersion time in the bath is a function of the desired part hardness, material chemistry and section thickness (Table 4). In general, immersion time decreases as the transformation temperature increases, and transformation time at the same transformation temperature increases with increasing steel carbon content.

Factors that favorably influence heat transfer from parts in the salt quench bath include a long free fall through the quench medium and/or agitation of quench medium or parts and water additions to the quench medium. Water additions to the salt bath improve heat transfer from the parts at times when steel of marginal hardenability or large sections are used.

Unfavorable factors are contamination of the quench bath (soot, scale, high heat salt carryover) and loss of water.

Rules of Thumb

The following rules are recommended for austempering to a given hardness value.

Table 1 Variation in salt bath temperature variation to obtain specific hardness (fasteners)

Part thickness, in. (mm)	SAE 1064/1065 @ 46-48 HRC	SAE 1064/1065 @ 42-44 HRC	SAE 1064/1065 @ 38-41 HRC
0.006-0.015 (0.152-0.381)	630-640°F (330-340°C)	630-635°F (330-335°C)	730-740°F (390-395°C)
0.016-0.025 (0.406-0.635)		635-640°F (335-340°C)	
0.026-0.035 (0.660-0.899)	635-645°F (335-340°C)	640-645°F (338-341°C)	740-750°F (395-400°C)
0.036-0.045 (0.914-1.143)		645-650°F (340-345°C)	
0.046-0.055 (1.168-1.397)	640-650°F (340-345°C)	650-655°F (343-346°C)	755-765°F (400-405°C)
0.056-0.065 (1.422-1.651)		655-660°F (345-350°C)	

Rule 1: Achieve a 100% bainitic microstructure.

- The section size that will through harden is a function of both the material hardenability and the equipment being used (e.g.,quench agitation, water addition, etc.).
- Critical diameter calculations are required (critical diameter is the diameter of a cylindrical bar that just quenches to the desired microstructure at the center).
- Material and process factors should be empirically determined.
- Raise the austenitizing temperature toward the upper end for a steel having borderline hardenability.

Rule 2: Operate the quench bath temperature at M_s +25°F (+ 14°C).

- Calculate the martensite start (M_s) temperature using commercially available methods.
- A higher quench bath temperature produces lower strength and hardness; a practical salt-

temperature equipment limitation is ~750°F (400°C).

Rule 3 Maintain a minimum time of 20 minutes in the quench (under the salt).

- Carbon content is the single greatest influence on time (e.g., a 0.50%C steel part has a 2-4 HRC difference in surface hardness compared with an 0.80%C part).

Rule 4 Use a slightly carburizing atmosphere (i.e., high carbon bainite is desirable).

- Decarburized areas require a higher Ms temperature.
- It is better to avoid pearlite than martensite.

Rule 5 Check your results.

- Use 10% sodium metabisulfite etchant to selectively color etch to differentiate between martensite and bainite; martensite is tinted brown and banite tinted blue.
- Use Villela's reagent to resolve bainite.
- Use a 2% nital (fresh solution) etchant to determine microstructural constituents; bainite has straw color while pearlite appears as little specs of blue, black or gray (due to different etching response).

Summary

Austempering is an oft times overlooked process which offers great value where parts require a combination of high hardness and high ductility. Austempering is also of great benefit for parts requiring less heat treat distortion or dimensional variation and when breakage must be kept to a minimum.

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What Do We Really Know About Tempering?

Tempering, also known as "drawing", is one of the most common heat-treatment processes – and one that is all too often taken for granted. We all know it's important, yet we spend little time focused either on the process or on the equipment in which it is performed. Let's learn more.

What is Tempering

When we temper a part, we tend to focus on a single parameter – hardness as a determination of the success or failure of the tempering operation. We must broaden our perspective and understand that tempering is done to "toughen" the steel that has been previously hardened or normalized. The as-quenched microstructure of steel being primarily martensite is highly unstable and in a strain-induced state. The resulting change of martensite during tempering into a mixture of cementite (Fe_3C) and ferrite typically results in an increase in grain size and a decrease in volume as a function of increasing tempering temperature.

Precipitation-hardened alloys – including many grades of aluminum and superalloys – are tempered so as to precipitate intermetallic particles that strengthen the metal. Tool and high-speed steels are often tempered multiple times to achieve proper hardness while transforming retained austenite first to untempered martensite and on subsequent runs to tempered martensite. What is important to remember in all of this is that tempering is the modification of the hardened microstructure toward an equilibrium state.

Tempering temperature, time at temperature, cooling rate from tempering temperature and steel chemistry are the variables associated with tempering that affect the mechanical properties and microstructure of the finished part. Changes to the microstructure by tempering typically decrease hardness and strength (tensile and yield) while increasing ductility and toughness. Tempering results in an increase in softness, malleability, impact resistance and improved dimensional stability.

A good rule to remember is that all steel should be tempered soon after being removed from the quench and before it is completely cold. Failure to temper correctly may lead to a myriad of performance problems such as premature failure or shorter than normal service life. A common call-out in various specifications is to "temper immediately," which is

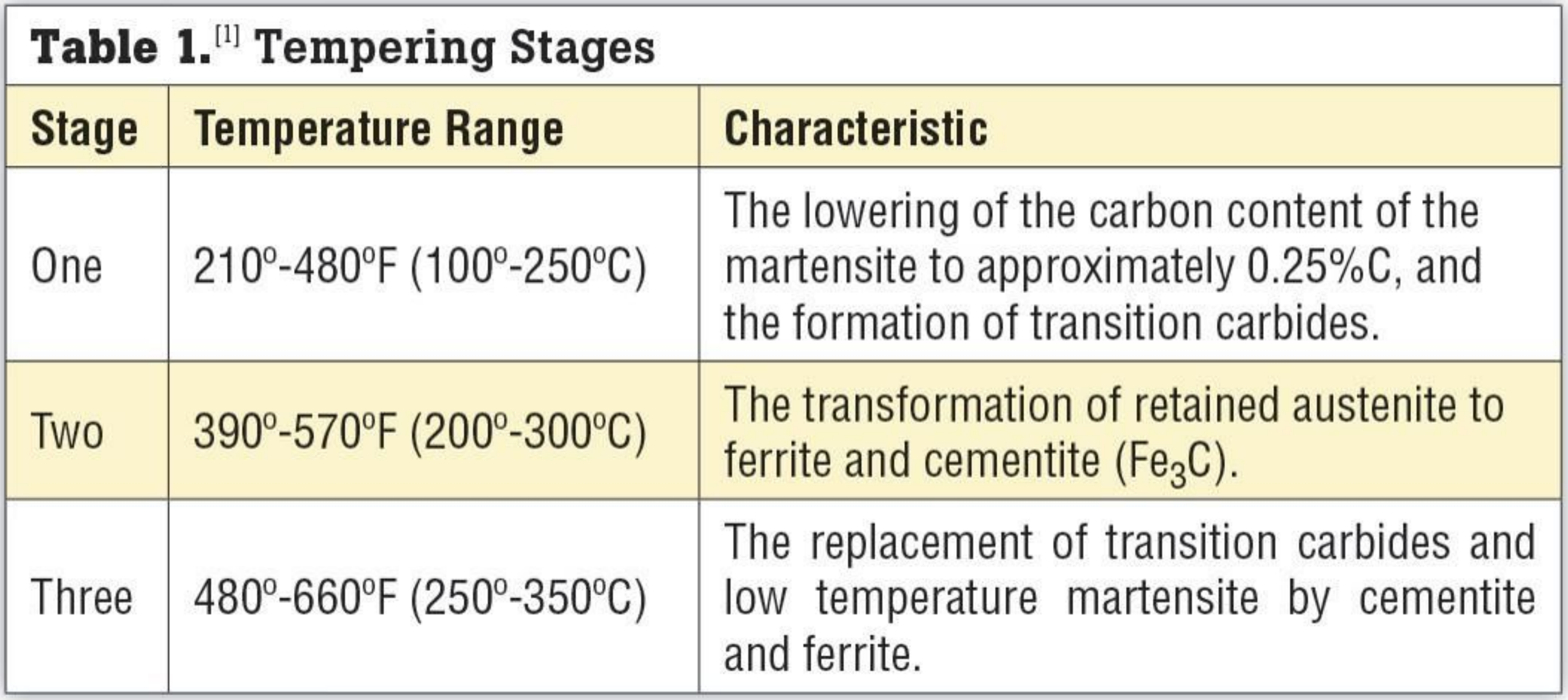

Table 1.[1] **Tempering Stages**

Stage	Temperature Range	Characteristic
One	210º-480ºF (100º-250ºC)	The lowering of the carbon content of the martensite to approximately 0.25%C, and the formation of transition carbides.
Two	390º-570ºF (200º-300ºC)	The transformation of retained austenite to ferrite and cementite (Fe_3C).
Three	480º-660ºF (250º-350ºC)	The replacement of transition carbides and low temperature martensite by cementite and ferrite.

subject to broad interpretation throughout the industry. For materials such as 4340, tempering must take place within 15 minutes of removal from the quench to avoid quench cracking. For high-speed steels, the tools should normally be allowed to cool to 120°-150°F (50°-65°C) and then tempered, but it is all too common to find loads sitting and waiting for a temper furnace for two or more hours.

Tempering is always performed below the lower critical temperature (A_1) of the steel, which differentiates tempering from such processes as annealing, normalizing and hardening. When hardened steel is reheated, tempering effects start to occur as low as 212°F (100°C) and accelerate as

Table 2. Steel color changes as a function of tempering temperature

Temperature °F (°C)	Temper Color
400 (205)	Faint straw yellow
425 (220)	Light straw yellow
440 (225)	Straw yellow
475 (245)	Deep straw yellow/light brown
500 (260)	Orange/brown
520 (270)	Bronze/dark brown
525 (275)	Light purple
545 (285)	Purple/peacock
560 (295)	Dark blue
590 (310)	Full blue
620 (325)	Gray
660 (350)	Gray-purple
705 (375)	Gray-blue
750 (400)	Dull gray
>750 (400)	Black*

*Oxide adherence issues occur above 750°F (400°C) with surfaces appearing first as a velvet-textured oxide, progressing to loose, flaky scale.

the temperature increases. By selecting a definite tempering temperature, you can predetermine the resulting hardness and strength. One caution is the avoidance of issues such as temper embrittlement (for more detail, see "The Embrittlement Phenomena in Hardened & Tempered Steel," Industrial Heating, October 2006).

Fig. 1. Example of tempering color variation (support plate)

The minimum temperature time for tempering should be one hour. A good "rule of thumb" for furnace or oven tempering is that if the part is more than 1 inch (25 mm) thick, increase the time by one hour for each additional inch (25mm) of thickness or fraction thereof.

Stages of Tempering

Tempering is said to occur in three distinct stages in overlapping temperature ranges (Table 1). The precipitation of finely dispersed alloy carbides

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responsible for secondary hardening (in highly alloyed steels) is sometimes referred to as the fourth stage of tempering. It is important to know what is happening to the part microstructure at the tempering temperature you select.

Considerations in Tempering Equipment

When designing a tempering process, consideration must be given to the type and condition of the tempering equipment. In particular, airflow and temperature uniformity play a critical role. Tight temperature uniformity – typically ± 10°F (± 5.5°C) – is required throughout the load with ± 5°F (± 2.75°C) preferred, especially for high-speed and precipitation-hardening steels. The ability to have a rapid heating rate will shorten overall cycle time.

Various forms of self-tempering and accelerated tempering via induction or ultrahigh air convection ovens and furnaces have shown promise in a number of applications [2,3]. Although soak times and temperatures are typically fixed by steel chemistry, substantial reductions in processing times can be achieved by accelerating the heat-up time. This can be accomplished by designing more efficient heat transfer between the heated atmosphere and the load using high-speed convective, turbulent flow patterns.

For example, it was found that effective

tempering of axle shafts after induction hardening was dependent on actual heat-transfer rates and component temperature rather than on time at a specified temperature. This study achieved the following results with a savings of over 80% in cycle time (4 hours to 36 minutes): equivalent post-temper hardness at the bearing journal surfaces; equivalent hardness and residual stress profiles within the shaft; equivalent yield and rupture strengths; and equivalent reverse torsional and rolling bending-fatigue life.

Temper Colors

The use of temper color is one method of not only visually determining if a part has been exposed to the proper tempering temperature but to check if all parts in a given load reached a uniform temperature (Fig. 1). When steel is heated and exposed to air (or an oxidizing atmosphere) for a short period of time, it will change color due to the presence of a thin, tightly adhering oxide. The temper color and thickness of the oxide layer varies with both time and temperature (Table 2). Different steel chemistries also result in slight color variations. The colors produced are typically not uniform because of surface condition and fluctuation of temperature. To see the colors clearly, you must turn the part from side to side and have good lighting. Natural lighting is always best.

Summary

Tempering – because it is often the last heat-treat operation – is considered by most to be relatively simple and straightforward. The heat treater, however, must remember that it is a complex process in which all of the process and equipment variability must be carefully controlled.

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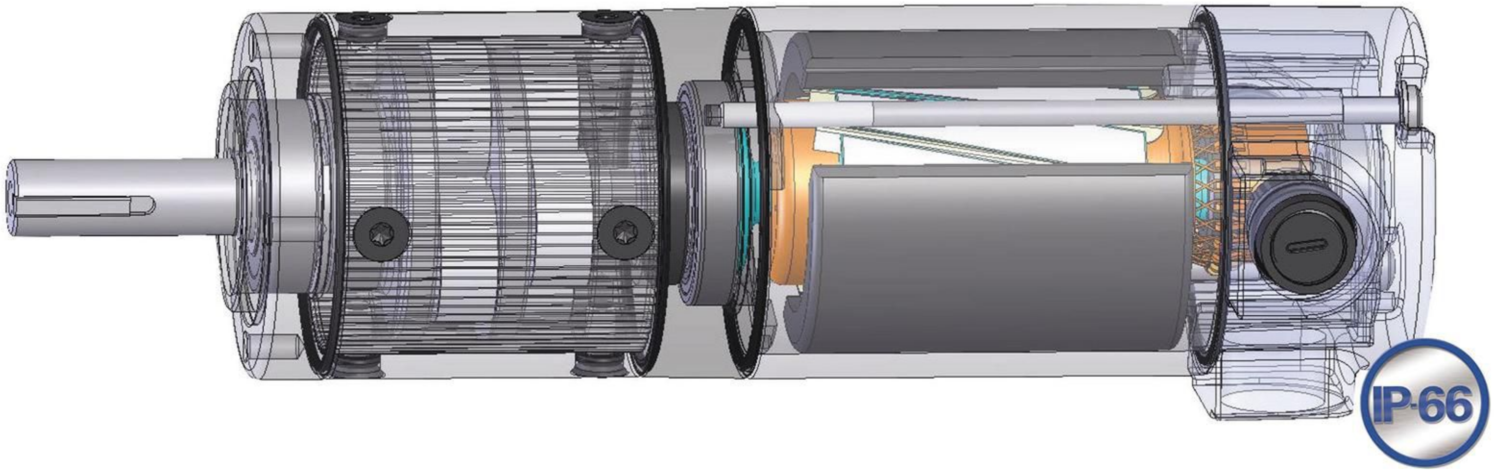
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Gear Materials and their Heat Treatment

Gears play an essential role in the performance of many products that we rely on in our everyday lives. When we think about gears we generally separate them into two categories – motion-carrying and power-transmission. Let's learn more.

Motion-carrying gears are generally nonferrous or plastics, while load-bearing power-transmission gears (Fig. 1) are usually manufactured from ferrous alloys and are intended for heavy-duty service applications.

Gear Materials

Power-transmission gears involve a wide variety of steels and cast irons. In all gears, the choice of material must be made only after careful consideration of the performance demanded by the application end-use and total manufactured cost, taking into consideration such issues as pre- and post-machining economics.

Key design considerations require an analysis of the type of applied load – whether gradual or instantaneous – and the desired mechanical properties, such as bending fatigue strength or wear resistance, all of which will define core strength and heat-treating requirements.

It is important for the designer to understand that each area in the gear-tooth profile sees different service demands. Consideration must be given to the forces that will act on the gear teeth, with tooth bending and contact stress, resistance to scoring and wear, and fatigue issues being paramount. In the root area, for example, good surface hardness

Fig. 1. Typical off-highway power-trasmission gears

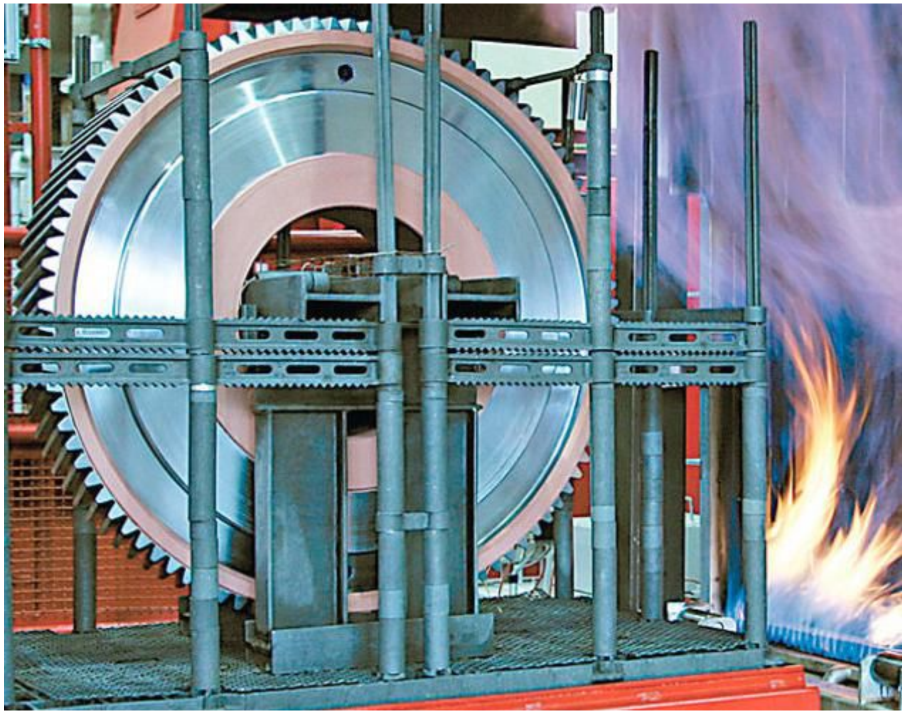

Fig. 2. Atmosphere carburizing of large gears (photograph courtesy of Aichelin USA)

Fig. 3. Typical commercial heat-treat load of gears for vacuum carburizing. (Photo courtesy of Midwest Thermal-Vac)

and high residual compressive stress are desired to improve endurance or bending fatigue life. At the pitch diameter, a combination of high hardness and adequate subsurface strength are necessary to handle contact stress and wear and to prevent spalling. As an example, some of the factors that influence fatigue strength are:

Hardness distribution, a function of:

- Case hardness
- Case depth
- Core hardness

Microstructure, a function of:

- Retained-austenite percentage
- Grain size
- Carbide size, type and distribution
- Non-martensitic phases

Defect control, a function of:

- Residual compressive stress
- Surface finish and geometry
- Intergranular toughness

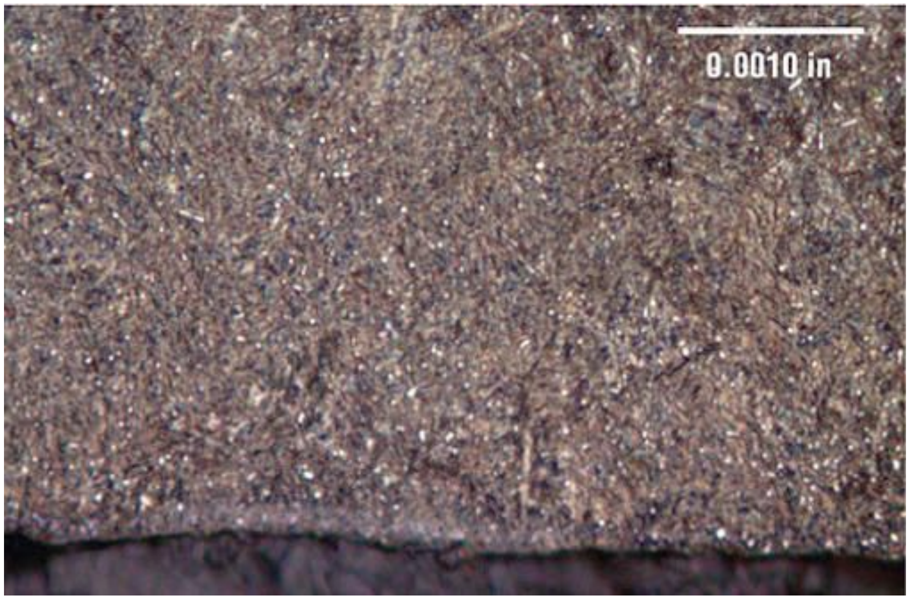

Fig. 4. Typical vacuum carburizing microstructure - M50NiL: 0.090-inch effective cast depth (50 HRC); 61HRC @ 0.035 inch; <5% retained austenite; 500X (Photograph courtesy of Midwest Thermal-Vac)

Thus, in the total manufacturing scheme, a synergistic relationship must exist between the material selection process, engineering design and manufacturing (including heat treatment). A balance of the priorities in each discipline must be reached in order to achieve the optimization necessary for the ultimate performance of the gear design. This is often not an easy task.

Atmosphere Heat Treatment Methods

Various atmosphere heat treatment methods are used for most types of gears including per-hardening steps (e.g. annealing, normalizing, stress relief) and hardening processes (e.g. neutral or through hardening and case hardening).

Neutral or through hardening refers to heat treatment methods that do not produce a case. Examples of commonly through hardened gear steels are SAE grades 1045, 4130, 4140, 4145, 4340, and 8640. It is important to note that hardness uniformity should not be assumed throughout the gear tooth. Since the outside of a gear is cooled faster than the inside, there will be a hardness gradient developed. The final hardness is dependent on the amount of carbon in the steel. The depth of hardness depends on the hardenability of the steel.

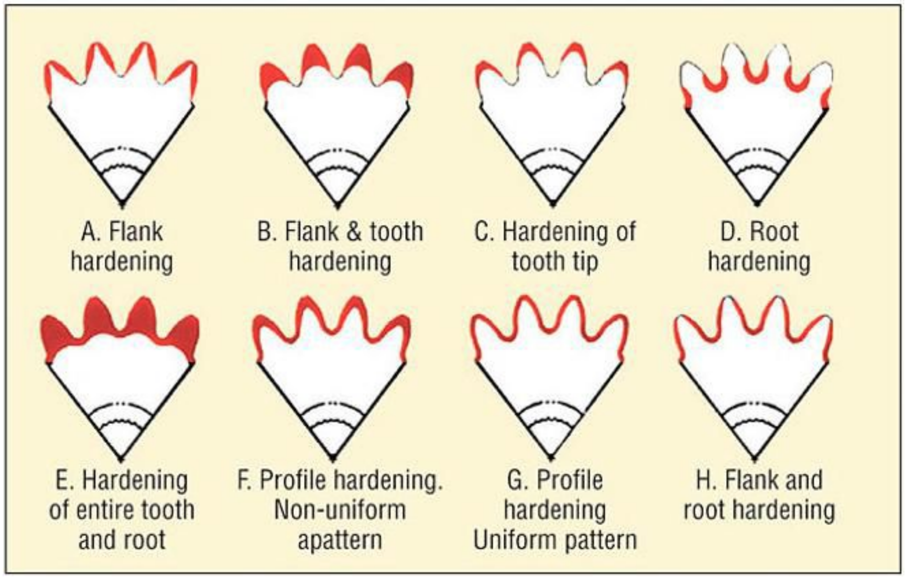

Fig. 5. Patterns produced by induction hardening [11]

Through hardening can be performed either before or after the gear teeth are cut. When gear teeth will be cut after the part has been hardened, machinability becomes an important factor on final hardness. The hardness is achieved by heating the material into the austenitic range, typically 815ºC - 875ºC (1500ºF - 1600ºF), followed by quenching and tempering.

By contrast, case hardening is used to produce a hard, wear resistant case, or surface layer, on top of a ductile, shock resistant interior, or core. The idea behind case hardening is to keep the core of the gear tooth at a level around 40 HRC to avoid tooth breakage while hardening the outer surface to increase pitting resistance.

Carburizing

Atmosphere carburizing is the most common of the case-hardening methods in use today and can handle a diverse range of part sizes and load configurations (Fig. 2). In general, a properly carburized gear will be able to handle somewhere between 30-50% more load than a through-hardened gear. Examples of commonly carburized gear steels include SAE grades 1018, 4320, 5120, 8620 and 9310 as well as international grades such as 20MnCr5, 17CrNiMo6, 18CrNiMo7-6 and 20MoCr4.

Atmosphere carburizing is typically performed in the temperature range of 870-955°C (1600-1750°F), although temperatures approaching 1010°C (1800°F) are used for deep-case work. Carburizing case depths vary over a broad range, typically 0.13-8.25 mm (0.005-0.325 inches).

Carbonitriding

Carbonitriding is a modification of the carburizing process, not a form of nitriding. This modification consists of introducing ammonia into the carburizing atmosphere in order to add nitrogen to the carburized case as it is being produced. Examples of gear steels

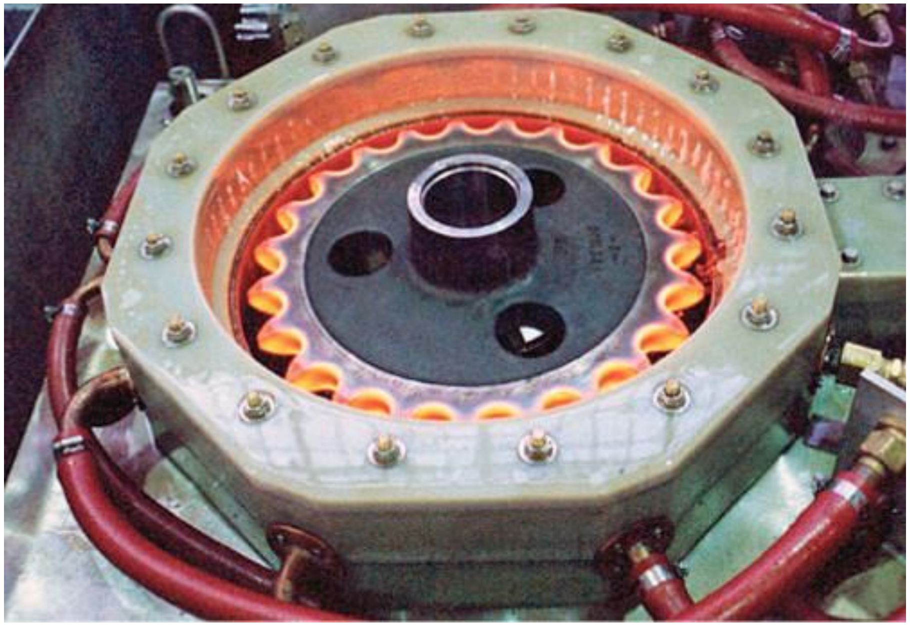

Fig. 6. Typical single-shot induction hardening operation (Photograph courtesy of Ajax-Tocc Magnethermic)

that are commonly carbonitrided include AISI 1018, 1117 and 12L14.

Typically, carbonitriding is done at a lower temperature than carburizing – between 700-900°C (1300-1650°F) – and for a shorter time. Combine this with the fact that nitrogen inhibits the diffusion of carbon and what generally results is a shallower case than is typical for carburized parts. A carbonitrided case is usually between 0.075-0.75 mm (0.003-0.030 inches) deep.

Nitriding

Nitriding is another surface-treatment process that has increasing surface hardness as its objective. One of the appeals of this process is that rapid quenching is not required. Hence, dimensional changes are kept to a minimum. It is not suitable for all gear applications. One of its limitations is that the extremely high surface-hardness case produced has a more brittle nature than that produced by the carburizing process. Despite this fact, nitriding has proved to be a viable alternative in a number of applications. Examples of commonly nitrided gear steels include SAE 4140, 4150, 4340 and Nitralloy 135M.

Nitriding is typically done in the 495-565°C (925-1050°F) temperature range. Case-depth and case-hardness properties vary not only with the duration and type of nitriding being performed but also with steel composition, prior structure and core hardness. Typically, case depths are between 0.20-0.65 mm (0.008-0.025 inches) and take from 10 to 80 hours to produce.

Nitrocarburizing

Nitrocarburizing is a modification of nitriding, not a form of carburizing. In the process, nitrogen and carbon are simultaneously introduced into the steel while it is in a ferritic condition (i.e. at a temperature

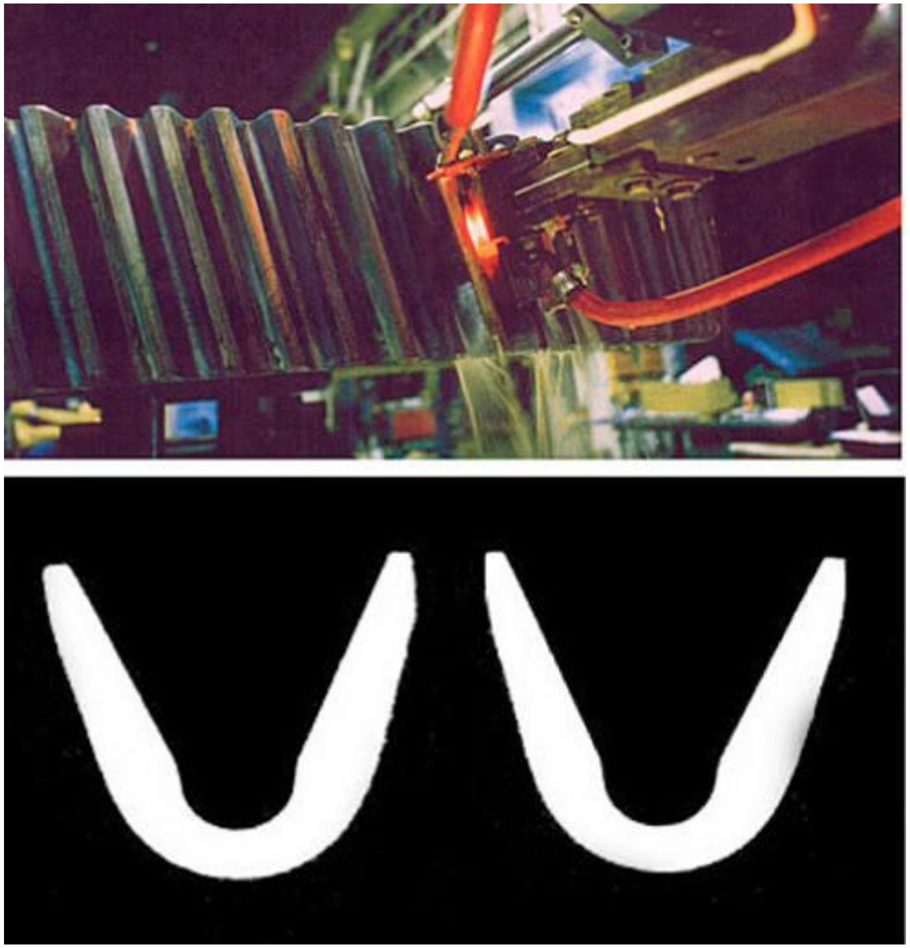

Fig. 7. (a) Tooth-by-tooth hardening of a helical gear and (b) resultant tooth profile (Photograph courtesy of Ajax-TOCCO-Magnethermic)

below that at which austenite begins to form during heating). A very thin “white” or “compound” layer is formed during the process as well as an underlying “diffusion” zone. Like nitriding, rapid quenching is not required. Examples of gear steels that are commonly nitrocarburized include SAE grades 4140, 5160, 8620 and certain tool steels.

Nitrocarburizing is normally performed at 550-600°C (1025-1110°F) and can be used to produce a 58 HRC minimum hardness, with this value increasing dependent on the base material. White-layer depths range from 0.0013-0.056 mm (0.00005-0.0022 inches) with diffusion zones from 0.03-0.80 mm (0.0013-0.032 inches) being typical.

Vacuum Heat Treatment Methods

Vacuum processing can be used for most of the atmosphere treatments mentioned above, including carburizing (Fig. 3). Progress continues to be made in low-pressure carburizing (LPC) of advanced applications in the aerospace, automotive, off-highway and motorsports markets,[1,3] particularly in teh development of carburizing cycles for high-performance materials (Table 1).

Effective case depths for most grades range from 2.0-3.0 mm (0.080-0.120 inches) without significant sacrifice of microstructure (Fig. 4). Furnace variables such as temperature uniformity (±3°C or ±5°F), control-of-cycle parameters for carbide control (flow rate, pressure, hydrocarbon type) and surface-

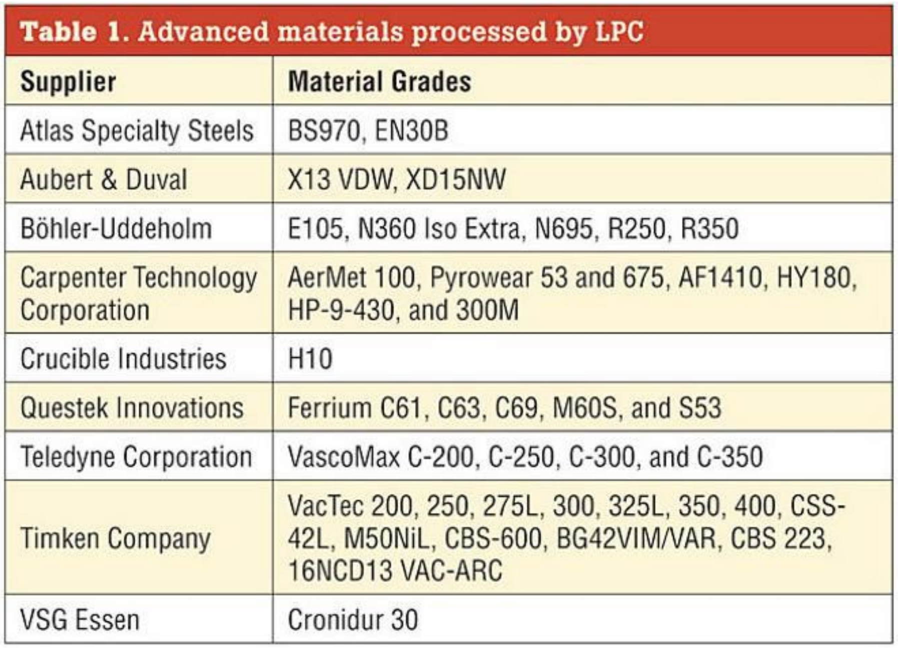

Table 1. Advanced materials processed by LPC

Supplier	Material Grades
Atlas Specialty Steels	BS970, EN30B
Aubert & Duval	X13 VDW, XD15NW
Böhler-Uddeholm	E105, N360 Iso Extra, N695, R250, R350
Carpenter Technology Corporation	AerMet 100, Pyrowear 53 and 675, AF1410, HY180, HP-9-430, and 300M
Crucible Industries	H10
Questek Innovations	Ferrium C61, C63, C69, M60S, and S53
Teledyne Corporation	VascoMax C-200, C-250, C-300, and C-350
Timken Company	VacTec 200, 250, 275L, 300, 325L, 350, 400, CSS-42L, M50NiL, CBS-600, BG42VIM/VAR, CBS 223, 16NCD13 VAC-ARC
VSG Essen	Cronidur 30

Table 1. Advanced materials processed by LPC

carbon selection (0.42-1.05% C) can produce case uniformities of ±0.05 mm (±0.002 inches). Where permitted, the range of carburizing temperatures now includes the use of high-temperature techniques. All of these advanced materials required extensive development testing to produce custom-designed recipes to optimize cycle parameters. Also, quenching methods[10] have improved, allowing us to achieve desired core properties with quenching-paramter selection (high-pressure gas or oil) for distortion-sensitive and distortion-prone part geometrics.[4,6]

Induction Hardening Methods

Various methods of hardening by use of applied energy are utilized in the manufacture of gears, including flame hardening, laser surface hardening and induction.

Of the various types of applied-energy processing, induction hardening is the most common. Induction heating is a process that uses alternating electrical current to induce a magnetic field, causing the surface of the gear tooth to heat. The area is then quenched, resulting in an increase in hardness within the heated area. This process is typically accomplished in a relatively short time. The final desired gear performance characteristics are determined not only by the hardness profile and stresses but also by the steel's composition and prior microstructure. External spur and helical gears, bevel and worm gears, racks and sprockets are commonly induction hardened. Typical gear steels include SAE grades 1050, 1060, 1144, 4140, 4150, 4350, 5150 and 8650 to name a few.

The hardness pattern produced by induction heating (Fig. 5) is a function of the type and shape of inductor used as well as the heating method. Quenching or rapidly cooling the workpiece can be accomplished by spray or submerged quench. The media typically used for the quench is a water-based polymer. The severity of this quenchant can be controlled by the polymer's concentration. Cooling rates are usually somewhere in between what would be obtained from pure water and oil. In some unusual situations, compressed air is used to quench the workpiece.

The most common methods for hardening gears and sprockets are by single shot (Fig. 6) or the tooth-by-tooth method (Fig. 7a). Single shot often requires large kW power supplies but results in short heat/quench times and higher production rates. This technique uses a copper inductor (coil) encircling the workpiece. An inductor, which is circumferential, will harden the teeth from the tips downward.

While the single-shot method is acceptable for splines and some gearing, the larger heavier-loaded gears (where pitting, spalling, tooth fatigue and endurance are an issue) need a hardness pattern that is more profiled like those produced with carburizing. This type of induction hardening is called tooth-by-tooth hardening. This method is limited for gear tooth sizes up to 5 or 6 DP using frequencies from 2 to 10 kHz and about 10 DP using a range of 25 to 50 kHz. The lower the frequency, the deeper the case depth. This is a slow process due to the number of teeth and index times, and it is usually reserved for gears and sprockets that are too large to single shot due to power constraints. The process involves heating the root area and side flanks simultaneously while cooling each side of the adjacent tooth to prevent temper-back on the backside of each tooth (Fig. 7b). The induction system moves the coil at a pre-programmed rate along the length of the gear. The coil progressively heats the entire length of the gear segment while a quench follower immediately cools the previously heated area. The distance from the coil to the tooth is known as coupling or air-gap. Any variation in this distance can yield variation in case depth, hardness and tooth distortion.

The gear is indexed after each tooth has been hardened, often skipping a tooth. This requires at least two full revolutions in the process to complete the hardening of all teeth. Straight, spur and helical gears up to 5335 mm (210 inches) and 6800kg (15,000 pounds) have been processed with this method. The entire process yields a repeatable soft tip of the tooth with hard root and flank. In other applications, the tip and both flanks can be hardened simultaneously and yield a soft root.

Summary

Today, the design engineer has the good fortune of being able to choose from a number of heat-treatment technology choices for any given type of gear material and gear design. The secret to success is to understand the advantages and limitations of each technology and take these into consideration when determining the overall cost of gear manufacturing.

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Case Studies: Creating Salvage Cycles for Carburized Gears

Case studies provide valuable insights into the heat-treat process. They expand our knowledge about the investigative methods needed to overcome specific problems. Such is the case when trying to determine if a material or quenching problem existed on a group of SAE 8822 carburized gears. Let's learn more.

The Problem

A manufacturer of AGMA Class-3 gears wanted to switch raw material from bar stock to forgings. During qualification trials, normal inspection practices using a test piece (an actual gear segment) produced from bar stock passed case depth and hardness (surface and core) requirements. However, validation testing on an actual part (produced from a forging) was found to have a variation in hardness in the root of the gear teeth. It was necessary to determine the root cause of the problem and to try to design a salvage cycle for the parts already produced.

The Investigation

The investigation into the cause of the root-hardness variation in these gears began by gathering information about the raw material as well as the heat-treatment practices. Testing followed on both gears made from bar stock and from forgings (Fig. 1). Both parts had been carburized to a targeted effective case depth of 0.070 inch (flank) and 0.035 inch minimum (root).

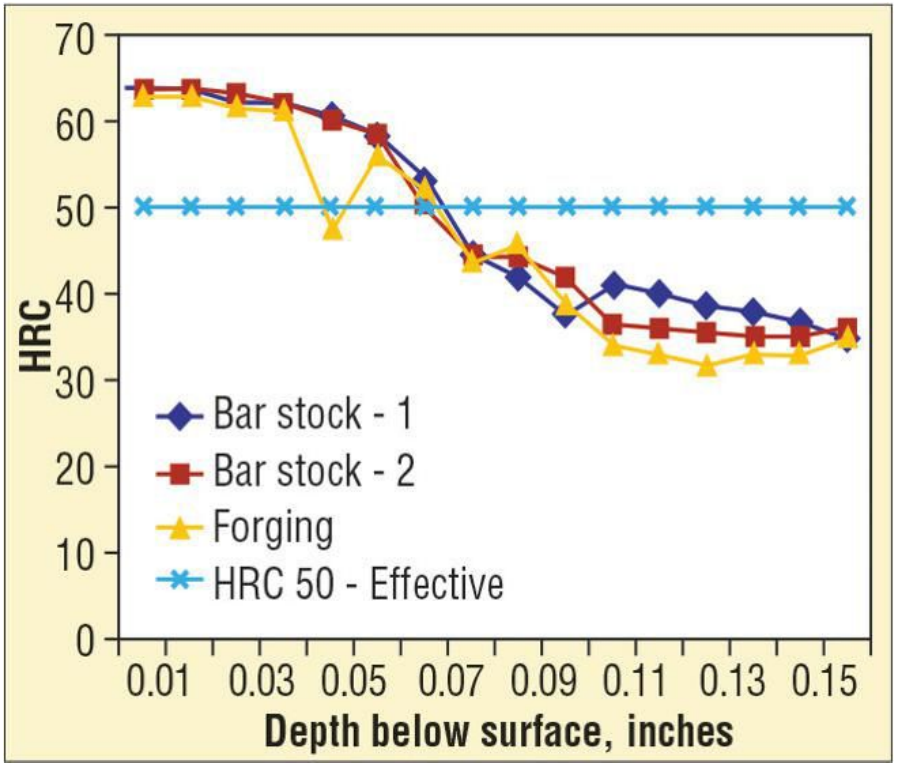


Fig. 1. Hardness profile test results for bar stock and forgings

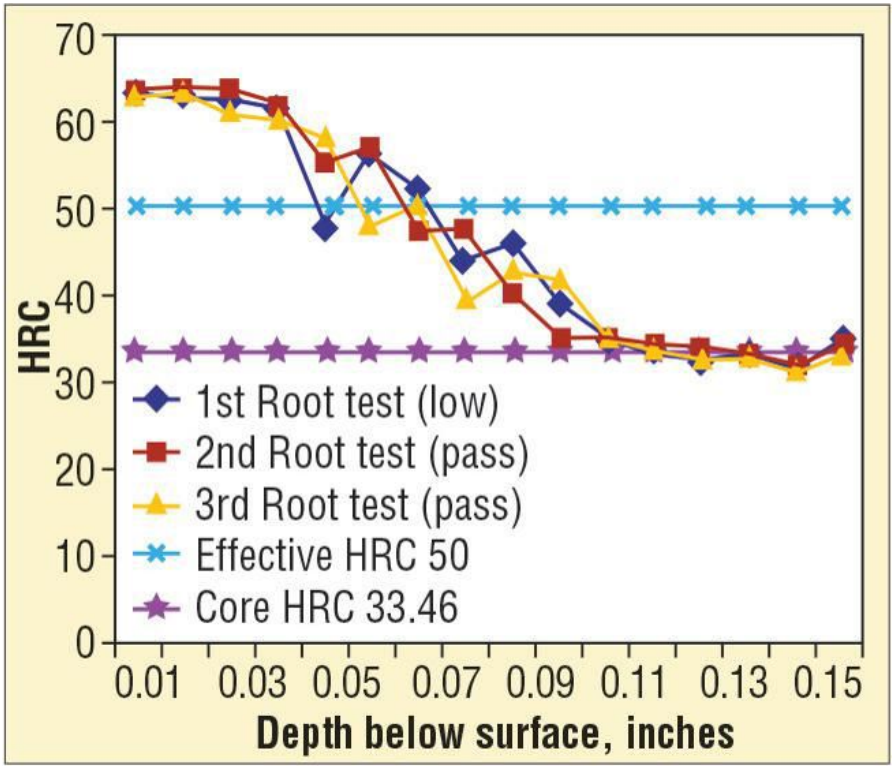


Fig. 2. Example of forging root hardness at three test locations

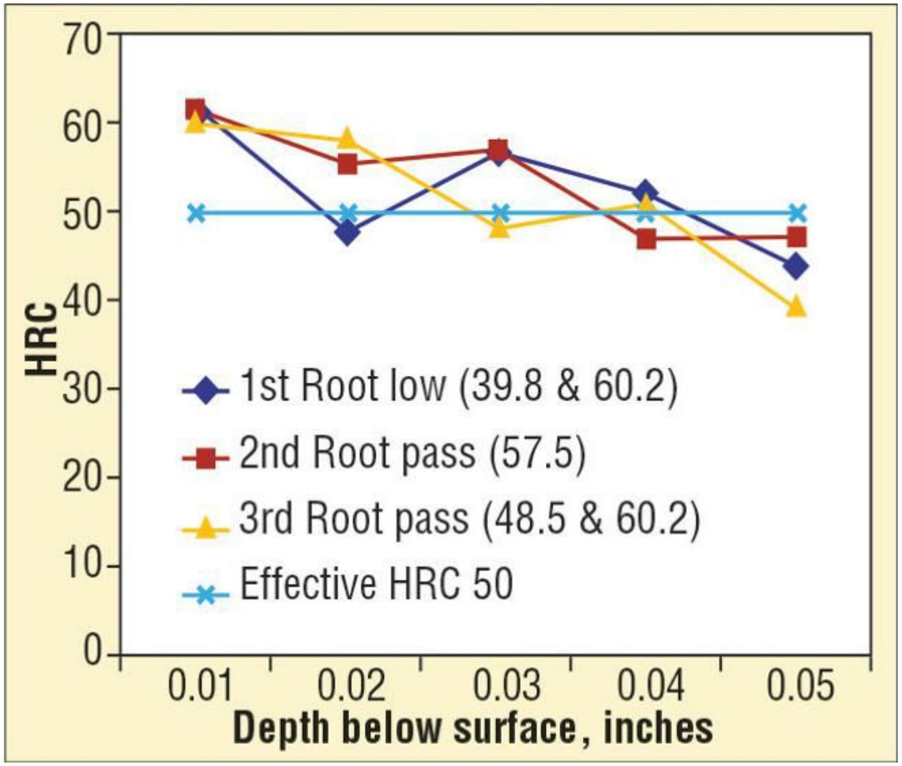


Fig. 3. Hardness of the forging at three root locations

The material certification sheets for the bar stock and forgings indicated that although the chemistry was within specification limits for an SAE 8822 material, the forgings had lower hardenability values (J4 and J8 positions). A comparison of the carburizing profiles by microhardness methods supported this finding, as the bar stock responded very differently than the forgings.

Several "dips" in hardness were consistently observed in the forgings (between the fourth and sixth indentations and between the ninth and 11th position) and were considered abnormal. Typically, this is an indicator of a significant change in the microstructure. However, the effective case depth (50 HRC) of both samples appeared to be essentially the same. This suggested additional testing be conducted in the root area of the forged gears (Fig. 2).

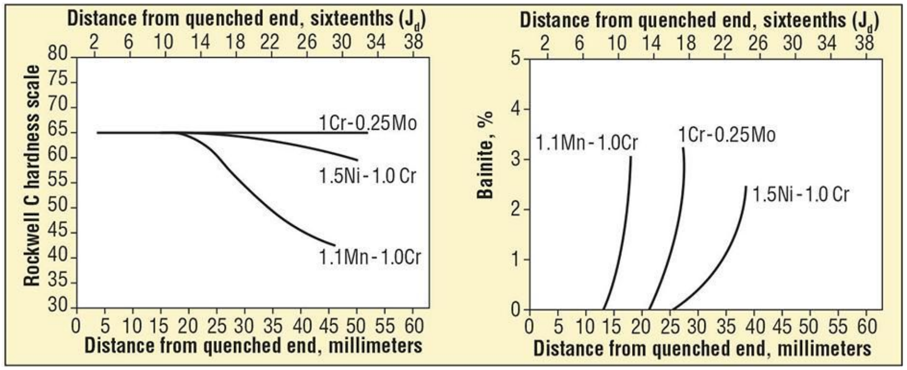


Fig. 4. Case hardenability data and corresponding bainite profile for three steels[1]

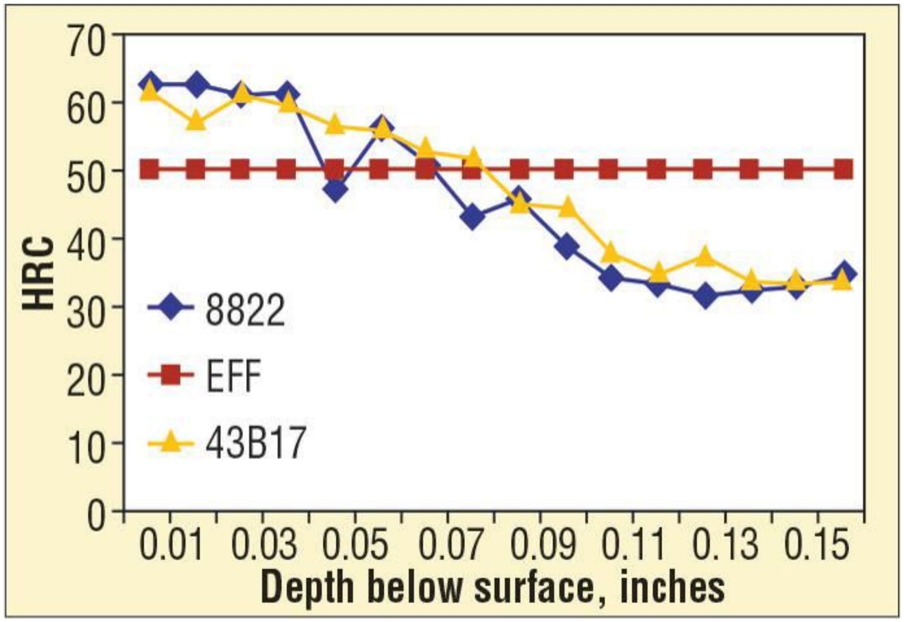


Fig. 5. Root effective case depth 8822 and 43B17 material – oil quenched

These results indicated that the effective case depth (50 HRC) of the forging is very dependent on the location sampled. It should be noted that the selection of the test location was random and done on a polished (not etched) sample. The additional test locations were adjacent to the original selected test zone. Focusing in on a depth of 0.030-0.040 inch (Fig. 3) provided more evidence.

First Bainite Test

In order to help determine the severity of the problem, parts were water quenched in order to obtain maximum case hardness in accordance with SAE J1975 (Case Hardenability of Carburized Steels). “Distance to the first appearance of bainite” (DFB) is defined as the location on water quenching where the hardness just begins to drop off.

DFB in the Carburized Case

For the greatest resistance to impact, data suggest that the as-quenched microstructure must be substantially free from bainite or pearlite.[2] The presence of very small amounts of bainite in the case has also been reported to reduce fatigue resistance.[3]

A detailed study of the occurrence of bainite in carburized end-quench hardenability specimens[4] involved some 81 alloys carburized at 925°C

(1700°F), cooled to 845°C (1550°F) and end-quenched. Case and hardness profiles were determined at a depth corresponding to 0.90% carbon. The amount of bainite as a function of distance from the quenched end of the bar was determined (Fig. 4). It should be noted that one cannot detect the presence of small amounts of bainite from hardness data alone.

Multiple regression analysis developed an empirical relationship for predicting DFB from composition (Equations 1a, 1b). The regression equations appear below, and they are valid at the 0.9% C level in the case for steels containing 0.5-1.1% Mn, 0-1.5% Ni, 0-1.0% Cr, and 0-0.5% Mo. Alloy contents are entered in weight percent:

(1a) DFB (in millimeters from the quenched end) = $54.79(\text{Mo}^2) + 6.4(\text{Cr}^2) - 76.1(\text{MnNi}) + 118.8\,(\text{MnMoNi}) + 106.1\,(\text{MnMoCr}) + 15.5(\text{MnNiCr}) + 52.9(\text{MoNiCr}) + 1.18$

(1b) DFB (in sixteenths of an inch from the quenched end) = $35.4(\text{Mo}^2) + 4.0(\text{Cr}^2) - 49.7(\text{MnNi}) + 74.8\,(\text{MnMoNi}) + 66.9(\text{MnMoCr}) + 9.8(\text{MnNiCr}) + 33.3(\text{MoNiCr}) + 0.7$

Table 1. Water-quench results

Location	Requirement (HRC)	Actual (HRC)	Material
J4	38	40	Bar stock
		34	Forging

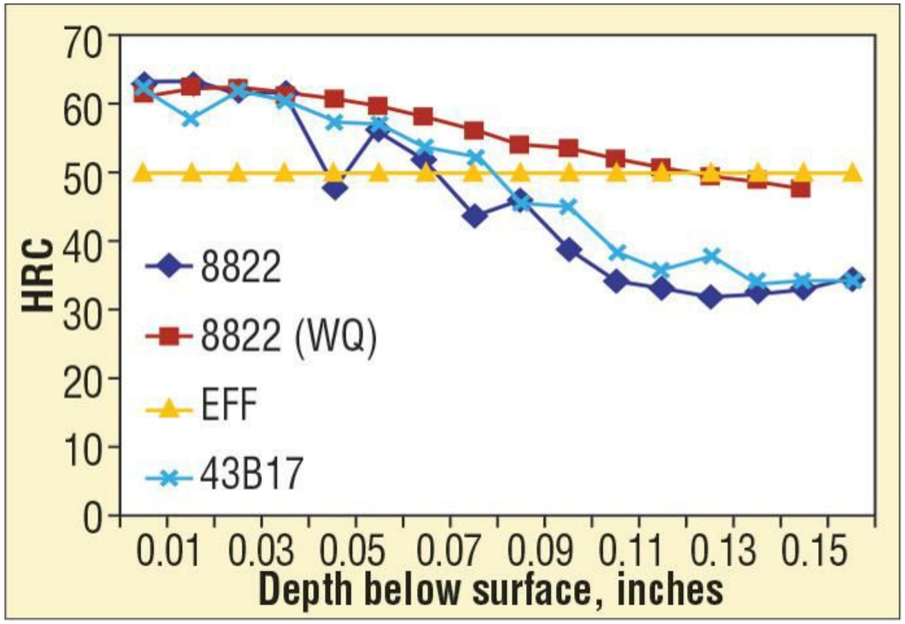


Fig. 6. Comparison of carburized 8822 (water vs.oil quenched) and 43B17 oil quenched

Alloy interactions influence the presence of bainite in the carburized case and come into play when modifying a carburizing-steel composition. The regression equations provide a convenient method of predicting the effect of changes in composition on DFB. They can also aid in the establishment of a minimum alloy content to assure a bainite-free microstructure in the carburized case.

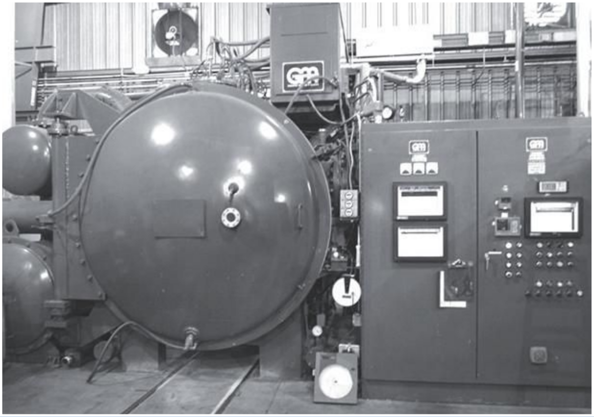
Test Results

Testing also revealed that the flank achieved a consistent effective case depth of 0.070-0.080 inch, while the root varied from 0.030-0.036 inch. The first bainite test (Table 1) indicated a quenching problem with the forgings.

Alternative Material Investigation

Oil-quenching tests were performed on similar gear components manufactured from SAE 43B17 to view the impact of a material change on hardenability response (Fig. 5). This investigation was triggered by the discovery that some of the 8822 forgings had boron additions. The profiles found are very similar in slope, but the point of first bainite has been moved to about 0.045-inch depth in the 43B17 material.

By comparing oil and water quenching, the increased hardenability of 43B17 (Fig. 6) is not as



drastic (as when water quenched) but does increase the reliability of the heat treatment and was a good alternative material choice to consider moving forward. First bainite in a boron steel can depend on carbon content, which must be taken into consideration.[5,6]

The Final Solution

While the carburizing furnace in question was capable of running up to 20 gears in a load, this investigation determined that after reheating, the gears should be independently quenched one at a time. This allowed the quench to be optimized for both the part and fixture in use and produced case-depth results without the abnormal "dip" observed. A recommendation was also made that future parts be produced from 43B17 forgings since boron additions improved hardenability.

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Fastener Industry Overview: Applications, Materials, Equipment (Part One)

Fastener materials cover the gambit from ferrous to nonferrous materials, including steel, stainless steel, tool steel, aluminum, titanium and exotic specialty grades. Coatings are also in common use and include zinc, cadmium, nickel, galvanized (hot dip, sherardizing), phosphate and PTFE (polytetrafluoroethylene) coatings to name a few.

Heat treatment plays a critical role in the manufacture of fasteners in order for them to achieve the desired performance properties. Heat treatment is typically conducted after the forming processes and before any coating or finishing process in both captive and commercial shops. The equipment necessary to effectively heat treat steel fasteners includes well-controlled atmosphere furnaces with temperature control, atmosphere control, quenching tanks and cleaning equipment. This equipment typically requires a large capital expense as well as continuing maintenance costs.

Heat-treating systems for fasteners come in a variety of shapes and sizes, but they all have one thing in common – flexibility, or the capability to produce large or small quantities of fasteners "on demand." It is not uncommon to see production lots as small as 4.5 kg/hour (10 pounds/hour) and as large as 4,535 kg/hour (10,000 pounds/hour) or more.

Fig. 1. Example of fastener usage in an aircraft engine *(courtesy of Performance Review Institute)*

The most common heat treatments for steel fasteners involve annealing of the incoming material, through or selective hardening, and case hardening such as carbonitriding and carburizing. Case depths are shallow, typically 0.0038-0.038 mm (0.0015-0.015 inch), specified in ranges of 0.127 mm (0.005

inch). Quench media runs the gambit from brine, water, polymer, oil and molten salt, depending on engineering requirements. Stainless steels and many nonferrous fasteners are solution heat treated and age hardened.

Fastener Market

The world market for industrial fasteners represents a $67 billion industry divided between Asia (38.2%), Europe (25.3%), North America (21.9%) and the rest of the world (14.6%).[1] Of this total, the Asian market is dominated by China (14.8%) and Japan (10.1%). Strong growth prevails in the automotive, aerospace and energy sectors. The industry is expected to show continued growth in the near future based on rapid global industrialization and rising demand for durable goods.

Fig. 2. Typical load of 17Cr-4Ni fasteners for vacuum heat treatment *(courtesy of Solar Manufacturing)*

Global Industry Growth

The demand for fasteners is strong. The aerospace industry is currently the largest user of fasteners, accounting for over 30% of the total market. Going forward, the use of fasteners by the construction industry is poised to overtake aerospace, based on recent activity worldwide. The construction industry's use of fasteners is expected to grow at a compound annual growth rate of just over 9% from 2012 to 2018.[2] Other OEM segments (e.g., fabricated metal products, electronic/electrical) have also exhibited strong growth in the last few years. The automotive sector is expected to continue to show above-average growth as well in the next five years.

The Asia Pacific region accounted for the highest demand for industrial fasteners last year and is expected to account for over 45% of the market by 2018. This trend is due to factors such as rapid industrialization and favorable economic conditions, which is expected to boost the demand for durable goods and other manufacturing and development activities.

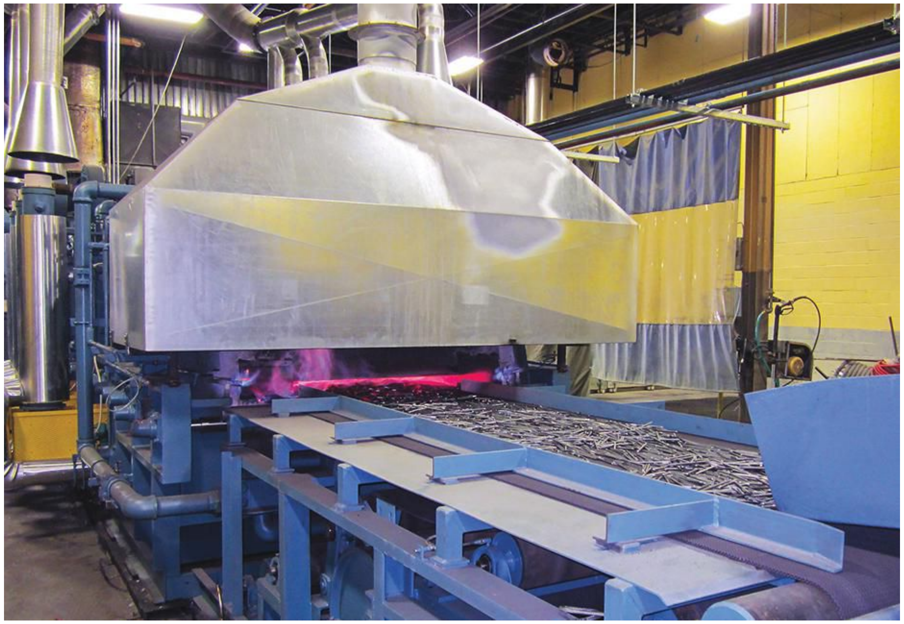

Fig. 3. Typical mesh-belt atmosphere system for fasteners *(courtesy of Surface Combustion, Inc.)*

Fastener Applications

The heat treatment of fasteners is also highly application- and industry-specific. Here are some highlights of major markets.

Aerospace Industry

Aerospace applications (Fig. 1) include aircraft (manned and unmanned, fixed and flex wing), rotorcraft (helicopters, gyrocopters) and space vehicles (shuttles, space stations, satellites).

Fasteners are one of the most critical components used in all of these applications and are required to meet the most demanding performance characteristics. The types of fasteners in the aerospace industry are quite diverse and include screws, rivets, bolts, nuts, pins, collars and washers.

Solution annealing and aging of aerospace fasteners (Fig. 2) in vacuum is an example of a thermal treatment. Stainless steel fasteners of Carpenter Custom® 630 (17Cr-4Ni), a martensitic precipitation/age-hardening stainless steel with high strength and excellent corrosion resistance, are processed using the following cycle:

Step 1: Solution heat treatment

1. Pump to 10^{-4} Torr range
2. Heat to 1040°C (1900°F)
3. Hold for 30 minutes (based on a workload thermocouple)
4. Quench in nitrogen to 32°C (90°F)
5. Unload

Step 2: Age hardening

1. Load furnace and pump down to mid-10^{-5} Torr range
2. Heat to 480°C (900°F)
3. Hold for 65 minutes (based on a workload thermocouple)
4. Nitrogen fan cool to 52°C (125°F)
5. Unload

The result is bright, shiny parts having a hardness of 42-44 HRC.

Fasteners account for a significant number of parts in aircraft and directly affect strength characteristics and weight of structural assemblies. According to The Boeing Company, the 747 includes over six million parts, half of which are fasteners. On average, for example, 2,400,000 fasteners are used to assemble a Boeing 787 aircraft. On average, 22% are structural bolts (mostly titanium) and the rest are aluminum rivets.

As the industry evolves to incorporate newer, more-exotic materials, fasteners continue to figure prominently in the manufacturing and assembly processes. Fasteners play a critical role in defining the longevity, structural integrity and design philosophy of most metallic aircraft structures.

Typical aerospace fastener materials include aluminum, steel (e.g., A286, H-11) superalloys (e.g., Waspaloy®, Hastalloy®, Inconel® 718), nickel alloys (e.g., Monel®, K-Monel®) and titanium.

Medical Industry

The medical industry relies heavily on the use of fasteners. For example, medical devices (e.g., dental and orthopedic implants, instruments) employ literally hundreds of different types of fasteners to hold their assemblies together. Even though the components in the medical devices are small or even tiny, when a fastener fails, the device will almost always fail as well.

Fig. 4. Load of fasteners on a mesh belt for carbonitriding

The correct fastener ensures that the device goes together and stays together for the intended life of the assembly and that the device performs as desired. Fasteners can overcome challenges in assembly, solve quality problems and significantly reduce the total cost of the device.

Medical devices fall into two broad categories: surgical/non-implant devices and implantable devices. The alloys and heat treatments for the fasteners involved in both are explained.

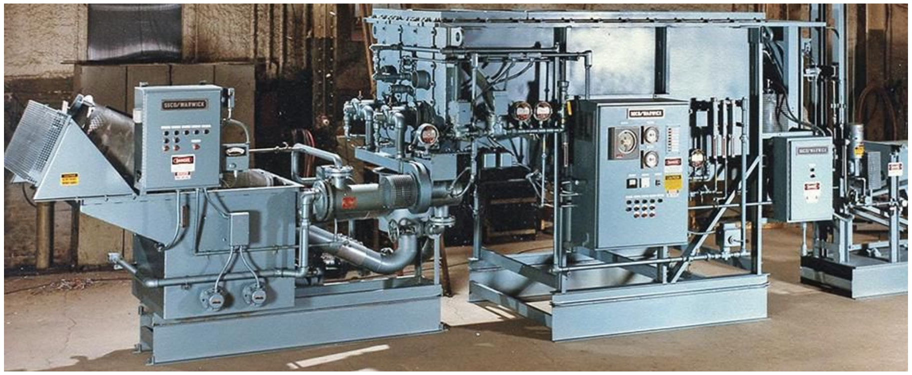
Fig. 5. Typical rotary-retort system with oil-, polymer- or water-quench capability *(courtesy of SECO/WARWICK Corporation)*

Surgical and Non-Implant Medical Devices

Surgical and dental instruments are examples of non-implant medical devices. They are typically manufactured from austenitic stainless steels where good corrosion resistance and moderate strength are required. Examples include canulae, dental-impression trays, guide pins, hollow-ware, hypodermic needles, steam sterilizers, storage cabinets, work surfaces and thoracic retractors to name a few. These applications often use a variety of stainless steels that can be easily formed into complex shapes.

Implantable Medical Devices

Specific grades of austenitic stainless steel and high-nitrogen austenitic stainless steels are used for some surgical implants. Examples include aneurysm clips; bone plates and screws; femoral fixation devices; intramedullary nails and pins; and joints for ankles, elbows, fingers, knees, hips, shoulders and wrists.

However, the vast majority of orthopedic implants worldwide are manufactured from titanium (e.g., Ti-6Al-4V alloy) or cobalt-based alloys (e.g., ASTM F75, a cobalt-based alloy or cobalt-chromium-molybdenum alloys). They are manufactured from castings, forgings or bar stock.

Medical application examples include pins, bone plates, screws, bars, rods, wires, posts, expandable rib cages, spinal fusion cages, finger and toe replacements, hip and knee replacements, and maxio-facial prosthetics.

Heat-Treat Equipment for the Fastener Industry

Fastener heat treatment can be performed in a wide variety of furnaces and ovens, and fastener designers should understand the multitude of choices available to them. Here are several examples of atmosphere furnaces used for this task.

Mesh-Belt Conveyor Furnaces

Today, mesh-belt conveyor furnaces (Fig. 3) are the dominant technology for the heat treatment of fasteners. These units are often part of a completely automated heat-treating system that includes loaders, pre- and post-washers, a hardening furnace with quench tank and

Fig. 6. Electrically heated shaker-hearth furnace for titanium fasteners *(courtesy of DF Industries, Inc.)*

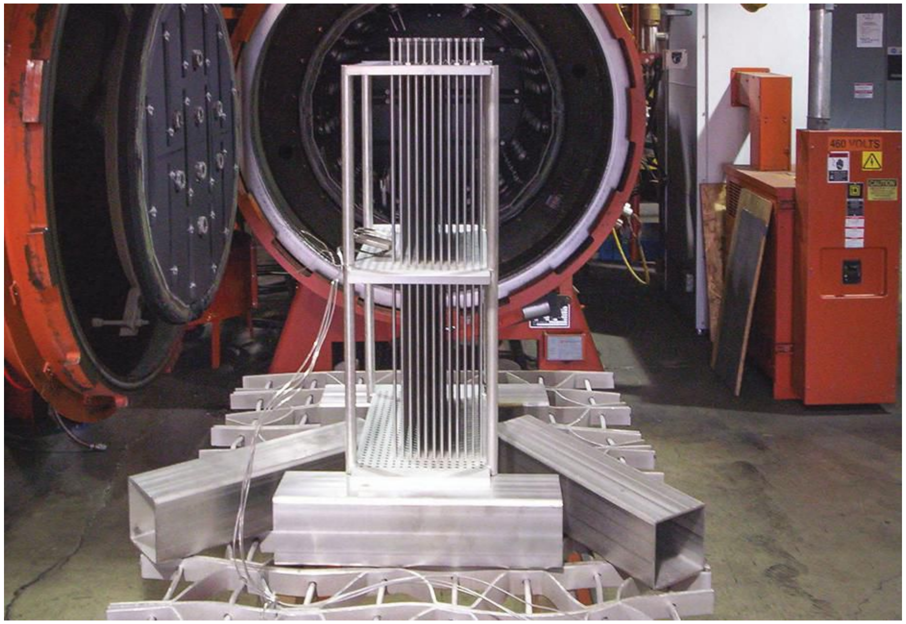

Fig. 7. Load of bone reamers processed in a vacuum furnace *(courtesy of Solar Atmospheres Inc.)*

a tempering furnace. Soluble oil tanks and endothermic atmosphere generators or nitrogen/methanol systems are common ancillary items.

Heat-treating systems for fasteners must be flexible enough to handle both large and small quantity demand. It is not uncommon to have production lots as small as 4.5 kg (10 pounds) and as large as 4,535 kg (10,000 pounds) or more. Standard production capacities in mesh-belt furnaces typically range from 100 kg/hour (250 pounds/hour) to 3,000 kg/hour (7,000 pounds/hour). It is also not uncommon to see fasteners loaded on the belt between 12.7-63.5 mm (0.5-2.5 inches) deep (Fig. 4).

Through hardening, selective hardening and case hardening (carbonitriding and carburizing) are typical heat-treat processes for fasteners. Case depths are typically shallow, in the order of 0.0038-0.038 mm (0.0015-0.015 inch). Quench media are more diverse, ranging from brine, water, polymer, oil and molten salt, depending on engineering requirements.

Other Technologies

Rotary retort (Fig. 5), shaker hearth (Fig. 6) and cast-link conveyor furnaces are the most common alternatives to mesh-belt conveyors and are usually floor mounted. The systems are typically automated and completely automatic after loading parts into the hopper at the front end. Vibratory hoppers and weight-actuated skip loaders deposit precisely measured charges into the furnace to ensure the uniform loading of parts.

Typical standard system capacities for rotary-retort furnaces vary from around 225 kg/hour (500 pounds/hour) to 450 kg/hour (1,000 pounds/hour), but they can be manufactured to handle 1,800 kg/hour (4,000 pounds/hour) or more. The retorts are either cast or fabricated from high-temperature alloys. In general, cast retorts have superior mechanical-strength characteristics compared to wrought fabricated designs but often come at a cost premium. Auger flights with the retort convey the fasteners through the furnace. Variable-speed rotation of the retort provides flexibility of time-based processing cycles.

The actual quenching process of the part is typically completed within two to five seconds from the time fasteners drop into the quenchant, with 10 minutes being a typical residence time in the quench for continued cooling. The exception is austempering, which requires extended times in the order of 20 minutes or more to produce a bainitic structure. It is critical that an adequate amount of fluid flow is delivered to the active quench area because overall quench-tank capacity is not enough to ensure individual quenching of each part. The quench-tank design must eliminate any clumps and clogs to ensure that both proper technique and adequate quenching is obtained.

Applications for Vacuum Furnaces

An increasing number of vacuum furnaces (Fig. 7) are being used for the processing of fasteners and fastener-related products due to the type of material being processed (e.g., bioengineering or nuclear) and the type of surfaces produced.

Other Processes

Tempering furnaces and ovens, whether continuous (e.g., mesh belt, pusher) or batch, use a recirculation system designed to allow heated air to circulate through the mass of fasteners to achieve uniform temperature throughout the load. Convection heating is used to full advantage for efficient heat transfer in a minimum floor space. Typically, operating-temperature ranges vary from 150-650°C (300-1200°F).

Induction heating is also used to selectively heat treat fasteners at high production rates. For example, high-frequency (10-50 kHz) systems can temper (draw) back bolt heads after carburizing to improve toughness at rates in the order of 1.0-2.5 pieces/second. Similarly, seat and seat-belt retention bolts can be case hardened (200 kHz) to depths up to 0.508 mm (0.020 inch) and surface hardness of 40-45 HRC to impart both strength and toughness.

Summary

Fastener heat treatment in atmosphere furnaces is widely used to meet the demand for higher quality in a broad range of industries and provides a highly cost-competitive technology based on high throughput and consistent, repeatable quality.

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Fastener Industry Overview: Applications, Materials, Equipment (Part Two)

Fastener applications are as diverse as the industries they service. In this article, we take a look at automotive, construction, mining, marine, petrochemical, nuclear and specialized applications. It is estimated that some 350 manufacturing plants produce more than 200 billion fasteners per year in the U.S. alone.[1]

Fastener Usage in the Automotive Industry

The automotive industry alone is estimated to use between 25-35 billion fasteners. This manufacturing segment continues to see sales of greater than 14.5-15.5 million vehicles in North America. The automotive market share (as of 2012) is divided as follows: GM 17.9% (up 3.7%), Ford 15.5% (up 4.7%), Chrysler 11.4% (up 21%), Toyota 14.4%, Honda 9.8% and Hyundai/KIA at 8.7%.

Steel and stainless steels, including duplex and austenitic grades (Table 1), as well as plastic fasteners dominate the automotive landscape. These four tables summarizing the types of materials used in the various automotive applications appear exclusively in the online edition of this article. Many fasteners are plated or coated for increased corrosion protection.

Fig. 1. Fasteners for high-vibration material-handling applications (courtesy of Security Locknut, Inc.)

Fastener Usage in the Construction Industry

Fasteners are the critical link in the load path of a building structure. They provide structural integrity and are a major point of energy dissipation under seismic and wind loads. Construction fasteners are generally classified as those fasteners that are used to secure building materials and can be classified as commodity fasteners or task-specific fasteners. Typical applications include:

- Roofing (flat, sloped)
- Decks (steel, wood, composite)
- Bridges (ladder deck, multi-girder, suspension)
- Buildings (residential, commercial, skyscraper)

Typical examples include cement-board screws, drywall screws, needlepoint screws, outdoor screws, pole-gripper screws, self-drilling screws and woodworking screws. Steel (Table 2) and stainless steel fasteners are commonly used in the construction industry.

Fastener Usage in the Mining Industry

A key requirement for fasteners in the mining and excavation industry is their resistance to vibration and corrosion. Vibrating conveyors, crushing and pulverizing equipment, mining machinery, railroad cars and material-handling devices are examples of applications where vibration is a significant issue that requires the fastener to be designed so that it won't "back off" while in service (Fig. 1). Furthermore, corrosion and/or cracking is often involved in mining fasteners being related to hydrogen-induced cracking (aka hydrogen embrittlement), often in the form of stress corrosion cracking (SCC) or sulfide stress cracking (SSC).


Fig. 2. Typical assortment of fasteners used in the nuclear industry

The types of fasteners used in the mining industry are quite diverse and include bolts (e.g., anchor, eye, structural, swing), rods (e.g., elevator, rock, tie, sag), washers, nuts and plates. Materials include carbon and alloy steels, construction steels and stainless steels.

Fastener Usage in the Marine Industry

The marine industry is literally held together by fasteners designed to survive the extremely harsh and

Table 1. Materials for Automotive Products

	Material/Product	Carbon steels for cold heading			Alloy steels for machine structural use					Boron Steels	Steels for special use			Bearing steels	Others			
											Stainless steels							
		Rimmed steels	Aluminum killed steels	Killed steels	Nickel chromium molybdenum steels	Chromium steels	Chromium molybdenum steels	Manganese steels for machine structural use	Alloy steels for bolts of high temperature use	Designated by customer	Austenitic	Marten-sitic	Heat resistant steels	High carbon chromium bearing steels	Non-heat treated steels	Titanium alloy	Cobalt alloy	High strength steels
		SWCH•••R	SWCH•••A	SWCH•••K	SNCM•••	SCr•••	SCM•••	SCm•••	SNB•••	———	SUS3•••	SUS4•••	SUH•••	SUJ•••	NHF•••	6AL–4V	MP35N	
Engine bolts	Cylinder head bolts					●	●			●								●
	Connecting rod bolts				●	●	●										●	●
	Bearing cap bolts						●			●								
	Flywheel bolts					●	●			●								
	Tips						●											
	Crank pulley bolts							●										
	Drive plate bolts					●												
	Metal cap bolts						●			●								
	Rocker stay bolts									●								
	Adjusting screws						●											
	Crank shaft bolts						●	●		●								
Brake parts	Guide pins						●				●				●			
	Lock pins						●			●					●			
	Wedges						●											
	Holder pins			●														
	Sleeve bolts	●					●											
	Adjusting screws	●		●	●		●							●				
	Adjusting nuts	●	●															
	Adjusting sockets	●																
	Adjusting Assys	●	●	●										●				
	Roller tappets													●				
Special shape parts	Stud bolts	●		●	●		●		●	●	●		●			●		
	Line head bolts	●					●			●								
	Joint bolts	●		●			●			●								
	Hub bolts						●											
	Serration bolts			●						●								
	Square head bolts														●			
	Socket bolts	●		●			●			●								
	Plungers			●			●					●						
	Weld bolts	●		●											●			
	Caulking bolts			●			●			●								
	Cap square neck bolts		●															
	Shoe bolts									●								
	Seal bolts			●			●			●								
	Rivets	●																
	Stepped bolts	●		●						●					●			
	Bolts			●											●			
	Adjusting bolts	●									●							
Nuts	Weld nuts	●																
	Nylon nuts	●																
	Multi-point head nuts	●					●											
Other parts	Parts by part-former	●		●						●								
	Automatic joint						●				●							
FT • FTN • FTY bolts		●	●	●			●					●						
Standard bolts	Hexagon head bolts	●	●	●		●	●		●	●			●			●		
	Hexagon head bolts with flange	●	●	●			●			●	●		●			●		
	Bolts with captive washer	●		●			●			●					●			
	Hexagon socket head cap bolts	●		●			●									●		

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Table 2. Selected properties of steel construction-grade fasteners

ASTM designation no.	Size range (inclusive), mm (inch)	Minimum proof strength, ksi (MPa)	Minimum tensile strength, ksi (MPa)	Minimum yield strength, ksi (MPa)	Material
A307	6-38 (0.25-1.5)	33 (227)	60 (413)	36 (248)	Low carbon
A325, Type 1	12-25 (0.5-1.0) 29-36 (1.125-1.4)	85 (586) 74 (510)	120 (827) 105 (723)	92 (634) 81 (558)	Medium carbon, Q&T
A325, Type 2	12-25 (0.5-1.0) 29-38 (1.125-1.5)	85 (586) 75 (517)	120 (827) 106 (730)	92 (634) 82 (565)	Low carbon, Q&T
A325, Type 3	12-25 (0.5-1.0) 32-41 (1.25-1.6)	85 (586) 76 (524)	120 (827) 107 (737)	92 (634) 83 (572)	Alloy, Q&T
A354, Grade BD	6-101 (0.25-4.0)	120 (827)	150 (1034)	130 (896)	Alloy, Q&T
A449	6-25 (0.25-1.0), 29-38 (1.125-1.5) 45-76 (1.75-3.0)	85 (586) 74 (510) 55 (379)	120 (827) 105 (723) 90 (620)	92 (634) 81 (558) 58 (400)	Medium carbon, Q&T
ASTM 490, Type 1	12-38 (0.5-1.5)	120 (827)	150 (1034)[a] 173 (1192)[b]	130 (896)	Alloy steel, Q&T
ASTM 490, Type 2	12-25 (0.5-1.0)	120 (827)	150 (1034)[a] 173 (1192)[b]	130 (896)	-
ASTM 490, Type 3	12-38 (0.5-1.5)	120 (827)	150 (1034)[a] 173 (1192)[b]	130 (896)	Low carbon, Q&T
ASTM A490M, Type 1 or 3[c]	12-36 (0.5-1.5)	17 (117)	150 (1034)	136 (937)	-

Notes: [a]Minimum value [b]Maximum value [c]Type 2 withdrawn

Table 3. Classification of exotic alloys for specialty fasteners

Class	Alloy	Examples
0	Steels[a]	A286
1A	Stainless steels[b]	Alloy 20, Alloy 50, Alloy 60, Custom 450, Carpenter 21, Carpenter CB3, 904L, AL6XN, Avesta 254SMo
1B	Stainless steels[a]	13-8, 17-4, 17-7
1C	Stainless steels[c]	2304, 2205, 2507, 2707, 3207, Ferralium 255-SD50
1D	Stainless steels[d]	50, 60
2A	Superalloys[e]	B, C-276, C-2000, C-22, C-4, G, X
2B	Superalloys[f]	230, HR120, HR160, ULTIMET 1233
2C	Superalloys[g]	600, 601, 625, 718, 800/800H, 825, 925, X750, 25-6Mo
2D	Superalloys[h]	Waspaloy
3A	Nickel alloys	B2, B3, G30
3B	Monel	400, 405, 500
4	Other	Co, Ta, Ti, Zr

Notes: [a]Precipitation-hardening grades [b]Chromium-nickel-molybdenum grades (with selected alloy additions) [c]Duplex and super-duplex grades [d]Nictronic alloys [e]Hastalloy grades [f]Haynes International alloys [g]Inconel® grades [h]Other superalloy grades corrosive environments to which they are exposed. Bolts, screws, washers, locking washers, wing nuts, split rings and slating nails are just some of the many marine construction fasteners used on docks and ramps, ships, tanks, winches and for underwater construction projects.

Because fasteners are generally much smaller than the components that they support, the fasteners generally must be more corrosion-resistant. As such, heat-treatment processes need to be carefully chosen and controlled in order to not have a negative impact on corrosion resistance or other important physical or mechanical properties.

In addition, since the fastener material is often dissimilar to the components being joined, galvanic-induced corrosion is a principal concern. Should the fastener be anodic to the remainder of the structure, the relative-size effect could cause severe corrosion and degradation of the fastener in a short period of time. Failure of a nut or bolt can have catastrophic results.

There are many copper alloys suitable for marine service, including coppers, copper-nickels, bronzes (aluminum and silicon bronze), brasses and copper-beryllium. For seawater systems, copper-nickel and aluminum bronze are often preferred, although other copper alloys are used in marine service and have their specific advantages. Copper alloys differ from other metals in that they have an inherent high resistance to biofouling, particularly macrofouling, which can eliminate the need for antifouling coatings or water treatment.

Many other materials can be used provided they demonstrate superior corrosion resistance. One such example is Inconel® 686, a high-performance nickel-based alloy that exhibits high tensile strength and fracture toughness as well as resistance to corrosion, especially crevice corrosion, superior to that of other nickel-based alloys (such as K500 Monel®).

Different combinations of properties can be produced in each of these materials by varying the heat treatment, which influences strength, hardness, ductility, conductivity, impact resistance and inelasticity.

Fastener Usage in the Petrochemical Industry

Petrochemical products are those derived from petroleum, being obtained from crude oil and natural gas. They are mainly used in the production of petrochemical derivatives (e.g., ethylene, methanol, butadiene, propylene, formaldehyde, polyvinyl chloride, acetic acid and epoxy resins among many others).

The growing demand of petrochemicals from major end-use industries, including transportation, construction and packaging, drives the petrochemical market. The demand for industrial fasteners is expected to grow at a rate of 5.4% through 2018[1] and is expected to fall just shy of $100 billion by 2018. In addition, the petrochemical industry is incredibly diverse, encompassing regional markets in North America, Latin America, Europe, China, Asia Pacific, Africa and the Middle East.

This diversity is also evident in the products and materials from which fasteners are constructed. Examples include threaded rod, studs (full-thread, double-end), studbolts, hex bolts and nuts, gaskets (spiral-wound, full-face, rings), setscrews, sockets, standard fasteners and specialty products such as tie bars, socket screws and machined parts.

The range of chemically resistant materials includes petrochemical grades of stainless steel, duplex and super-duplex steels, nickel and cupronickel alloys, carbon and alloy steels, titanium and other nonferrous materials, and superalloys. Coatings are common and include zinc, cadmium, nickel, galvanized (hot dip, sherardizing), phosphate and PTFE (polytetrafluoroethylene) coatings.

Fastener Usage in the Nuclear Industry

A typical nuclear power plant contains some 40,000 fasteners, including bolts, washers, nuts, studs, cap screws, pins, rivets and machine screws.[1] These fasteners are subjected to very specific design rules. Problems can develop, however, either immediately or over time.

The most common cause of fastener failure in the nuclear industry is reportedly due to SCC of certain stainless steel alloys.[1] Other contributory factors include improper material selection, poor installation practices (e.g., improper torque), bolts that were not properly sized and failures induced by a variety of heat-treatment issues (e.g., overheating, decarburization and quench cracks).

Fasteners for the nuclear industry are made from a wide variety of materials (Fig. 2). These include carbon and alloy steels, stainless, Nitronic® and Monel® alloys, Inconel® and titanium as well as brass, bronze and copper. This diversity means that required properties and heat treatments are always specified, often in ASME and SAE standards (e.g., ASTM SA193/SA194/SA276/SA307 and SAE J429/J995). A variety of coatings can be applied after heat treatment. These include zinc, cadmium, phosphate, ceramic, chromium and black oxide to name a few.

Fastener Usage in Specialized Applications

Critical applications require the use of materials whose performance envelope encompasses both normal-duty and extreme-duty demands. It is the latter that differentiates specialty fasteners and components from standard ones.

What are specialty fasteners?

Specialty fasteners are those whose applications demand performance over cost. Mechanical, physical and metallurgical properties are more stringent than those involving standard fasteners. Examples of equipment and industries that rely on specialty fasteners include power generation (e.g., gas turbines, offshore performance platforms), pulp and paper mills and electronic devices.

In aerospace (aircraft, rotorcraft, space) applications, specialty fasteners are used are on exteriors, interiors, avionics and flight systems (e.g., landing gear). Product examples include captive screws, rivets, gas springs, clamshell flexible couplings, quick-release pins, tension latches and telescopic slides. In automotive (motor sports, cars, off-road, heavy truck), fasteners and clamping device are commonplace throughout, including the engine, body and subsystems.

Alloy Fastener Materials

There are some unique material selection challenges in the design of very high-strength/high-performance fasteners. These type of fasteners are often exposed to high stress concentration in the thread roots (caused by the tensile stresses produced from extremely high clamping loads), on top of which are superimposed any fatigue loads. To meet these challenges, the designer often selects alloys classified as "exotic" primarily due to their chemistry and ability to perform at elevated temperatures (Table 3).

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It's Time to Clean Up Our Act!

As heat treaters, one of our New Year's resolutions should be to clean up our act! Cleaning parts before and after heat treatment is a critical part of what we do. Most of us feel we clean parts "good enough" and feel we know what cleaning is, but few actually do. Let's learn more.

Cleaning is the application of time, temperature, chemistry and energy to remove contamination from the surface of a part to a level appropriate for the intended application. In other words, cleaning is simply moving contaminants from where they are not wanted (on the parts) to where they should be (in the waste-disposal system). If all four aspects of the cleaning process are not working together, the parts will not be properly cleaned. Although heat treating demands only a moderate level of cleanliness compared to many industries, contamination left on parts can cause significant problems in our equipment (Fig. 1) and on the parts themselves (Fig. 2).

All cleaning systems depend on one or a combination of three basic actions:

- A physical action – mechanical force – such as spray agitation, dunking, ultrasonics or even hand (abrasive) cleaning to remove the contaminants from the part surface
- A thermal action to improve the activity of the cleaning solution and increase the kinetic energy of the system
- A chemical action to allow contaminants to be either desorbed from the part surfaces with the aid of surface active agents or dissolved by an action of absorption and dilution

Aqueous Cleaning

Aqueous cleaning is the dominant approach used in our industry. Its simplicity, ease of use and overall flexibility is what makes it an attractive process. Aqueous cleaning uses detergents to lift contaminants from the surface of the parts; heat to make the detergents more compatible with the contaminants and to soften them; fluid force to dislodge the contaminants from the parts and to collect the insoluble contaminants in some removal systems; and time to allow the process to take effect.

Aqueous cleaning is not perfect, however. It often leaves a surface residue or "film" on the parts, which may interfere with certain processes such as brazing or nitriding and thus require subsequent removal. Aqueous cleaners don't dry well and the solution is often difficult to remove from internal part surfaces

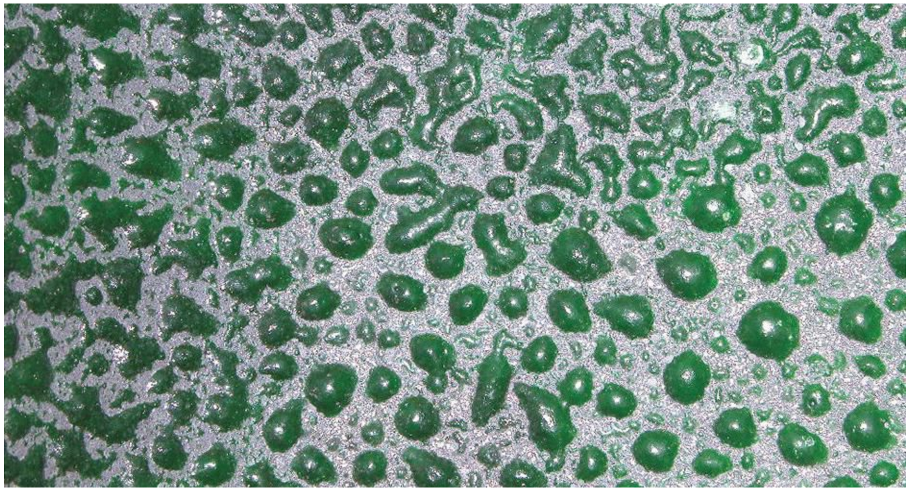

Fig. 1. Green internal furnace-refractory contamination due to excess cleaning compound on parts

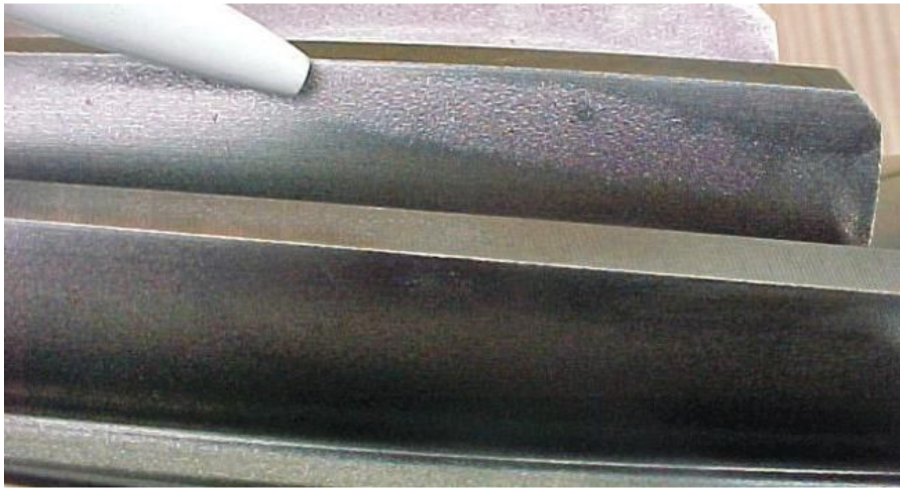

Fig. 2. Soft spots on carburized parts after shot peening

such as holes, crevices and recessed areas. Finally, aqueous cleaners evaporate slowly, requiring large amounts of energy to dry them, and have been known to damage certain sensitive parts.

Solvent Cleaning

Cleaning in a solvent offers a level of simplicity and forgiveness not seen in aqueous methods. Solvent cleaning involves three basic steps: wash, rinse and dry. Washing is where the parts are immersed in or contact the typically boiling solvent to help the removal process. The purpose of rinsing is to bring "fresh" or clean solvent in contact with the parts. The aim is to dilute the contaminated solvent present on the surface of the parts from washing. It is important to remember that the rinse solvent must be kept clean. Contaminated solvent is a very common problem and will only reintroduce contaminants back onto the surface. The drying step evaporates the solvent and separates the rinse solvent from the parts.

Solvent cleaning has a negative connotation in the heat-treating industry primarily due to environmental concerns, safety and cost issues. The emergence of vacuum vapor degreasing in a sealed vessel offers an attractive alternative that takes advantage of the best aspects of solvent cleaning. The size and amount of residual contaminants are reduced while meeting the most stringent cleaning requirements and avoiding the traditional problems of open degreasing systems.

Cleaning Tips

1. Clearly understand what your cleaning needs are. That is, what types of contaminants are present and in what quantities. Also, know to what extent they must be removed. In other words, answer the question, how clean do I need to be?
2. If you currently own cleaning equipment, spend your time making sure the system is well maintained, operating properly and performing up to expectation.
3. If you are considering the purchase of a cleaning system, narrow the choices to one or two suppliers, using published information and referrals, then witness a cleaning trial on your parts by the supplier you most prefer. Finally, understand that your time is best spent making the selection work.
4. Rinse the parts thoroughly. Rinsing is needed to separate the dirty chemistry from the more pure chemistry. The benefits of a good rinse should not be underestimated.
5. Dry the parts thoroughly. Drying is separation of parts from pure cleaning agent. Dry only to the extent necessary and consider non-evaporative methods of separating the cleaning agent from the parts.
6. Measure your cleaning effectiveness constantly to ensure the system is not degrading. If you are using an aqueous system, invest in a good oil skimmer. Be sure that it is well maintained and operates consistently over time.
7. Don't re-contaminate clean parts.
8. Clean the cleaning equipment – thoroughly and often. Replace the bath on a routine basis and don't try to extend its life.
9. Do all cleaning, rinsing and drying at as low a temperature as practical. Balance process efficiency and cost of cleaning.
10. Recognize that the average life expectancy of a cleaning system is three to five years. Replace your equipment regularly to keep the cleaning process working at optimum efficiency.

How Long and How Clean is Clean?

Cleaning time depends to a large extent on the system and the parts. Typically, an aqueous-based process needs to run for 10-20 minutes while solvent-based techniques need only 5-10 minutes to complete the cleaning process. As a rule, it is more difficult to clean clean parts than dirty ones. A key question is always how do we know when the parts are clean?

There are a large number of tests to measure cleaning effectiveness. These include visual inspection, macroscopic (5-50X) inspection, "white glove" inspection, observation under ultraviolet (black) light, tape sampling, "water break" tests, surface-tension test fluids, the Nordtest method and gravimetric methods. A number of technical organizations including ASTM (www.astm.org) offer cleaning standards, often based on the type of material to be cleaned. Remember, clean is generally observed, not measured, and cleaning effectiveness is established by answering the question, can we do what we need to do next? However, cleaning tests provide quantification of the nature of the part surface so that the influence of the remaining contaminants can be factored into the heat-treatment operation as well as subsequent manufacturing operations.

Other Types of Cleaning

Blast cleaning, pressure washing, steam cleaning, abrasive cleaning and other mechanical methods can be used to "clean" part surfaces. Fluidized beds have also been used for years to remove contaminants. In cases where it is beneficial to remove imperfections such as stains or surface corrosion, heat discoloration, oxide films, weld marks, scratches and particles of all sizes, electropolishing can be used. These "nontraditional" cleaning approaches have value and present their own unique set of challenges.

Where the Industry Is Headed

It is important to recognize that both aqueous and solvent cleaning processes can be made to clean almost any parts requiring subsequent heat treatment. Therefore, the choice between them will most often be made by factors other than expected part cleanliness. The focus today is on improving physical action (force and volume) in combination with a chemistry choice balanced for the type of cleaning required.

Finally, take a systems approach. Consider the manufacturer of the cleaning system and the supplier of the cleaning agent as partners in the long-term success of any cleaning operation.

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Cold and Cryogenic Treatments

Thermal treatments can involve heating or cooling. When it comes to low-temperature processes, there is a distinction made between subzero, or cold treatment, to –84˚C (–120˚F) and deep cooling, or cryogenic treatment, to –195˚C (–320˚F). A question many designers, metallurgists and heat treaters ask is: Can these treatments be used for anything besides converting retained austenite to martensite? In other words, are performance enhancements achievable with materials such as aluminum, brass, bronze, carbide, cast iron, copper, stainless steel (austenitic and martensitic), steel and titanium, as well as tool steels? And is trial and error on a case by case basis still necessary? Let's learn more.

The Basics

The concept of changing the properties of metals through the use of low temperature had its origins in this country back in World War II when the life of steel cutting tools was improved by immersing them in liquid nitrogen prior to being placed in service. Today, the technique most commonly used involves gradual changes in temperature. Of course, anyone familiar with a Siberian winter might speculate that the Russians have been testing the theory of cryogenics for a very long time. Because the mechanisms involved have been poorly understood, more research is needed. Today, the technique most commonly used involves gradual changes in temperature rather than direct immersion. It is a fact that exposure to very low temperatures does produce permanent changes in materials. Reported benefits include:

- Increased fatigue resistance
- Precipitation of fine carbides
- Increased abrasion resistance
- Change in vibrational dampening
- Increased electrical conductivity
- Stabilization and reduced warpage
- Transformation of retained austenite

Tempering is necessary after treatment, especially on hardened steels. Cold and cryogenic processes affect the entire part. Thus, subsequent manufacturing steps, such as machining, can be performed after treatment without loss of properties.

Bearings	Engine blocks	Knives
Brake rotors	Extruders	Lifters
Cams & camshafts	Gears	Piston rings
Clutches	Granulators	Punches
Crankshafts & rods	Gun barrels	Push rods
Cutters	Hammermills	Reamers
Cylinder heads	Heads	Shear blades
Dies	Hobs	Slicers & slitters
Drill bits	Inserts (carbide)	Valve springs
End mills	Intake manifolds	Welding electrodes

Table 1. Selected products that are given a cold/cryogenic treatment to enhance performance

Applications

Cryogenics has found its way into a number of product applications (Table 1), including some that are unique. For example, musical instruments have better tonal quality and sports equipment (e.g., tennis rackets, golf balls and clubs, baseball bats and firearms) show better performance in distance, accuracy and life. Even flags are treated to prevent their colors from fading and panty hose to reduce the risk of tearing.

Cryogenic treatment of brake pads and rotors (Fig. 1) in automotive applications is said to improve service life. In police cars, where a combination of high speeds and frequent braking translates to brake-component replacement around 8,000 miles, cryogenic processors report having extended the time between replacement of these components consistently up to 24,000 miles. The brake rotor is a pearlitic cast iron (Fig. 2).

In another example, connecting rods made of 300M alloy steel used in certain V8 racing engines are processed in custom designed heat treatment/cryogenic treatment cycles to hardness values (approx. 42 HRC) generally thought too hard/brittle for the application. These components are able to withstand repetitive stress of reversing the piston direction at the top of its stroke at over 9,600 rpm.

Documented improvement

A scientific study[1] conducted to investigate the wear resistance and microstructural changes of quenched and tempered alloy tool steels, followed by either cold treatment (–60˚F or –50˚C) or cryogenic treatment (–300˚F or –185˚C) produced some interesting results. An almost identical volume fraction of retained austenite was found using x-ray phase analysis in samples austenitized at approximately 1875˚F (1025˚C) and treated

Fig. 1. Automotive brake pads and rotors
(Courtesy of Controlled Thermal Processing, Inc.)

Fig. 2. Pearlitic Structure of a Cryogenically Treated Gray Cast Iron Break Rotor: 100X, 2% Nital *(Courtesy of Controlled Thermal Processing, Inc.)*

using one or the other low-temperature process. However, cryogenic-treated specimens had improved (sliding) wear resistance (Fig. 3). The wear rate of as-quenched samples was greater than those of cold- and cryogenic-treated specimens at sliding distances of 656 ft (200 m). At double and triple these distances, cold-treated samples had almost the same wear rate as the as-quenched samples. However, cryogenic-treated specimens had a smaller wear rate than the as-quenched samples and cold-treated samples for any sliding distance.

The volume fraction of retained austenite was also analyzed for samples austenitized at 1875 and 2000˚F (1025 and 1095˚C). As-quenched samples had about 12% retained austenite compared with approximately 6% for cold- and cryogenic-treated specimens. Cold treatment drastically reduced the volume fraction of retained austenite, while cryogenic treatment only reduced it slightly relative to cold treatment.

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Summary

This research shows cryogenic treatment increased (sliding) wear resistance dramatically, especially at high sliding speed, and the cryogenic-treated specimens had minimum wear rates. The microstructure was also dramatically different between cold- and cryogenic-treated samples. Unlike cold treatment, cryogenic treatment promotes preferential precipitation of fine eta-carbides. It is assumed that the formation mechanism of eta-carbides involves the expansion and contraction of iron or substitutional atoms, allowing carbon atoms to shift slightly due to lattice deformation as a result of cryogenic treatment. The contribution of cryogenic treatment to wear resistance is believed to be precipitation of fine eta-carbides, with enhancements in strength and toughness of the martensite matrix rather than the removal of retained austenite.

Cold and cryogenic treatments are not a cure-all. However, they are gaining in popularity. For this reason, many heat treatments include this step as a logical continuation of the heat treating process, as well as to add a measure of safety to product performance. However, a thorough review of the design is imperative to ensure that the correct heat treatment for the material has been specified and the use of cold or cryogenic treatment produces tangible, documented benefits.

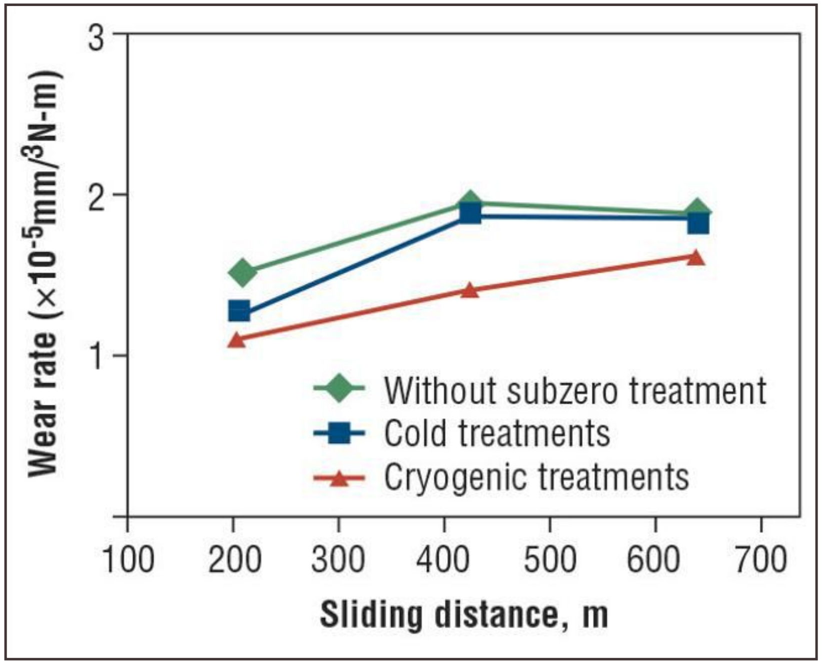

Fig. 3. Wear rate versus sliding distance[1]

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A Tutorial on Shot Blasting and Shot/Laser Peening

Not all similarly sounding processes are created equal and a good example of this is the difference between shot blasting and shot/laser peening. Let's learn more.

Shot Blasting

Shot blasting or blast cleaning is a process in which an abrasive material (Fig. 1) is accelerated through a pressurized nozzle or centrifugal wheel and directed at the surface of a part to clean or otherwise prepare the part surface for further treatment. The media used will vary as a function of the type of cleaning process and includes sand; steel shot; cut wire shot; chilled iron; garnet, a sharp hard abrasive which is used to prepare surfaces on non-ferrous metals; olivine, a soft abrasive material for use on decorative stone or non-ferrous metals; and glass beads used to polish rather than remove surface coatings on soft metals and plastics.

Shot blasting can be used on castings, forgings, and stampings to produce a uniform surface texture and for descaling, deburring, and deflashing. A certain degree of fatigue resistance is imparted to the material due to compressive stresses produced at the surface although the effects are non-uniform and thus shot

Fig. 1. Abrasive shot blast media (photograph courtesy of Eisenwerk Würth GmbH, www.eisenwerk.com)

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blasting is not considered a controlled process. Shot blasting is used in a wide variety of industries including automotive, marine, mining, and medical applications.

Shot Peening

By contrast, shot peening is a cold working process, which uses the mass and velocity of a shot stream to produce residual compressive stress at the surface of the part. Each piece of shot striking the metal surface acts as a tiny ball peen hammer, imparting a small indentation, or dimple on the surface. At the time of shot impact, the metal surface yields in tension due to localized stretching that occurs, while the near surface layer is left in a residual compressive state due to the material's attempt to restore the surface to its original shape (Fig. 2).

Media used for shot peening consist of small spheres of cast steel, cut wire formed into nearly round shape (both carbon and stainless steel), ceramic, and glass material.

A variety of different metallic based materials are currently shot peened including: high strength steels, carburized and decarburized steels, cast and austempered ductile iron, and nonferrous alloys of aluminum, titanium, and magnesium. Powder metal parts also benefit from shot peening. Common application uses for the technology include, for

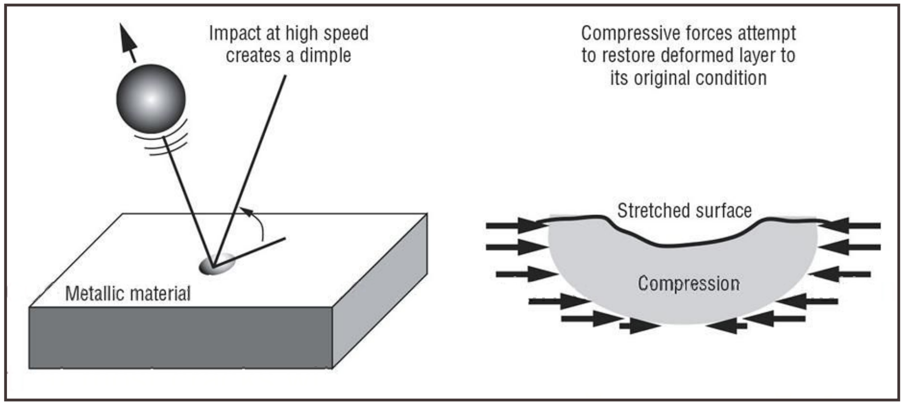

Fig. 2. The shot peening process

compressively stressed zone. Since nearly all fatigue and stress corrosion failures originate at or near the surface of a part due to tensile stress, the compressive stresses induced by shot peening (Fig. 3) provide increased part life. The magnitude of the compressive stress produced by shot peening is at least as great as half the tensile strength of the material being peened.

Shot peening is a precisely controlled process relying on careful selection and control of media, intensity, coverage and equipment. Peening media must remain predominately round and uniform in diameter to avoid surface damage upon impact and to maintain a uniform compressive stress layer. Damaged or mixed size media must be removed and replaced. Intensity (or shot stream energy) is measured using

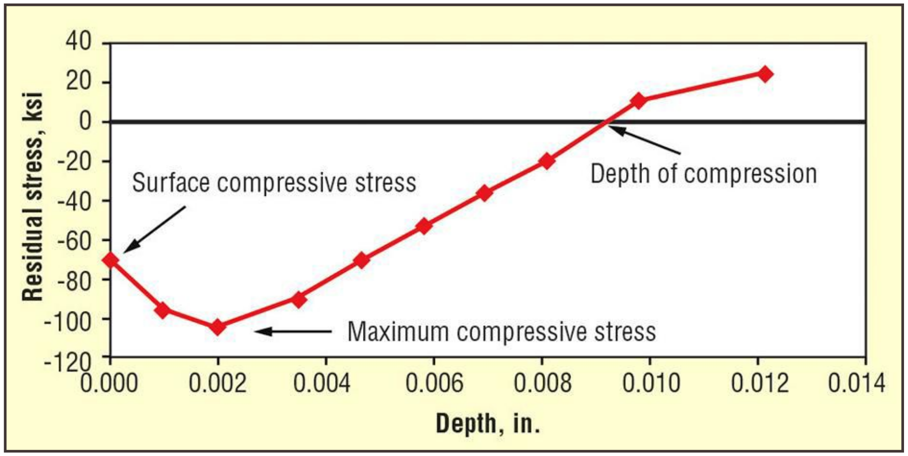

Fig. 3. Typical shot peening curve

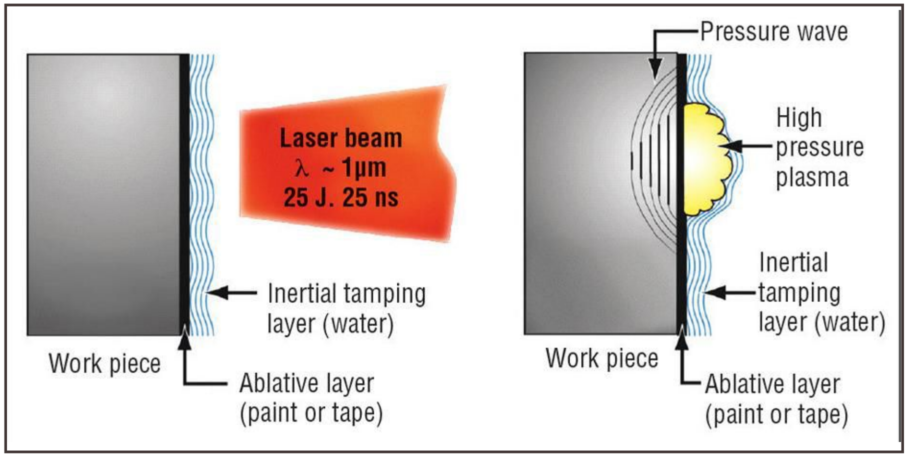

Fig. 4. Laser peening process *(Courtesy of Metal Improvement, Inc.)*

Almen strips, a spring steel peened on one side only whose height is measured as an indication of induced residual stress. Complete coverage control is critical and must be at least 100% of the surface area.

Dual shot peening can also be performed to further enhance fatigue performance at increased cost. Dual peening is a secondary peening application that results in improvement of the compressive stress at the outmost surface thus imparting additional crack resistance.

Laser Peening

An emerging technology in the peening industry uses the energy of a laser pulse to impart the residual compressive stress into a material's surface (Fig. 4). The primary benefits of laser peening are a residual compressive layer that is 4-5 times deeper than traditional shot peening with minimal cold work of the surface which can be a detriment when shot peening at high energy levels.

Laser peening generates high-pressure plasma that creates a high intensity pressure wave in a localized area. The pressure wave is capable of driving the compressive stress much deeper into the material. Some failure modes such as foreign object damage (FOD) and fretting fatigue respond significantly better to a deeper residual compressive layer. This can be best illustrated by looking at a comparison of the compressive stress depth achieved by a typical shot peen versus a typical laser peen for Inconel® 718 (Fig. 5).

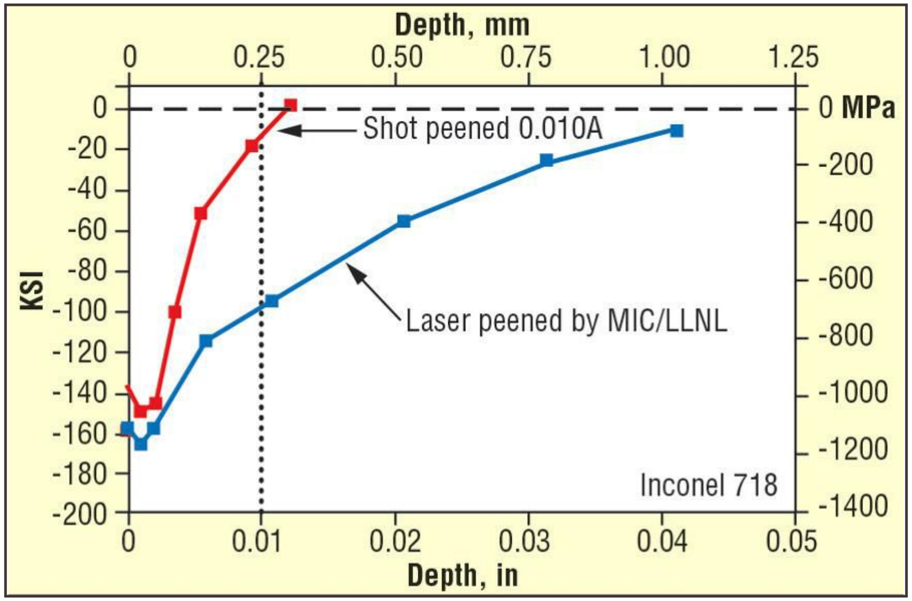

Fig. 5. Comparison of shot and laser peening (Inconel 718)

While both peening processes offer a high magnitude of compressive stress at the surface, the shot peen compressive layer is mostly dissipated by 0.25 mm (0.010 inch) deep. Should this peened surface incur a scratch 0.25 mm (0.010 inch) deep, there would be little protection preventing further potential growth of the flaw. With laser peening there is approximately 100 KSI of residual compressive stress at 0.25 mm (0.010 inch) deep. This compressive stress would attempt to arrest potential growth of the flaw.

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Temperature Uniformity Survey Tips Part One: Atmosphere Furnaces

Temperature uniformity surveys (TUS) are a key part of any good heat-treat operation. Specifications such as AMS 2750 (Pyrometry) have been referred to by some as the nemesis of many a good heat treater or quality engineer. However, if our goal is to ensure we are heat treating to the tightest practical tolerances, we must find ways to streamline the process for checking uniformity so that we will want to do it more often and with better accuracy. Let's learn more.

Up front, several important decisions need to be made: Will the surveys be conducted with or without atmosphere, and should they be done with or without a load? Due to safety concerns, most people agree that surveys in atmosphere furnaces should be run without an atmosphere present. The subject of using empty racks or workloads is more contentious. Some feel a survey using baskets or empty racks represents a worst-case scenario for uniformity. Others feel that an empty furnace does not mirror production conditions.

Commercial heat treaters often prefer using empty racks because they cannot predict what a typical load size or configuration will be. It is important to note that heating profiles should duplicate process parameters (i.e. do not slow down ramp to avoid overshoot with an empty furnace). Auditors will look for this type of detail. For captive shops (such as those running automotive components) that heat treat the same size and type loads every day, it may make more sense to run the survey with a load as it is more representative of production conditions. In addition, the heat-up data can be used to determine how long it takes a load to reach temperature so as to better predict soak times.

Conducting A Successful TUS and System Accuracy Test (SAT) in Atmosphere Furnaces

Here is some advice gathered from years of doing surveys and from a number of industry experts on what you should watch out for when conducting a TUS in atmosphere furnaces. Although many of these comments apply to all types of furnaces, atmosphere styles are particularly challenging due to the amount of manual intervention needed to "feed" the thermocouple (T/C) wires through doors, etc. (Fig. 1). For this reason many companies offer devices that can be placed on a grid or in a basket and run

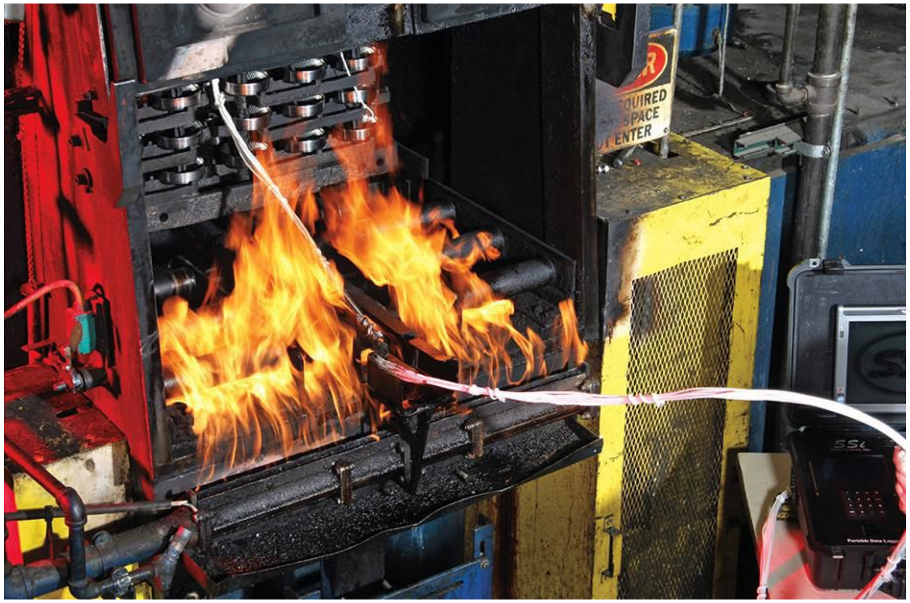

Fig. 1. Manual survey method for an integral-quench furnace (Courtesy of Super Systems, Inc.)

through the furnace. Be aware that there are certain restrictions (e.g., time at temperature) that apply to these devices

Survey Tips

1. Make sure the proper classification is given to each furnace. Keep in mind that one furnace can be classified and used in more than one way. You cannot use a furnace for a process for which it has not met temperature uniformity.
2. Take the proper number of measurements. Surveys require data points in two-minute intervals for each T/C, including clear evidence of ramp-up and clear evidence of soak (Fig. 2).
3. Be thorough. Make sure your records show all required details.
4. Make sure any resident SAT probes are not of the same T/C type as the control T/C. Also, the SAT thermocouple must be located within 3 inches of the control T/C to be valid. Have a calibration report for both the SAT probes and T/Cs. You now need to periodically perform SAT tests on freezers and quench oils (monthly is acceptable). Frequency of SAT depends on instrumentation type (as does TUS). You are now rewarded with longer times between surveys if you use load T/Cs.
5. Instrumentation calibration is mandatory. Make sure the company meets all calibration requirements for instrumentation. The certification must clearly state each is met, along with objective evidence. Each instrument parameter should be calibrated at least three points, and make sure the points calibrated cover the entire operating range.
6. T/C wire traceability is required. Calibration records are needed with clearly stated correction or error factors (these must be properly applied

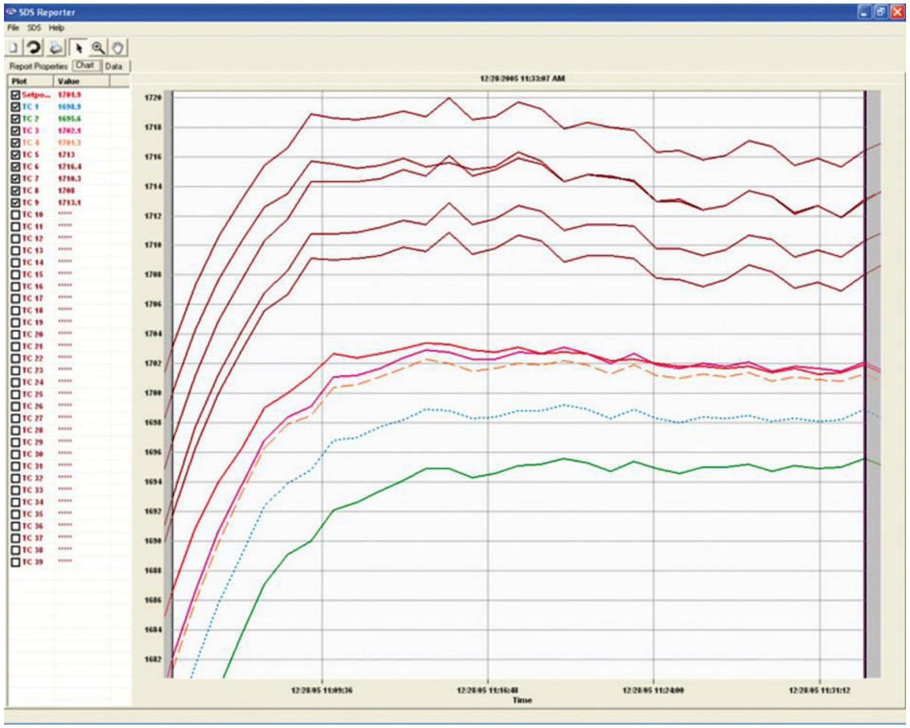

Fig. 2. Typical survey chart (Courtesy of Super Systems, Inc.)

algebraically). Make sure any rolls of wire greater than 1,000 feet are calibrated at both the beginning and end of the purchased roll (use average between beginning and end in TUS), and make sure the beginning and end values do not differ by more than ±2˚F (±1.1˚C).

7. Use Type N for survey T/Cs. In many cases, they are now preferred over type K, from an initial cost and re-use basis.
8. Selection and placement of T/Cs is critical. Attention to every detail demanded by the specification is mandatory.
9. Be sure to maximize the contact between the T/Cs and the rack, test slug or part. If the integrity of the contact between these two surfaces is not maintained, you might be logging an extraneous temperature instead of process temperature.
10. Do not weld heat sinks to the survey frame since the contact area makes the heat sink larger than it should be. Heat sinks have been found to make the difference with respect to overshooting and also the degree of overshoot.
11. Understand the impact of the T/Cs' thermal mass. Every T/C has an effect on the temperature or heating characteristics of the product being measured. It's important to match the T/C to the job required.
12. Understand the compromise that must be made between T/C life and maneuverability. For example, while 1/8 inch (3.0 mm) diameter T/Cs are common, they are more rigid than say 1/16 inch (1.6 mm) diameter T/Cs.
13. Know and record the position of the control T/C in relationship to the survey T/Cs. Control T/Cs located too close to the walls or too far into the chamber can cause large temperature variations

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and different response times. After surveying, if the control T/C was replaced with a different type, different gauge-thickness wire, different protection tube or located in a different location, the survey must be redone.

14. Clearly define the work envelope. Workloads or parts that fall outside survey dimensions must be considered having been run in a nonconforming piece of equipment.
15. Have procedures for performing a TUS clearly documented. Make sure that during an audit, procedures for the TUS activity are readily available and that the in-house technicians who perform the surveys know the procedures and where to access them. If a third party performs the survey, make sure that the quality department reviews and signs off on the paperwork prior to filing it.
16. Make sure the equipment being used meets the specified accuracy and readability requirements. AMS 2750, Table 3 defines a TUS device as a field test instrument. Calibration accuracy for the device is ±1˚F (±0.6 ˚C) or ±0.1% reading in ˚F, whichever is greater.
17. Have documented procedures on how correction factors are used. Make this procedure as simple a possible, and provide examples so that pyrometry personnel and auditors can easily evaluate survey results and documentation on corrections used.
18. Make reporting easy. Since surveys take time to perform, make the reporting process simple, consistent and repeatable so that survey results can be documented and signed off, and technicians can focus on spending their time getting equipment ready for production.
19. Don't try to qualify a furnace for uniformity that is not required. If you are only running parts that require a class-5 furnace (AMS 2750, ±25ºF (±14ºC) for example), don't try to achieve a uniformity that is tighter than that.
20. Design and fabricate a proper test rack. This is especially important when running a survey without a load. Be sure to place the T/Cs in the same location each time and run each TUS in as similar a fashion as possible.

Summary

Remember that each type of furnace is unique with different methods of construction, different heating/cooling capabilities and in a different state of repair. For this reason, we need standards that level the playing field. The intent of AMS 2750 and other specifications of this type is to ensure that one of the most common equipment variables, namely temperature, is taken into consideration in the overall quality scheme. Part Two will provide similar tips for vacuum furnaces while Part Three looks at ovens.

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Temperature Uniformity Survey Tips Part Two: Vacuum Furnaces

We have been discussing how to conduct temperature uniformity surveys (TUS) and system accuracy tests (SAT) to comply with specifications such as AMS 2750 (Pyrometry), and we want to continue to expand that discussion. Let's learn more.

Tips for Conducting a Successful TUS and SAT – Vacuum Furnaces

Here is some advice gathered from years of doing surveys and from a number of industry experts on what you should watch out for when conducting temperature uniformity surveys in vacuum. Specifically:

1. The advertised "working zone" must be the same size as any racks or setup used (Fig. 1). In the field, it is not uncommon to find survey racks or thermocouples placed well within the actual working zone of the furnace. Nadcap auditors, for example, often measure the rack and compare it against the advertised working zone. If the advertised working zone is bigger than the rack, it will be a major noncompliance issue.
2. Make sure the thermocouples (used) are correct to the certification of calibration. Correction factors can be as much as 2.2°C (4°F) or 0.75% of the reading (whichever is greater), and if the wrong correction factors are used, the survey can be highly inaccurate.
3. Make sure the location of the sensors is correct to the record. If performing a TUS, be sure adjustments made to bring the working zone into a better uniformity and the sensor locations are correctly logged. It is not uncommon to find people adjusting locations incorrectly, making it worse and wasting time.
4. When crunching the numbers afterwards, make sure the approach to temperature is included in the "high" reading calculation for the test sensors and any other recording sensor(s). A sensor could overshoot a little, and if you do not analyze the approach and include it in the report, you are not certifying the furnace accurately.
5. Make sure the thermocouple (TC) wires are not bent sharply or stretched too tightly when

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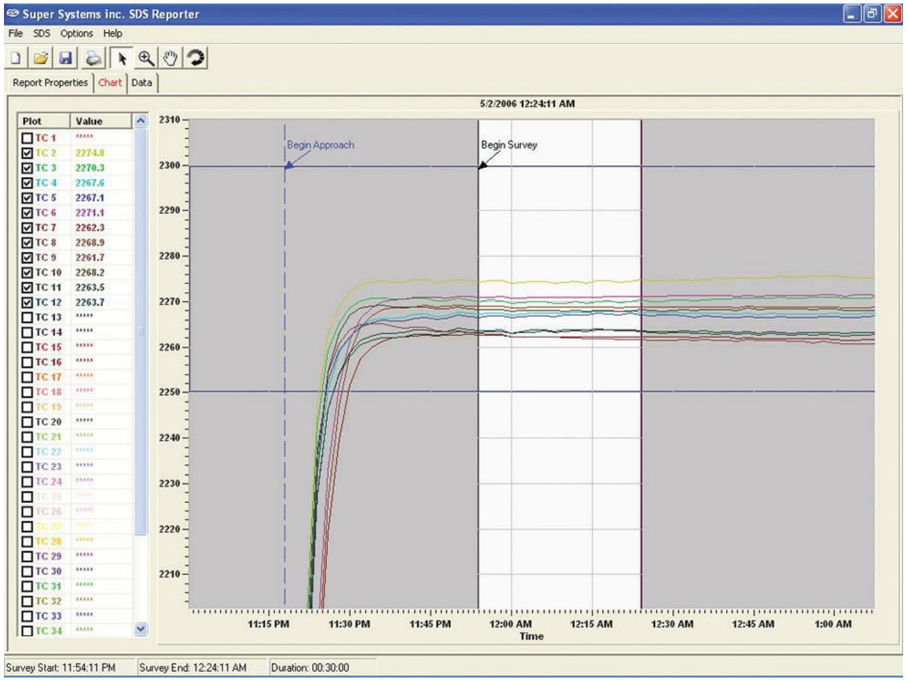
Fig. 1. Single-chamber vacuum furnace temperature-survey rack in position (Photograph courtesy of Solar Atmospheres Inc.)

routing them to their respective locations. The wires themselves go through phases of expansion and contraction. If too tight, they can become shorted during the process. A shorted reading will most certainly skew the results or cause a survey to be aborted.

6. Make sure to include the known deviation of the portable TUS recorder into the correction of the readings. An instrument may be off as much as 0.6°C (1°F) or 0.1% of the reading (whichever is greater). This could add a deviation of 1.1°C (2°F) at 1095°C (2000°F). If not compensated for, this can seriously affect the results of the TUS, and it could be a major finding in a Nadcap audit.
7. Do not "step" the ramp for a TUS (to prevent overshoot). It is never a good idea to attempt this in production either. For example, do not ramp 16.5°C (30°F) a minute to 510°C (950°F), then 5.5°C (10°F) a minute into the 540°C (1000°F) setpoint of the TUS. It is imperative that production ramp rates are used for the TUS (Fig. 2). An auditor knows this can be a bad habit and will typically look for this. Again, this is a major finding should it be noted in a Nadcap audit.
8. Always measure the insertion of the controlling thermocouple before each survey. It must always be the same, which is critical to setpoint versus uniformity distribution. If a thermocouple is replaced and inserted to a different depth, the uniformity from setpoint may change.
9. Thermocouple wire feed-through assemblies and jack panels are an insidious source of EMF error. If using a thermocouple jack



Plot	Value
☐ TC 1	*****
☑ TC 2	2274.8
☑ TC 3	2270.3
☑ TC 4	2267.6
☑ TC 5	2267.1
☑ TC 6	2271.1
☑ TC 7	2262.3
☑ TC 8	2268.9
☑ TC 9	2261.7
☑ TC 10	2268.2
☑ TC 11	2263.5
☑ TC 12	2263.7
☐ TC 13	*****
☐ TC 14	*****
☐ TC 15	*****
☐ TC 16	*****
☐ TC 17	*****
☐ TC 18	*****
☐ TC 19	*****
☐ TC 20	*****
☐ TC 21	*****
☐ TC 22	*****
☐ TC 23	*****
☐ TC 24	*****
☐ TC 25	*****
☐ TC 26	*****
☐ TC 27	*****
☐ TC 28	*****
☐ TC 29	*****
☐ TC 30	*****
☐ TC 31	*****
☐ TC 32	*****
☐ TC 33	*****
☐ TC 34	*****

Survey Start: 11:54:11 PM
Survey End: 12:24:11 AM
Duration: 00:30:00

Fig. 2. Typical temperature uniformity survey chart (Photograph courtesy of Super Systems Inc.)

panel, make sure the panel is absolutely clean between the legs as even the slightest film could produce errors in the readings (Fig 3). Always keep dummy plugs in unused jack locations. The wires from the chamber feed-through to the back of the jack panel are also a potential source of error. Fiber insulation attracts and holds contamination and water vapor that can bridge the gap between the wires, causing a slight EMF error. It is important to keep in mind that even on the more forgiving type-K thermocouple, the difference between 300°C (572°F) and 301°C (574°F) is a mere 0.041 millivolts. It doesn't take much contamination to cause an error in the difference between the hot and cold junction of a thermoelectric circuit.

10. With a calibrated run-up box (thermocouple generator source), inject a signal into the jack panel to simulate a given temperature through every channel to ensure correct readings back to the official recording system. On vacuum systems, temperatures in the ranges of 93°C (200°F), 538°C (1000°F), 800°C (1475°F) and 1205°C (2200°F) are not uncommon input signals.
11. On average, TC jack-panel feed-through plates should be replaced every year as the female connecters become impregnated with process debris and hot-zone trace elements. Coupled with this, the springs become annealed causing loose connections and subsequent errs in the readings.
12. Be absolutely certain that the instrument used to calibrate the portable TUS recorder meets the accuracy requirements of a

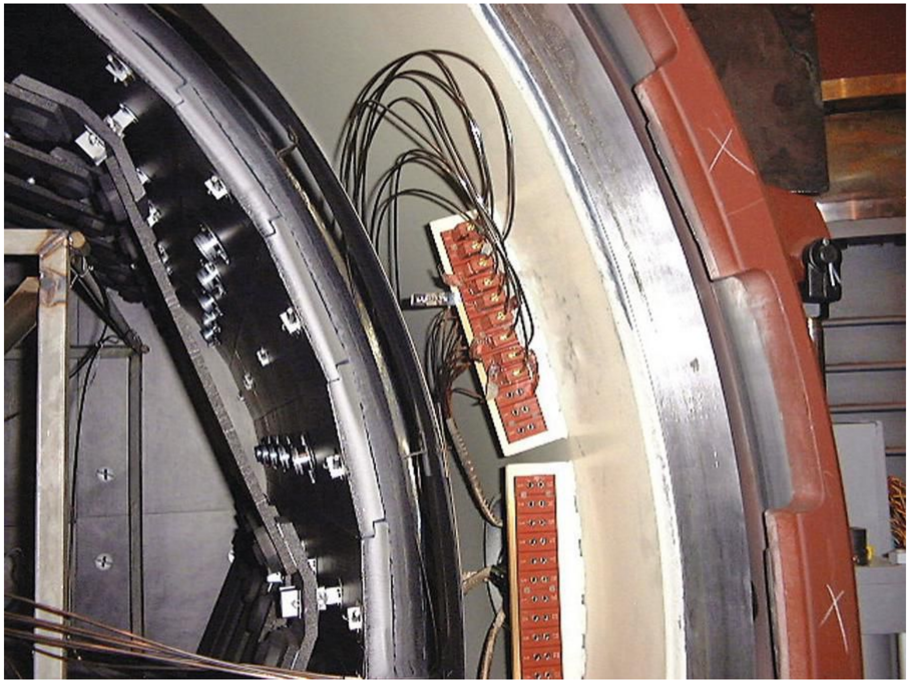


Fig. 3. Typical jack panel (Photograph courtesy of Ipsen USA)

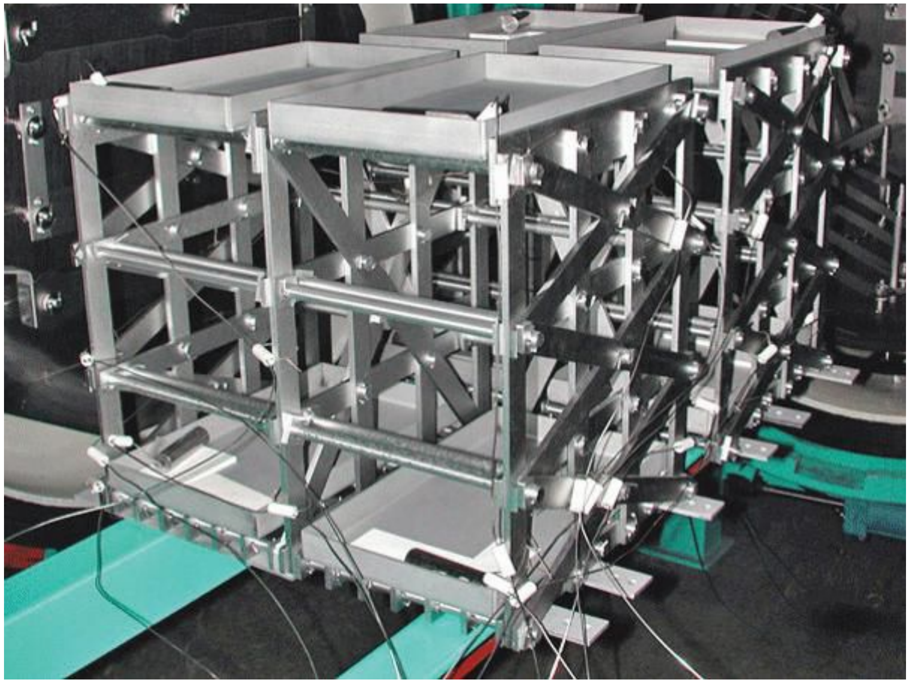


Fig. 4. TUS rack with metallic-sheathed thermocouples (Photograph courtesy of Vac Aero International Inc.)

"secondary standard," which is ±0.2°C (±0.3°F) or ±0.05%, whichever is greater. Many common field-test instruments do NOT meet this accuracy requirement. If you outsource the calibration, you should always have a current copy of the calibration from the field test instrument (used) and make sure it meets this requirement. In addition, the calibration should have been performed as the instrument is used. For example, if the field-test instrument is used as a thermocouple simulation source (sending the temperature signal to the device being calibrated), the appropriate thermocouple types should be checked while the instrument is in the source mode. Having a calibration certificate that shows how accurate an instrument is in read mode does not always qualify it in the source mode. Communicating these kinds of requirements to your supplier is very important to getting the service you require to pass your Nadcap audit without findings.

13. First, check the element resistance to ground. For example, one manufacturer's equipment is designed for 87-110 ohms prior to attempting a temperature survey. Do not attempt a survey if the resistance is bad on any particular zone. The readings should be fairly balanced prior to attempting a survey.
14. Ensure load TCs are new and preferably (not required) from the same lot of source wire for ease of tracking deviation factors.
15. Make sure that your load TCs maintain correction factors that are less than the survey spread allowance. For example, surveying for a spread of ±5.5°C (10°F) and utilizing load TCs with correction factors of ±6°C (11°F) or higher is not desirable.
16. Extrapolating setpoint temperatures on load TCs in steps greater than 120°C (250°F) are not allowed. Load TCs for vacuum furnaces tested to AMS 2750 are supplied with certification sheets typically from 540°C (1000°F) up to 1205°C (2200°F) in 120°C (250°F) steps. Hence no extrapolation required.
17. Before starting a TUS survey, check that the furnace has recently passed its reference probe check.
18. Check all element connections to be sure that they are tight and show no signs of arcing.
19. Remove all lower-temperature materials and alloys from previously processed runs prior to conducting surveys.
20. Where possible, use metal-sheathed load TCs (not cloth versions) (Fig. 4). Nadcap may ban the use of cloth TC in vacuum environments due to the vaporization of the actual TC materials coupled with impregnation of process (vaporized trace elements, which all lead to downward temperature readings).
21. To prevent transient noise from reaching the control system, use load TCs that are ungrounded.
22. Always utilize a temperature-survey fixture with appropriately sized heat sinks unless otherwise specified by a particular customer-driven internal specification.
23. Always center the temperature-survey fixture front to back and left to right for consistencies in readings and repeatability in future surveys.
24. Design the load-survey fixture and heat the furnace at a ramp rate and vacuum level that

best emulates your process requirements.

25. After heating to the first test temperature, allow the furnace ample time to reach steady-state power consumption prior to entertaining any adjustments. For most vacuum furnaces this will be approximately one hour at the first setpoint.
26. Observe the temperature spread throughout the load at a given setpoint before making adjusts (e.g., manual trimming, PID values and offsets). Remember that the best adjustment strategy may depend on instrument type or manufacturer.
27. If available, adjust zones in order to balance the load front to back and/or top to bottom again depending on particular product line and manufacturer. Adjustment must be made slowly at the lower temperatures and one zone at a time. The cause-and-effect relationship from these adjustments is not realized for at least 15 minutes after the adjustments are made. Patience is a virtue that manifests itself when running temperature surveys.
28. All adjustments on an older system need to be made and locked at the lower temperature. Newer systems fall under slightly different guidelines.
29. One should never be too concerned about deviation from setpoint when making the preliminary trimming adjustments. Remember that trimming yields uniformity while control TC placement rectifies load-to-setpoint anomalies.
30. Once trimmed at say 540°C (1000°F), the low TC reading should be at least 540°C (1000°F) while the high TC reading is still within the required uniformity range. The goal is to have the mean within specification but on the high side of the lowest setpoint, as this ratio will drift downward during incremental upward temperature steps through the survey.
31. Prior to moving away from the 540°C (1000°F) adjustment permissible range, the mean of the load is now adjusted to reduce deviation from actual setpoint by adjusting the control TC either inward or outward depending on what direction the survey fixture needs to be adjusted to be compliant to control.
32. Each progressive setpoint will take less time to soak out because the radiation levels are increasing over the black-heat area of the 540°C (1000°F) range.
33. Be sure in-house and AMS 2750 documents for sampling rates, time at temperature and stabilization periods are compatible.

Summary

The requirements of AMS 2750 for temperature uniformity testing are an excellent place to start when developing a statistical capability study for any type of heat-treating furnace or oven. These types of surveys give the heat treater a greater level of confidence that the equipment will be able to perform to the required temperature tolerances. When working to the requirements of AMS 2750, proper planning avoids failing a survey. If a survey fails, all jobs that were run in this furnace to the AMS 2750 requirement would have to be reviewed for affect and disposition. In a job-shop environment especially, this can be a daunting task. Using statistical analysis techniques to monitor the temperature uniformity can eliminate such discrepancies.

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Temperature Uniformity Survey Tips Part Three: Ovens

Airflow, airflow, airflow" is to the world of ovens what "location, location, location" is to real estate. Temperature uniformity in ovens is something that is often assumed, but it needs to be checked on a routine basis. One of the most common problems witnessed in the field by the knowledgeable observer is the number of older ovens (some dating back over 60 years) and ovens in terrible shape still being used in critical applications. Many times ovens are overloaded or restrictions on uniform temperature areas ignored. How do we do it right? Let's learn more.

Tips For Proper Oven Operation

Here is some advice gathered from years of working to enhance oven performance and doing surveys and from a number of industry experts on how to ensure proper operation and what you should watch out for when conducting temperature uniformity surveys (TUS) in ovens.[1] Specifically:

1. Deliver the air where it's needed. So-called combination airflow (Fig. 1) ensures that heat is uniformly distributed evenly along the length of the chamber. In this flow pattern, air is delivered along the full length of the work chamber via supply ducts located on the sidewalls. A return duct normally located in the roof helps to evenly remove the air after it passes over the load. Both the supply and return ducts should have adjustable louvers in order to achieve the best uniformity. Horizontal airflow is also common. Here, all the air is delivered to one side of the heating chamber, flows across the parts and returns on the opposite sidewall. This design is used for multi-layer loads (e.g., shelf or rack ovens) that would prevent air from passing vertically through the load.
2. Maintain a positive pressure inside the oven. To operate efficiently and achieve good temperature uniformity, the pressure inside the oven must be neutral or slightly positive. The proper balance prevents cold air from being drawn in. To check your oven's pressure balance, use a pressure gauge calibrated in inches of water column (inches w.c.) or Pascals (Pa) mounted through the oven wall.

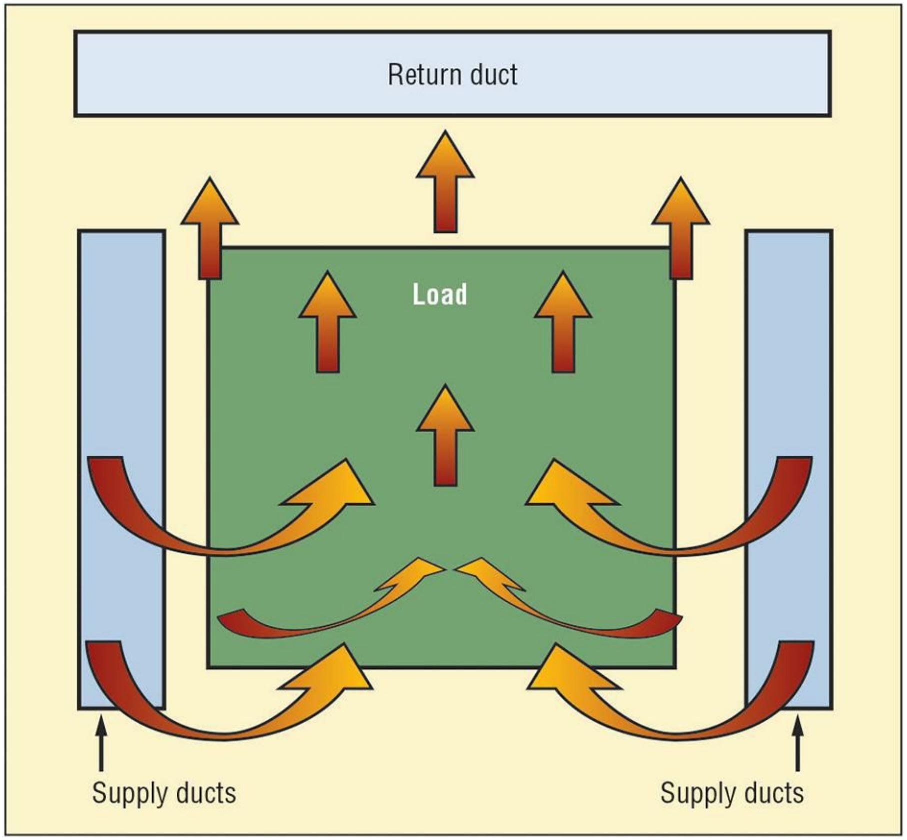

Fig. 1. Combination airflow oven design

A well-balanced oven will show a positive pressure of 0.0-0.2” w.c. (0-49.8 Pa) in relationship to the ambient conditions.

3. A simple method of checking to see if you have proper air flow throughout the oven work chamber is to tie ribbons or hang pieces of yarn onto parts and fixtures. Then with the oven at room temperature and the door blocked by a Plexiglas sheet (so you can see through it), turn on the fan and observe the pattern of the streamers. Adjust louvers as necessary to improve the flow pattern, then remove the test rig and perform a TUS before returning the oven to service.
4. Leakage around the door can also be checked using streamers. If the streamer hangs limp or is blown slightly outward by air escaping from below the door, the oven balance is usually acceptable. If the streamer is being sucked in, the oven has a negative pressure, which is undesirable. The balance can be corrected by adjusting the exhaust and air inlet dampers. If the oven pressure is negative, open the inlet damper or close the exhaust damper until balance is achieved.
5. Work with your supplier to ensure adequate airflow is inherent in the design. An oven with insufficient air will not achieve proper temperature uniformity and be a poor performer overall. Generally, the greater the volume of air circulated, the tighter the temperature uniformity. Work with your selected oven vendors to understand how they

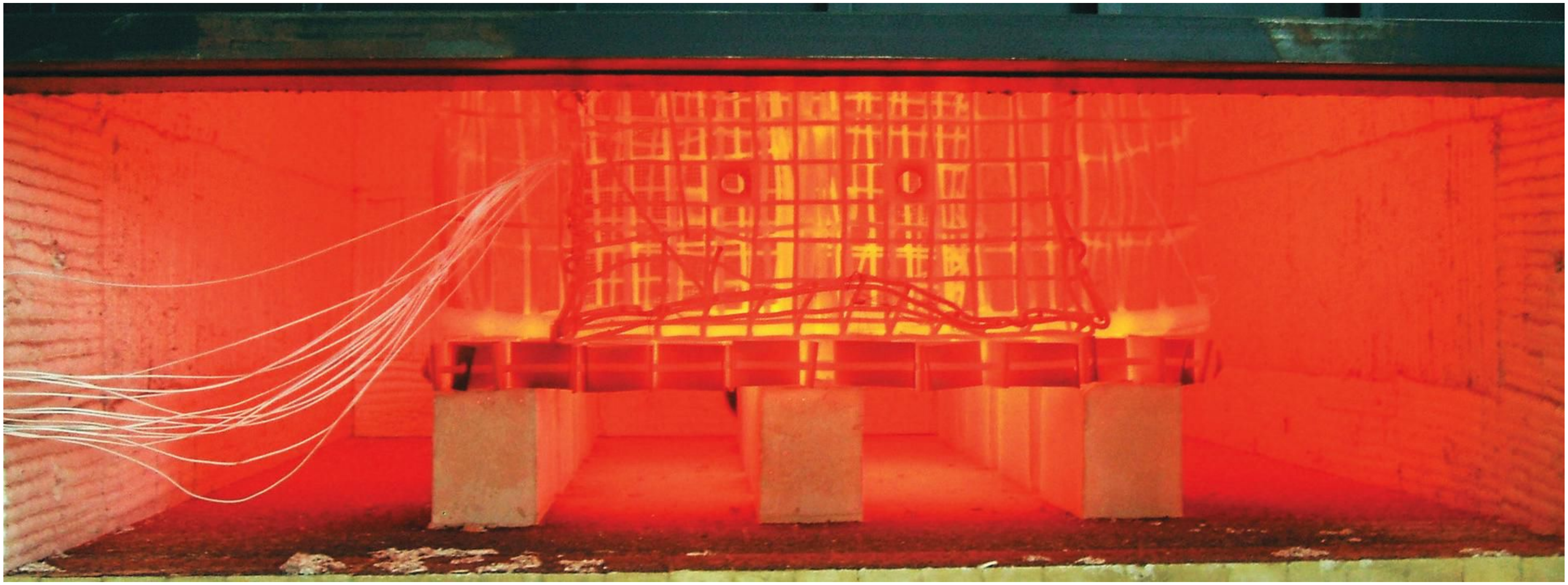

Fig. 2. Oven temperature-uniformity survey (Photograph courtesy of Super Systems Inc.)

calculate airflow and what exit velocity can be expected coming out of the ducts (so you can measure it over time once the unit is in the field). Increasing the blower RPM can improve the uniformity as well (this is also true for existing ovens). Finally, pay attention to the style of adjustable duct louver and the ease of its adjustment. These are key to long-term temperature uniformity.

6. Set the intake and exhaust dampers properly. One of the most overlooked items when running ovens day in and day out is damper adjustment, both intake and exhaust dampers. It is not uncommon to find that these settings have never been adjusted and that no one can recall how they were originally. Static pressure inside the oven and velocity in the ductwork can be negatively affected.

7. Pay attention to exhausters. Ovens, especially those running workloads that involve potentially combustible/flammable gases or other hazardous materials, are normally equipped with exhausters designed to dilute the mixture to non-explosive limits. Exhausters pull a tremendous amount of air through the oven, which can have a dramatic influence on heating and temperature uniformity. Remember that ovens should be tested under the conditions in which they will be used.
8. Size ovens properly. It is too often the case that large loads are placed in oven chambers that are too small for them or small loads placed in large ovens. In either case there is a danger that the loads will not heat uniformly. The oven should be sized properly for the load being processed.
9. Perform regular TUS. It is important to understand that operating conditions change over time, and ovens (just because they operate at fairly low temperatures) are no exception. As time passes, fans go out of balance, belts stretch and wear, heating elements burn out, door seals become frayed and worn, louver alignments need to be changed, louvers become damaged and air passages can become restricted. Separately or in combination, these factors lead to a loss of temperature uniformity. So performing TUS (Fig. 2), perhaps even more frequently than specification requirements, is very necessary.
10. Understand temperature rating. Ovens come in a variety of temperature ranges. It is not uncommon to purchase ovens designed for a limited temperature. Heat input is often limited as is the amount of insulation used. Most ovens operating up to 500˚F (260˚C) will have at least 4 inches (50 mm) of insulation, typically mineral wool. Ovens operating in the range of 500-1000˚F (260-538˚C) require a minimum of 6 inches (75 mm) of insulation. Today, ovens can be rated as high as 1400˚F (760˚C), so insulation type and thickness are important considerations for good temperature uniformity. If purchasing a new oven, remember that adding an additional 1 or 2 inches of insulation is an inexpensive way to improve uniformity. In older ovens, hot spots (often indicated by peeling or discolored paint) are signs of areas where insulation has deteriorated or compressed and are often indicators of temperature-uniformity problems.
11. Properly arrange the load in the oven. To

maximize even heating, the parts must be arranged in a manner that allows the heated air to reach all areas. If racks or shelves are used, be sure they elevate the load off the floor and do not block the air. There also must be sufficient space between each layer for air to circulate and transfer the heat properly. This sounds obvious, but overloading is frequently a hidden cause of heating and uniformity problems. In addition, the cart or racks should be constructed from as lightweight materials as possible to minimize the energy required to heat them.

12. One size does not necessarily fit all. To ensure good temperature uniformity and even heating, the oven chamber should only be slightly larger than the load being heated. For example, in a small or large oven air may pass around rather than through the load, resulting in poor uniformity between the outside and the center of the parts being treated.

Summary

Ovens are simple machines, really. Their fans need to be well maintained, balanced and operating within their design parameters. Louvers need to be properly adjusted, and workloads need to be placed within them with adequate space for good airflow for convective heating. If you pay just a little attention, you will find that ovens can easily be made to conform to the requirements of AMS 2750D and other temperature-uniformity requirements.

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A Quench-and-Temper Technique for Evaluating Carburized Case Depths

They say that you can't teach an old dog a new trick, but what about teaching a new dog an old trick? Did you know there is a simple and highly accurate shop-floor technique to measure case depth that does not involve microhardness testing or carbon-bar analysis? Let's learn more.

The Method

The method was developed by Rowland and Lyle[1] and rests on the principle that you can create a clear and precisely measureable line of demarcation (Fig. 1) between tempered martensite formed in a quench bath held for a short time at the martensite-start (M_s) point of the steel and the freshly formed martensite produced by subsequent water quenching.

The technique involves taking a small specimen from a carburized load and re-austenitizing it followed by quenching into a bath maintained at the martensite-start point of the steel corresponding to the carbon content at which the case-depth measurement is desired. The method is quick for a large number of samples and has good repeatability.

The Theory

The method relies on the principle that a properly austenitized sample can be quenched into a bath maintained at the M_s temperature corresponding to the carbon content at which the case measurement is desired, held at this temperature for a very short time (typically seconds) and water quenched to achieve a line of demarcation between the case and core. Case depths measured at various carbon levels typically range from 0.40–0.70% C.

The effect of carbon on the M_s point is independent of either the type or amount of alloying elements associated with it. The effect of elements other than carbon on the M_s point is sufficiently small that normal variation of analysis – from heat to heat within a given type – can be generally ignored and a single set of conditions used. Chemical segregation or extreme variations of both manganese and chromium in the same direction away from the middle of the range can affect local M_s temperature and have to be taken care of by slight adjustments.

A great deal of work has already been done to

Table 1. Steel grades and test conditions

Steel	Thickness Inches (mm)	Austenitizing Conditions		Quench-bath conditions at the indicated carbon level							
				0.40% C		0.50% C		0.60% C		0.70% C	
		Temp °F (°C)	Time, min	Temp °F (°C)	Time, sec	Temp °F (°C)	Time, sec	Temp °F (°C)	Time, sec	Temp °F (°C)	Time, sec
1018 1020	0.0625 (1.60)	1550 (840)	10	635 (335)	5	570 (299)	10	510 (265)	10	450 (232)	10
3310 3312	Open	1650 (900)	15	450 (232)	10	390 (199)	20	320 (160)	20	260 (127)	20
4028	0.0625 (1.60)	1550 (840)	10	635 (335)	10	570 (299)	10	510 (265)	10	450 (232)	10
4320	Open	1525 (830)	15	550 (288)	30	490 (254)	30	430 (221)	30	375 (190)	30
4620 4720	Open	1525 (830)	15	580 (304)	30	515 (268)	30	460 (238)	30	400 (204)	30
4820	Open	1525 (830)	15	515 (268)	60	460 (238)	60	390 (199)	300	335 (168)	300
8620 8720	0.250 (6.00)	1525 (830)	15	595 (313)	10	530 (277)	30	480 249)	30	415 (213)	30

accurately determine the M_s temperature for most steels.[3] It is well known that the M_s temperature is strongly dependent on the composition of austenite, the parent phase. The effect of individual alloying elements upon the M_s temperature for iron-based (binary) alloys has also been extensive studied. The results show that Al, Ti, V and Co effectively increase the M_s temperature, whereas Nb, Cu, Cr, Mo, Ni, C and N cause it to decrease.

The Process

Typically, a small sample (e.g., 2.25-inch OD x 1.25-inch ID x 1.25-inch long) is austenitized in either a standard laboratory box furnace or a salt bath. If a box furnace is used, the samples are typically coated with either a boric-acid slurry or a stop-off paint to prevent decarburization. The samples are heated and held long enough to produce complete carbon solution at the desired carbon level. The sample is then rapidly

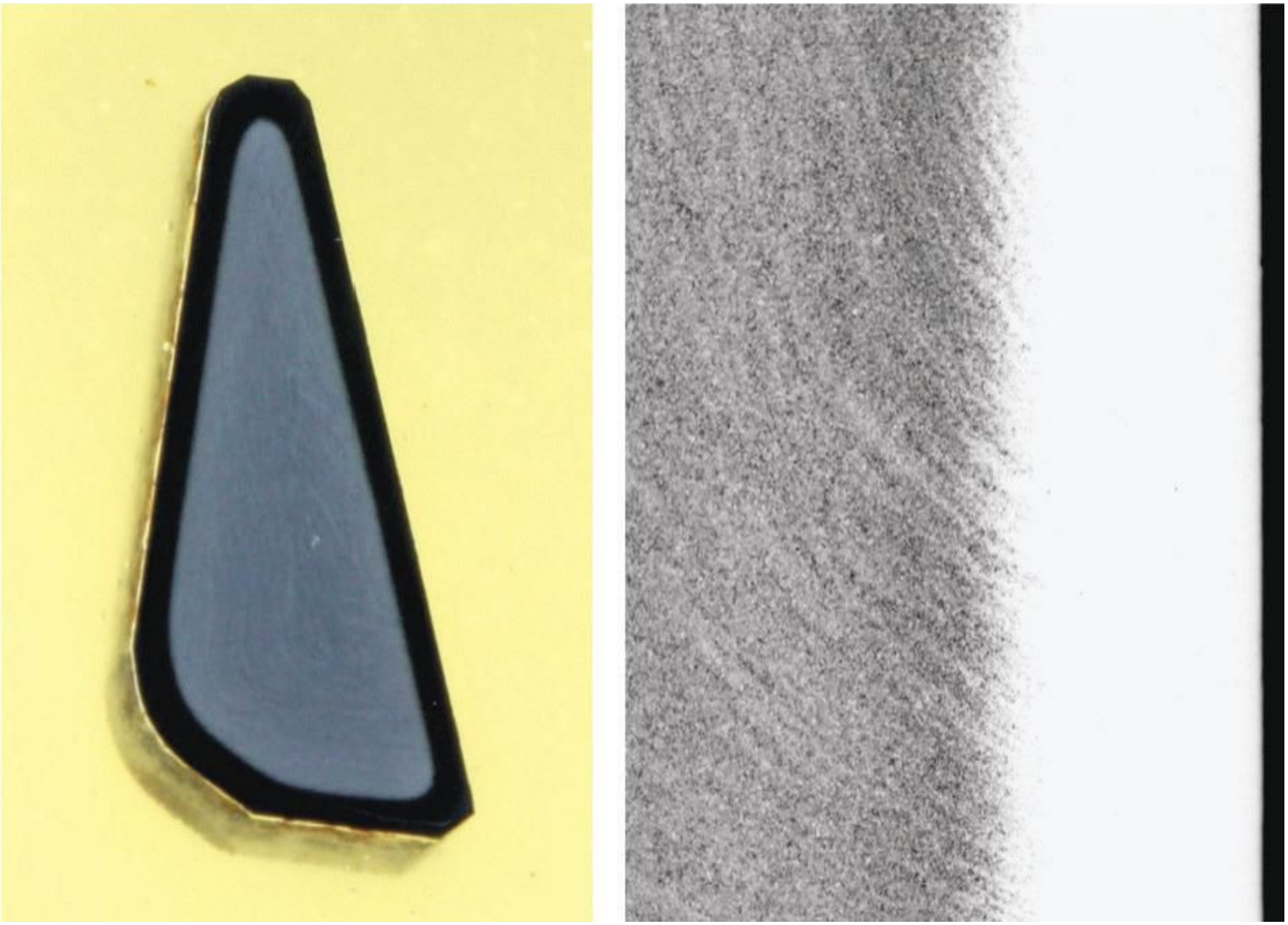

Fig. 1. Case/core demarcation by Ms point method (0.50% C sample) (a) Ms test specimen (2-3X) that has been heat treated, mounted, polished and etched. The lighting makes the unetched (fresh martensite) zone on the outer edge look black, but it is actually a shiny polished surface. (b) Close-up of the demarcation zone between the etched (tempered-martensite) zone and the unetched (fresh-martensite) zone. The line of demarcation is not exact. Generally, the depth is "read" using a Brinell scope at approximately 12-15X. When you read the depth, you read to the depth where the tempered martensite layer is consistent (not to the outer edges of the tempered martensite wisps). At 12-15X the line is not as fuzzy and the depth can be pretty consistently measured to ±0.001 inch (0.025 mm) or so.

quenched into a salt or oil bath and held for the appropriate temperature/time (Table 1) prior to water quenching. For higher M_s temperatures, salt quenching is common. For lower temperatures (up to around 300–350°F), oil quenching is often used (provided the oil doesn't smoke too much).

This technique works for any steel for which you can calculate or experimentally determine the M_s temperature for the desired carbon level. The technique is easy to perform and does not restrict control of conditions except on very low-carbon or low-alloy steels. It is important to hold at the isothermal quench temperature longer at lower temperatures so that the martensitic portion formed at that temperature will isothermally temper and etch dark (2% nital) while the outer portion, which remains austenitic until the final water quench transforms it to fresh martensite, does not etch. At higher isothermal hold temperatures, the auto isothermal tempering occurs more rapidly, reducing the need for long hold times.

Pros and Cons

The biggest advantage of this test is that it is quick and easy. It is also highly accurate for carbon levels above 0.40%.

One of the great advantages of this method is that the line of demarcation between the tempered and freshly formed martensite (due to quenching) is sufficiently sharp to permit the average heat treater to determine case depth within 0.002 inch (0.050 mm) using a simple calibrated loop or low-power stereomicroscope. This level of accuracy is true whether comparing to microhardness or carbon-bar (gradient) methods.

Further, none of the test conditions require close control (except for carbon and very low-alloy steels); a change of 6°F (3.5°C) represents only one point (0.01% C) of carbon. And almost any sized specimen convenient for (hand) polishing can be used. Finally, the effect of elements other than carbon on the M_s point is sufficiently small that normal variation of analysis from heat to heat can generally be ignored allowing a single set of parameters to be used.

The principal limitation of this method lies in its application to low-carbon (e.g., SAE 1018/1020) and very low-alloy (e.g., 4028) steels because of bainite formation in the constant-temperature bath. In these cases, a maximum specimen thickness of 1/16 inch (1.60 mm) is required, and this presents both difficulties in handling and control of quench-bath temperature. For example, a 1020 steel quenched to the 0.40% C level at 635°F (335°C) held for three seconds is too short a holding time for good delineation, five seconds is satisfactory while 10 seconds is too long (completely obscuring the line of demarcation because of bainite formation). Precautions should also be taken to avoid pearlite formation on quenching as well as to ensure complete carbon solution at the desired level of measurement before quenching.

Finally, the dividing line between the higher-carbon fresh martensite and the lower-carbon tempered martensite is often not a perfectly straight line, and different operators will read slightly different values depending on where they judge the line of demarcation to be. Readings can vary from operator to operator, especially if the depth is determined by a Brinell scope.

Tips

To make the job easier:

1. Samples must be cut before applying this technique and not afterward to avoid tempering the freshly quenched martensite.
2. Good etching practices are essential as the sample should be etched just enough to develop maximum contrast.
3. Material that is heavily banded can still be evaluated using this method, but tight control of the process parameters is mandatory.

Summary

This method is simple, straightforward and can be easily performed by shop personnel, making it a valuable addition to the heat treater's arsenal of testing methods.

Acknowledgment

The author would like to thank Mr. Craig Darragh, senior product technologist – steel, The Timken Company, for providing both the idea and inspiration for this article.

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Hardness and Hardenability Part One: Tips and Techniques for Hardness Testing

For the heat treater, the concept of hardness is well known and the act of hardness testing routine. In fact, it is so repetitive that at times it is taken for granted, leading to sloppy procedures and false readings. Today, the ability to test that the heat-treating processes have achieved the proper hardness is more important than ever. Let's learn more.

Hardness measuring methods can be divided into three general categories depending on the manner in which the tests are conducted: scratch hardness, rebound (dynamic) hardness and indentation hardness. For most of us, hardness is simply the measure of the resistance of a material to a force applied by a hardness tester and involves the use of an indenter of fixed geometry under static load (Fig. 1). The ability of the material to resist plastic deformation depends on the carbon content and microstructure of the steel. Therefore, the same steel can exhibit different hardness values depending on its microstructure, which is influenced by the cooling (transformation) rate.

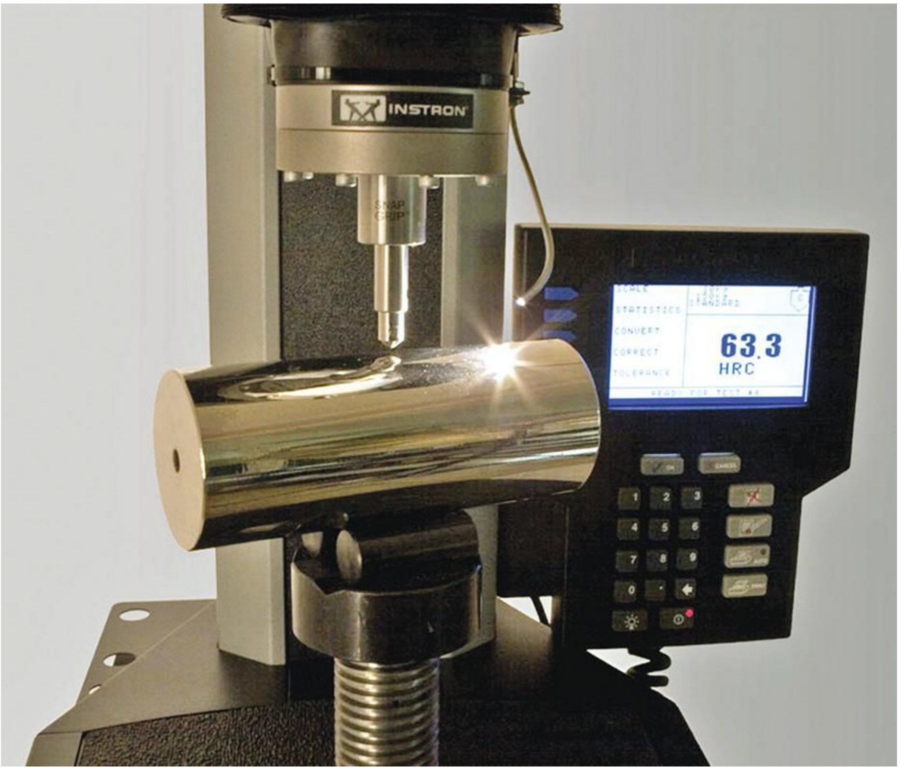

Fig. 1. Typical hardness tester (Photograph courtesy of Wilson® Instruments)

Hardness testing is one of the most common (and often the only) quality control check performed. It is relied upon to determine the success or failure of a particular heat-treatment operation or to understand the material's current condition. Hardness testing is one of the easiest tests to perform on the shop floor or in the laboratory, but it can be one of the hardest tests to do properly.

Tips for Successful Macrohardness Testing (Rockwell®, Rockwell® Superficial, Brinell, etc.)

A number of simple rules should be followed when hardness testing. These include:

1. Select the proper hardness scale. Understand which scales are best suited for which materials and be careful when hardness testing at the extreme end ranges of a particular scale. For example, performing Rockwell testing in the 90 HRB to 25 HRC scale might best be performed using the "A" scale.
2. Use your calibrated test block (and be sure to select the anticipated hardness range). As a minimum, calibration blocks should be run at the beginning and end of each day, preferably at the beginning and end of every shift. In extreme situations, calibrations might need to be run before and after a particular set of hardness tests.
3. Clean the part and tester. Take the time to remove and clean the indenter and anvil prior to each operation and at shift change. A small amount of debris can alter the reading by several points.
4. Check the curvature of the part surface. A correction factor must be added to the hardness reading of small-diameter shapes and varies with the scale, apparent hardness and part diameter. Wall charts are available from all major hardness-tester manufacturers that show the correction factors. ASTM E18 also lists these corrections.
5. Watch out for non-flat surfaces. Extremely rough or textured surfaces may give inconsistent readings. Lightly sand both the top and bottom side of a sample to ensure flatness and removal of all marks. Remove any scale, debris, dirt and oil before testing.
6. Keep surfaces perpendicular to the indenter. Surfaces should be flat within 2 degrees. Be careful when taking readings on mounted samples. They must be flat, thick and not flex (inside the mount) under load. A microhardness test may be more appropriate.
7. Do not take readings taken too close to the sample edge. Indentations should be no closer than 2½ times the indenter diameter from the edge. If the metal buckles outward, the indenter is too close to the edge and the reading is invalid.
8. Do not take readings too close together. Indentations should be a minimum of three diameters apart.

9. Beware of parts or section of parts that are too thin. Unless a special anvil is used, the material should have a thickness at least 10 times the depth of the indentation. ASTM E18 contains thickness guides.
10. Adequately support the parts. Large and irregularly shaped parts need to be well supported. Parts that move, even slightly during the test, produce a false reading. If possible, change the anvil to one that keeps the part stationary. Also, a component may require the application of lower loads because case depths are too shallow or samples are too small for the applied load. The specimen may not be able to physically support the hardness test load without deflection. Samples may need to be externally supported or even fixtured.
11. Check frequently for a damaged diamond or flattened ball. At least once a day, or if readings are suspect, remove the indenter from the hardness tester and inspect the tip using a low power (10-50X) stereomicroscope or loop.
12. Apply common sense. For example, poor lighting or lack of proper magnification on the sample are common sources of accuracy errors. Clamp-on accessories are available to correct these situations but are seldom used.
13. Throw out the first several readings after changing the indenter or anvil, even if they are within range.

Tips for Successful Microhardness Testing (Vickers, Knoop, etc.)

Microhardness tests are typically used for very small, intricate shapes, thin parts and case-depth determination. It helps to:

1. Be sensitive to where microhardness tests should be performed on the part. Microhardness tests are conducted on very small areas, so choose where to do the testing carefully. Also, testing machines can only accommodate samples or mounts within a certain size range.
2. Mount samples whenever practical. Where possible, mount and polish samples using standard metallographic techniques. There is a common misconception, however, that all samples must be mounted in order to be microhardness tested. This is not the case if a special jig or fixture is used.
3. Watch sample orientation. The sample or mount has to be flat so that the indenter is in contact with the surface evenly. Furthermore,

the sample surface to be tested must be perpendicular to the indenter.

4. Be aware of the effect of surface finish on readings. The condition of the surface affects the ability to read and interpret results.
5. Be aware of sample-preparation-induced effects. Certain materials are more affected by sample-preparation methods than others. For example, aggressive grinding of stainless steels or nonferrous alloys can work-harden material surfaces.
6. Provide the best surface possible for testing. While it is possible to microhardness test a sample after grinding (only), the accuracy of the test may be improved by polishing the specimen as well. Freedom from scratches should be the goal.
7. Be sensitive to vibration. Microhardness testers are sensitive to vibration, which can cause erroneous readings. Do not lean on the bench or table during the test. Location and shock mounting pads will isolate the tester from excessive vibration. Remember, microhardness testers seldom change locations, but the addition or movement of other equipment can change their environment.
8. Watch out for multiphase sample materials. A microhardness test is often used to show hardness gradients within multiphase materials. For general microhardness testing, a homogeneous sample is optimal, but in the case of a multiphase alloy, it is often necessary to take multiple hardness measurements so as to obtain an adequate sampling of different phases (grains) within the alloy. Microhardness testing of cast iron is another example. Often, the sampling scheme is determined on the basis of statistical calculation or understanding which areas to measure for representative results.
9. Try to establish the same testing regime. Ideally, the same operator would perform tests using the same testing apparatus. However, do not assume that your operator or quality control person is performing the tests or documenting results accurately – double check. Repetitive actions lead to problems such as improper rounding to the nearest integer, using the wrong conversion scales and the like.
10. Watch out for the springboard effect. Taking readings too close to the edge (<0.0025 inch, 0.06 mm) of a material with too heavy a load will cause erroneous readings. If lighter loads (<500 grams) are used, comparative results (only) are possible.

11. Remember, not all loads can be converted to Rockwell "C" readings. ASTM E140 states loads of 500 grams or above can be converted to Rockwell "C" readings. The conversion error is skewed higher with increased hardness or decreased loads.
12. Select the heaviest test load weight practical. The lighter the load, the more inaccurate the readings.
13. Know as much of the manufacturing history and heat treatment as practical. The manufacturing processes to which a sample is subjected prior to testing can cause microhardness data to display extreme scatter or be skewed.

Summary

Most people need not be experts in all the intricate details of hardness testing. However, it is important that the user selects the appropriate hardness-testing method and scale, considers part geometry and test location, and be aware of equipment limitations. Failure to do so can lead to improper interpretations of the true material condition, properties and hardness.

Should you find yourself in a dispute regarding hardness and hardness-testing methods, the first item to confirm is that the specified hardness is appropriate for that material. Next, investigate how the hardness was measured and if it was a suitable method for that sample. While there can be shades of gray and varying levels of uncertainty between hardness-testing machines or laboratories, expect some level of consensus if the methods are correct.

ASTM guidelines[1] state the Rockwell readings should be reported to the nearest integer. Although machines do vary (for example, if measuring on the Rockwell "C" scale), different machines should vary by no more than one HRC point of the accuracy range.

Everyone involved with hardness testing should have and be familiar with the appropriate ASTM specifications, including E3, E10, E18, E103, E140, E384 and others as necessary. These specifications address proper sample preparations, selection of loads and penetrators, sample geometry, minimum sample thickness considerations, roundness corrections, spacing and edge considerations and conversions between scales.

Part two will focus on issues concerning hardenability and hardenability testing.

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Hardness Testing: Problems and Solutions

Rockwell hardness testing is arguably the most common tool used by the heat-treating industry to measure the success or failure of a heat-treatment process. It is one of the seemingly easiest tests to perform on the shop floor, in the quality-control department or in the metallurgical laboratory but arguably one of the hardest tests to perform properly. Let's learn more.

What is hardness?

A common definition of hardness is the measure of the resistance of a material to an applied force and involves the use of an indenter of fixed geometry under static load. However, hardness can also refer to stiffness (temper resistance) or to resistance to scratching, abrasion or cutting. It can be thought of as the ability of the material to resist permanent deformation (i.e., to be bent, broken or have its shape changed) in service. The greater the hardness, the greater its resistance. It is important to recognize that the hardness of a material is highly dependent on its microstructure, however, which in turn is influenced by the heat-treatment process.

Hardness is not a fundamental material property but rather a composite value. As such, it is an indication that all is well, not a guarantee that it is so. It is of great interest because hardness can be directly related to the expected strength of the material, which would otherwise require destructive testing to measure. The location of the hardness indentations, away from working surfaces, is often an important consideration if hardness testing is to be considered a nondestructive test.

Hardness measuring methods fall into three general categories, depending upon the manner in which the tests are conducted: scratch hardness, indentation hardness (Fig. 1) and dynamic rebound hardness (Fig. 2). Rockwell hardness testing machines can be either benchtop or portable devices.

Rockwell Testing Tips

Many different factors can affect the results of a hardness test. In most instances, incorrect or misleading readings are the result of poor testing practices that violate simple yet basic testing rules. This leads to good parts being discarded or scrapped or, worse yet, bad parts being accepted and used. By following the rules, accurate hardness readings can

Fig. 1. Fixed-location indentation-style bench hardness tester

be obtained. To aid in this endeavor, we have gathered some of the most common Rockwell hardness testing problems and solutions faced by the heat treater (in no particular order).

1. Follow the testing guidelines outlined in ASTM E18 (latest revision), "Standard Test Methods for Rockwell Hardness of Metallic Materials" or the appropriate ASTM standard for the material being tested.
2. Always use a hardness tester that has an up-to-date calibration sticker from an approved outside service provider.
3. Use a test block to verify hardness readings. Typically, three readings are performed on a test block and the average compared to the value stamped on the block. This should be done daily. Never use a test block on both sides. It is intended to be used on one side only because diamond marks on the bottom side facing the anvil will cause incorrect readings. Old test blocks can be ground down, marked as scrap and used to "set the indenter" when either the indenter or anvil is changed. Remember, the first reading on a part after an indenter or anvil change should be discarded.
4. Inspect the indenter for damage (chipped or cracked diamonds or flattened balls) that will produce erroneous readings. Perform the inspection on a weekly basis by removing the indenter from the machine and inspecting the tip using a low-power magnification (10-50X) such as a stereomicroscope or jeweler's eye loop to check for damage. Flattened balls are sometimes difficult to detect unless you inspect all surfaces – often at an angle. If you see consistently high or erratic readings, perform this inspection immediately. An old heat-treater's trick is to

Fig. 2. Change "Portable ... tester" to read ""Equotip 550 display unit compatible with 3 hardness test methods - Leeb (rebound), UCI (Ultrasonic Contact Impedance), and Portable Rockwell Hardness (shown above)" (courtesy of Columbia Machine Inc.)

place one finger over the tip of the indenter when removing it to prevent accidental damage.

5. Cleanliness of the part and tester is paramount. Remove any scale, debris, dirt and oil on the part or the machine before testing. Even a small amount of debris can alter the reading by as much as several Rockwell points. Remove and clean the indenter and anvil prior to operation and at shift change. Lightly sand then clean both the bottom and top surfaces of the part before hardness testing.
6. Non-flat surfaces can alter readings. Extremely rough or textured surfaces (e.g., machining marks) may give inconsistent readings. Lightly sand both the bottom and top surfaces before hardness testing.
7. Take into account the curvature of the surface. Remember that a correction factor must be added to the hardness reading of small-diameter shapes for Rockwell A, C and D scales and varies with the apparent hardness and part diameter. The correction factor to be added is shown in the appropriate ASTM E18 tables. In addition, do not rotate a previous diamond mark downward toward the anvil because this indent will cause microscopic movement and a resultant low reading.
8. If the part moves, the reading is invalid (even if it is within the specified range). Discard these readings and do not include them in your average. In many instances the readings are recorded with an appropriate note as to why they are not being used.
9. Remember that a minimum case depth is

Table 1 - Minimum effective case depth required for accurate hardness test results

Effective case depth[a], in.	Hardness test method
<0.010	File test or microindentation
0.011-0.015	Rockwell 15 N scale
0.016-0.018	Rockwell 30 N scale
0.019-0.021	Rockwell 45 N scale
0.022-0.027	Rockwell A scale
>0.028	Rockwell C scale

[a] Case hardness must be SSHRC min.

needed to support a given Rockwell scale and produce a valid reading (see ASTM E18 for details). On the Rockwell-C scale, for example, a minimum case depth of 0.030 inch is needed to hold a 60 HRC reading. A shallower case depth may yield a soft reading, so changing to either the A scale or a superficial scale such as the N scale is appropriate.

10. Surfaces not perpendicular to the indenter will give false readings. Remember that surfaces should be flat within 2 degrees. Be careful when taking readings on mounted samples. They must be flat, thick and not flex under load. A microhardness test may be more appropriate.
11. Remember that the A scale spans both the Rockwell C and Rockwell B ranges and is often a good referee.
12. Readings taken too close to the sample edge may damage the indenter and will produce false readings. Per ASTM E18, indentations should be spaced no closer than 2.5 times the indenter diameter from the edge. If the metal buckles outward, the indenter is too close to the edge and the reading is invalid.
13. Readings taken too close together will give false (higher) hardness readings. This is also true on the test (calibration) blocks, where often one tries to place too many readings in one area to save the cost of buying a new block. Indentations should be spaced per ASTM E18 – three diameters apart.
14. Parts that are not properly supported will give false readings. Large and irregularly shaped parts need to be well supported. Parts that move, even slightly during the test, produce a false reading – even if that reading falls within the desired hardness range. Change the anvil to one that keeps the part stationary. Additional outside support devices (such as a Steady-Rest®) may also be required.
15. A sample that is too thin will yield false readings. The material being tested should have a thickness

at least 10 times the depth of the indentation. The minimum acceptable thicknesses can be found in ASTM E18 tables. Special (prehardened) anvils can be used when hardness testing thin sheet or foil material.

Finally, put up wall charts and have laminated cards in convenient places that show the various hardness testing scales and their relationships to one another. Almost all companies that manufacture hardness testing equipment offer them for free.

Summary

Most people need not be experts in all the intricate details of hardness testing. However, it is important that the user selects the appropriate hardness testing method and scale, considers part geometry and test location, and accounts for equipment and testing limitations. Failure to do so can lead to improper interpretations of the true material condition, properties and hardness.

Should you find yourself in a dispute regarding hardness and hardness testing methods, the first item to confirm is that the specified hardness is appropriate for that material. Then confirm how the hardness was measured and if the method was appropriate for that sample. While there can be varying levels of uncertainty between hardness testing machines or laboratories, expect some level of consensus if the methods are correct

For carburizing or carbonitriding, the required hardness scale that should be used to obtain accurate hardness test results is a function of effective case depth (Table 1).

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Trends in Mechanical Testing Methods

In the last few years, mechanical testing equipment and methods have evolved at a rapid pace, with impressive advancements in reliability, repeatability and accuracy. These tools and the software systems that drive them deserve a closer look by the heat treater, not only because they reveal far more information than we can gather from the usual tests performed on the shop floor, but also because they assure us that our heat treatment processes and procedures are correct. Today it is critical that we have a complete understanding of how our products perform. Let's learn more.

Understanding Mechanical Properties

Mechanical properties (Table 1) reveal the reaction of a material to an applied force. This reaction can either be elastic or inelastic in nature. Heat treaters and design engineers need to know how structural materials respond to external forces, such as tension, compression, torsion, bending, fatigue and shear. These forces result in stresses in the material, which may be tensile (pulling), compressive (pushing), or a combination of the two. The material may respond elastically or plastically, by creep or even fracture.

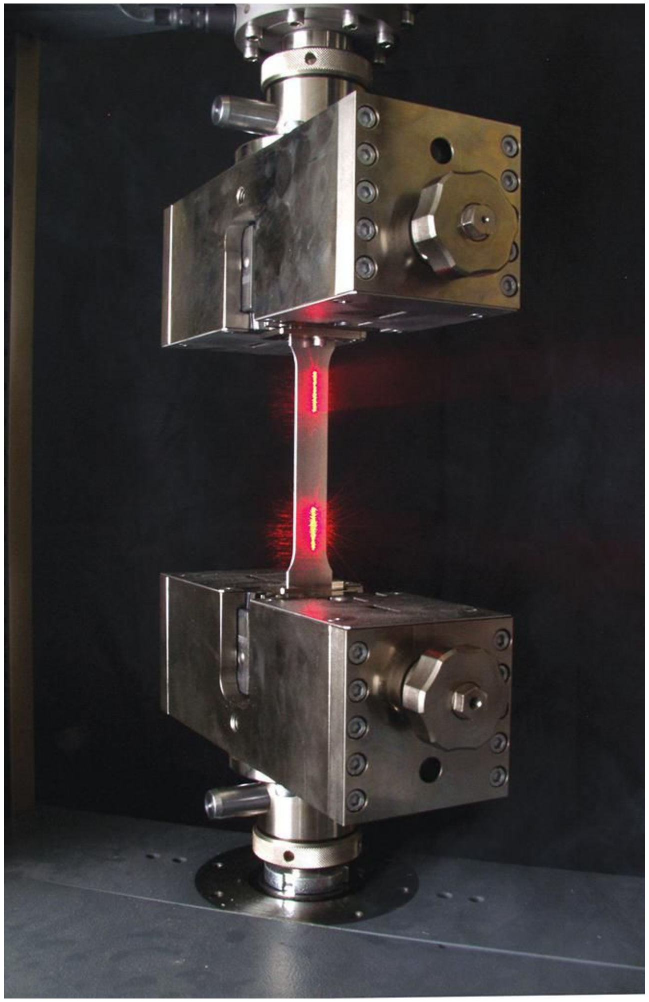

Fig. 1. OptiXtens laser extensometer. Courtesy of Zwick USA., Kennesaw, Ga.

Table 1 Tests to determine mechanical properties

Test method	Property	ASTM standard
Charpy, Drop, Izod	Impact, bending	E23-02a
Compressive load	Compressive strength; crush (collapsing) strength; elastic limit; proof stress; proportional limit	
Corrosion fatigue	Corrosion-fatigue limit	
Creep	Creep strength	E139-00e1
Cupping	Drawing	
Endurance	Endurance (fatigue) limit	
Fatigue	Fatigue limit; thermomechanical properties;	
Flexure	Modulus of rupture; cold bending	E290
Peel	Cracking; shear strength	E647-95
Penetration resistance	Hardness (static or dynamic)	E10; E18
Salt Spray	Corrosion	B117-03; G85
Shear	Shear strength	B565-04;
Tensile	Elastic limit, elongation, proof stress, proportional limit, reduction of area, tensile strength; transverse elongation and reduction of area; transverse tensile and yield strength; yield point; yield strength	E83-02 (Extensometers)
Tension	Strength, ductility	E8-03
Torsion (twist)	Torsional impact, torsional strength	A938-97(2003)

Standards such as ASTM A370-03a (Standard Test Methods and Definitions for Mechanical Testing of Steel Products) are valuable guides to test methods, procedures and definitions for the mechanical testing of wrought and cast steels, stainless steels and related alloys.

Selecting the right test methods and taking advantage of the latest testing equipment is important, since today these tests provide considerably more information than in the past. Mechanical testing in combination with metallographic analyses can provide a detailed picture of the entire manufacturing process seen by the part.

Today, two distinct paths have emerged in mechanical testing: macromechanical methods, such as tensile and impact testing; and micromechanical testing, such as nanomechanical testing. Let's see what's new in each area.

Trends in Tensile and Fatigue/Fracture Testing

A clear trend in tensile testing is the movement away from static systems and into testing systems operating in a dynamic mode. Dynamic tests include high- and low-cycle fatigue, fatigue crack growth and fracture toughness. Incorporation of robotics has made it possible to meet demands for higher throughput systems at reduced cost.

Recent research in fatigue testing has focused on the areas of micromechanisms and validation of computational models within the traditional test areas, such as corrosion fatigue, fatigue crack propagation, high-temperature fatigue and fracture, high-cycle fatigue, low-cycle and thermal fatigue, KIC and JIC fracture toughness, creep crack growth and fatigue testing.

A basic accessory for tensile testing is the extensometer. A material test system requires the capability to measure values such as load, strain, displacement and crack length, and to control their rate of change accurately. Many variables, such as load, can be measured from the load train rather than from the specimen. However, variables like strain or crack length are most accurately determined when measured directly from the sample by direct contact or noncontact extensometers. Having a broad measurement range and the ability to avoid a mechanical effect on the sample are highly important. Strain gauge or linear variable differential transformers are common contact types.

Eliminating the need to mark specimens is highly desirable since it removes a step in the testing process and increases productivity. In addition, marks fall off specimens, especially when testing in temperature chambers. Finally, some materials are so thin/sensitive that it is nearly impossible to mark them without damage.

An emerging trend involves noncontact extensometers using video or laser (Fig. 1) techniques for elongation measurement in tension and compression. Bob Donohue of Zwick USA feels that a unique aspect of this technology is the combination of very high resolution (0.1 mm) and large measurement range (500 mm), making it suitable for use on high elongation materials as well as determination of Young's modulus.

Trends in Impact Testing

The purpose of impact testing is to determine the toughness of a material by measuring the amount of energy absorbed by a specimen as it fractures while being struck by a moving pendulum at high speed. Although the basic equipment has remained essentially the same, the method of data acquisition has changed dramatically.

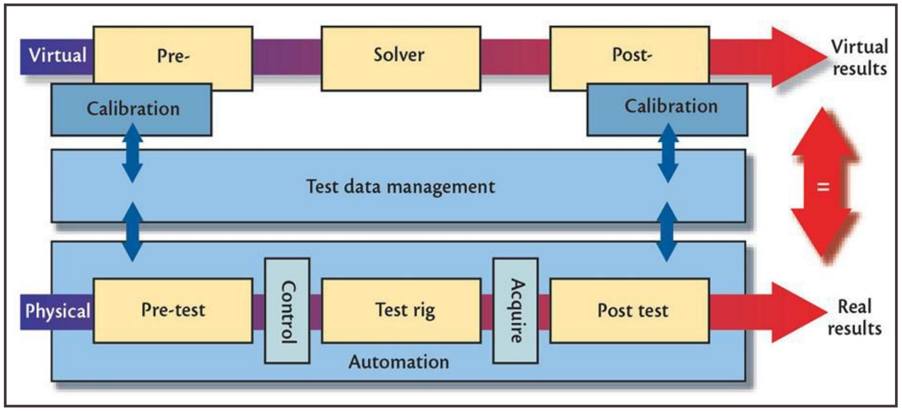

Fig 2 Synchronicity matrix. Courtesy of MTS Systems Corp., Eden Prairie, Minn.

Wayne Howard of Tinius Olsen Testing Machine Co. (Willow Grove, Pa.) sees an exciting trend in fully instrumented impact testers. In these machines, the striking tup is equipped with strain gauges, and high-speed data capture techniques can acquire up to 20,000 datapoints during the 10 ms test duration. A computer then creates a profile which, with a little imagination, enables us to "see" the striker as it progresses through the material giving an almost real time picture of the test and test conditions.

Trends in Nanomechanical Testing

At first glance, the word nanomechanical seems an improbable addition to the heat treater's vocabulary. It really isn't. Manufacturers of automobiles, electronics, optical and medical devices, as well as consumer goods are demanding faster and more accurate mechanical characterization for many different types of surfaces. Nanomechanical testing methods can be applied to understand properties like hardness and modulus of elasticity and to evaluate their effect on the performance and longevity of coatings and other surface morphologies. Characterizing surfaces down to the level of a few nanometers has always been difficult, but tremendous advances are being made.

Techniques in use now include depth-sensing indentation, which involves applying a specific force-time history on a rigid indenter while continuously monitoring the displacement of the indenter into the surface. Frequency-specific, depth-sensing indentation testing applies a small harmonic force on the indenter and measures the harmonic response of the indenter at the excitation frequency.

Trends in Software and Controls

The purpose of software is to turn data into useful information. Advances in instrumentation, particularly automation, have made vast strides in improving efficiency, making tests easier and quicker to perform. Instrumentation, in combination with a drive towards more rigorous calibration, has done much to improve the accuracy and repeatability of results. It is only because of automated instrumentation that tests such as thermal analysis as a function of temperature and frequency have become viable. Also, test needs change, and today's software is extremely accommodating.

In this era of new material introduction, advanced designs and quality assurance, it is widely suggested that practical testing will be increasingly replaced by computerized simulation – virtual testing.

Testing is now being enhanced by a powerful array of software solutions that promise to deliver faster, more dependable product development cycles, increased savings and better overall product designs. Matt Struve of MTS Systems Corp. (Eden Prairie, Minn.) talks about a new era of information powered by the "Science of Synchronicity;" that is, the pursuit of a more productive resonance between the physical test and virtual simulation worlds to realize the full potential of virtual product development. The Science of Synchronicity comprises four core principles (Fig. 2), each of which can be used to enhance the testing and development process.

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The Do's and Don'ts of Field Failure Analysis

In the real world, components fail (Fig. 1 - 2). There are many reasons why. If a premature failure occurs, it is important to evaluate all possible causes and then isolate a root cause that can be corrected to assure that the problem will not reoccur. Let's learn more.

Failures that occur during testing on the factory floor in controlled laboratory conditions or during product manufacture can be classified as "regular" failures, ones in which a significant amount of resources (people and test equipment) can be brought to bear on the problem. By contrast, field failures are those in which an assembly has failed in its normal working environment, often remote to the manufacturer, and where resources for analysis can be limited and must be transported to the jobsite. Field failures are particularly challenging. To help, here are some simple suggestions.

A Baker's Dozen

1. **Have a plan.** Failure analysis starts with documented procedures and good training. Anticipate your needs. Don't be surprised or confused with what has to be done. Do

Fig. 1. Power generation diesel engine failure (photograph courtesy of Materials Engineering Inc.)

Fig. 2. Piston head component from failed power generation engine *(photograph courtesy of Materials Engineering Inc.)*

not attempt to investigate a failure in a haphazard way.

2. **Set up a team** composed of representatives of all disciplines (management, purchasing, engineering, service, metallurgy, manufacturing, quality and legal). Meet to discuss the problem and review the facts (this step is often overlooked but can be invaluable if someone is able to recall details of this circumstances surrounding how the part was originally manufactured). Make recommendations as to the type and extent of the analysis that should be performed in the field. Understand the implications of the failure and the potential liability exposure of the company.
3. **Use your time efficiently.** Be prepared for the inspection. Plan your work carefully to obtain as much evidence as possible. Gather the right people and tools. Don't become distracted.
4. **Inspect failed components immediately.** Information relating to the failure should be compiled as soon and as thoroughly as possible. A "rapid response team" should be in place. Know that the longer the time between the failure and the inspection, the greater the risk that external influences will induce error into the analysis process.
5. **Preserve the evidence.** The ideal situation is to have information gathered on-site by a person or team familiar with failure analysis methods. If you can't get to the failure site, have someone at the site you can trust report the situation to you or follow your explicit instructions. Photograph everything (especially in this age of digital photography), even details that seem like incidentals. Try to emphasize the importance of having quality tools at the jobsite (especially the right digital camera—the ones we use can photography up to 1.2 in. away from an object with 8 megapixal resolution).
6. **Evaluate if field disassembly is a good idea.** In a perfect world, the assembly that failed would be undisturbed when the people assigned the task of field failure analysis arrive on the scene. Often, however, the assembly has already been taken apart; which, admittedly, may have been necessary to determine which component had failed. In some instances, the equipment has already been placed back into service. This usually makes the job of determining the root cause of failure much more difficult. Prior to arrival, if asked by field personnel for permission to disassemble and return equipment to service before a thorough

inspection by the failure analysis team takes place, resist the urge to say yes. It may be necessary to ship the assembly back to the factory in one piece for a more controlled analysis where more specialized resources may be available. If field disassembly is required, the failure analysis team should participate and not defer "hands on" work to technicians or less qualified individuals.

7. **Document each step of the disassembly** (whether in the field or in the laboratory). Do not hurry. Disassembly should stop while you inspect and document the condition of a component before proceeding to the next component.
8. **Do not form premature conclusions**, no matter how obvious the cause may seem. Concentrate on gathering facts (not speculation) along with all the evidence (and not just the evidence that may support a hastily drawn conclusion). Consider but don't be swayed by the opinion of others.
9. **Be patient.** Gather your thoughts and analyze the collected data. Don't let fatigue or conditions at the jobsite (wind, cold) become distractions or cause you to lose perspective. Be prepared for the environment you will find yourself in.
10. **Document what you see**, and (perhaps of equal importance) what you don't see. Remember that there is a reason for everything you are observing, and it may become important later on when you reconstruct the failure or when you consider the evidence as a whole. Taking photographs at each step of the disassembly is perhaps the best method of documentation, but should be supplemented with notes of your observations and measuring key dimensions.
11. **Control the investigation.** Make sure you come away with what you need. This includes such items as: service conditions (temperature, pressure, chemical or environmental exposure); time in service; circumstances surrounding the failure; cyclic conditions (if any); complete service history of both the failed component and assembly or machine which it is a part of (cleaning procedures, maintenance history); related or similar failures in the same or similar applications.
12. **Secure the evidence** for shipment back to the laboratory. Identify which components need to be analyzed with more powerful techniques than might be available in the field. Take more evidence than you think you might need. And be aware of the special handling of the failed components. Spare no expense in seeing to it

that the components will not suffer additional damage during transportation back to the laboratory for a more detailed analysis.

13. **Do not bow to unreasonable pressure.** Insist on privacy and don't become distracted. Do not report your findings until the investigation is complete and until you've had a chance to think through the consequences of your conclusions.

And remember, failures occur for a variety of reasons including:

- Overloading: tensile stresses, torsional forces and shear forces
- Impact: mechanical shock, thermal shock
- Wear: abrasive, adhesive, fretting, cavitation damage, erosion
- Fatigue: vibration
- Corrosion: uniform, pitting, galvanic, crevice, intergranular
- Stress corrosion cracking: hydrogen embrittlement, sulfide embrittlement
- Heat Treatment: improper hardening, tempering, quenching, cryogenic treatment

The Best Laid Plans…

Unfortunately, it's not unusual for a failed part or assembly to arrive at the lab accompanied by cryptic and/or incomplete information. If this occurs, or if adequate resources were not available to do a thorough field investigation, remember that this increases the uncertainty of your conclusion. Learn from the experience and educate others on the proper techniques for next time.

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The Embrittlement Phenomena in Hardened & Tempered Steel

While the end-use application of a component dictates its heat treatment, as heat treaters we know that we must achieve a delicate balance between the properties of strength and ductility. Nowhere is this fine line more evident than in the tempering process where precise control of time and temperature are critical to help produce a part with optimized microstructure and mechanical properties.

Essentially, tempering is the modification of the newly formed microstructure toward equilibrium Almost all steels that are subjected to any type of hardening process are tempered. A temper is a subcritical heat treatment that alters the microstructure and properties. In general, tempering lowers strength and hardness while improving ductility and toughness of the as-quenched martensite. However, this is not always the case. Let's learn more.

What is Temper Embrittlement?

In general, embrittlement is a reduction in the normal toughness of steel due to a microstructural change and chemical effects. Temper embrittlement is a phenomenon inherent in many steels, characterized by reduced impact toughness. It occurs in certain quenched and tempered steels and even in ductile irons with susceptible compositions. This form of embrittlement does not affect room-temperature tensile properties but causes significant reductions in impact toughness and fatigue performance. Although normally associated with tempered martensite, temper embrittlement can also occur if the matrix is tempered to the fully ferritic condition.

Types of Temper Embrittlement

When tempering steel, several types of embrittlement must be avoided. The first type, tempered martensite embrittlement (TME), is an irreversible phenomenon that occurs in the range of approximately 250–400°C (480–750°F) and is often referred to as "blue brittleness" or "350°C (500°F) embrittlement."

The second type, temper embrittlement (TE), is a reversible phenomenon occurring when steels are heated in or slow cooled through the temperature range of 375–575°C (705–1070°F).

Recently it has been reported that a transition from ductile to intergranular fracture in steel having greater than 0.5% C has been observed in martensitic

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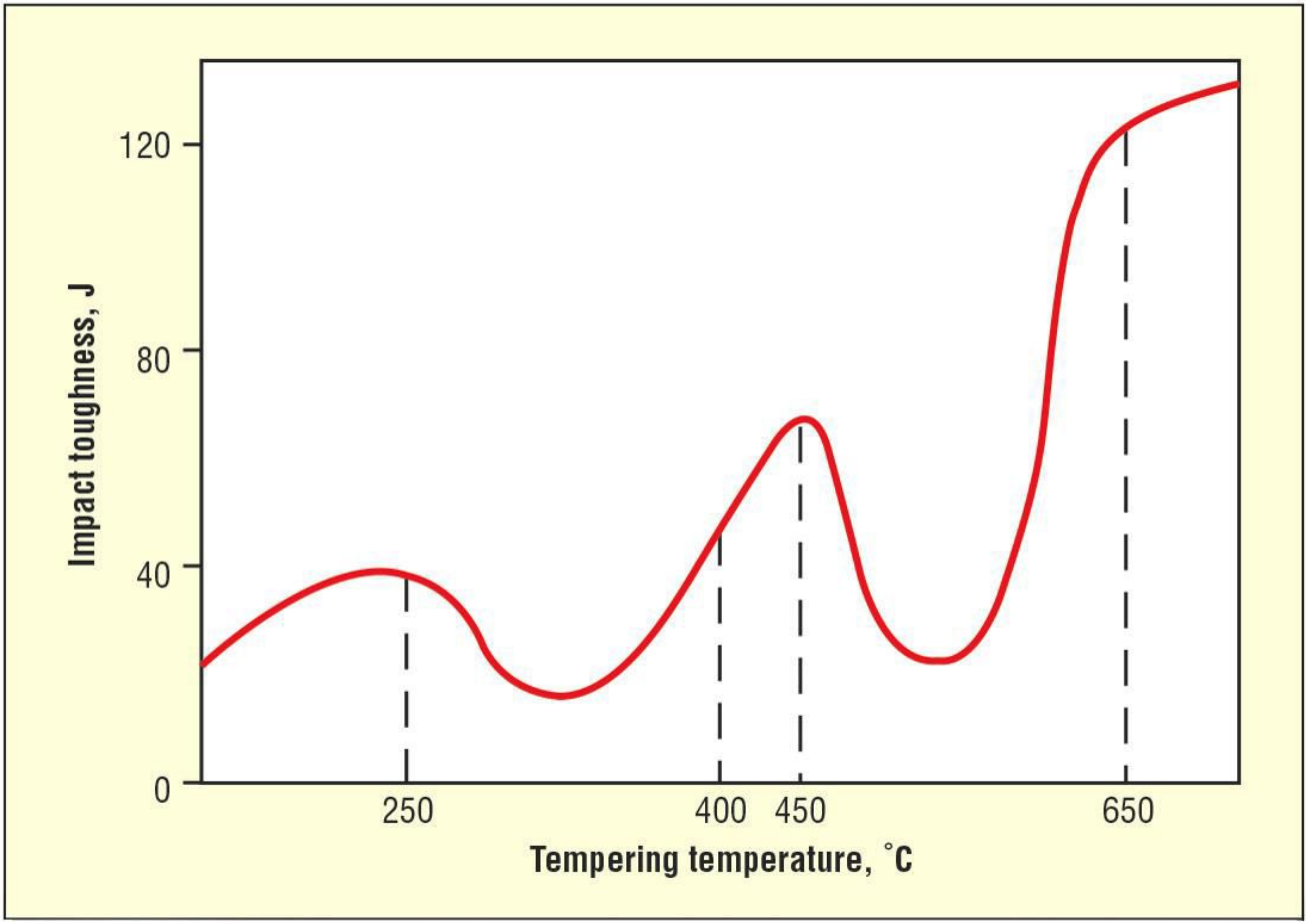

Fig. 1. Effect of temperature on impact toughness[5]

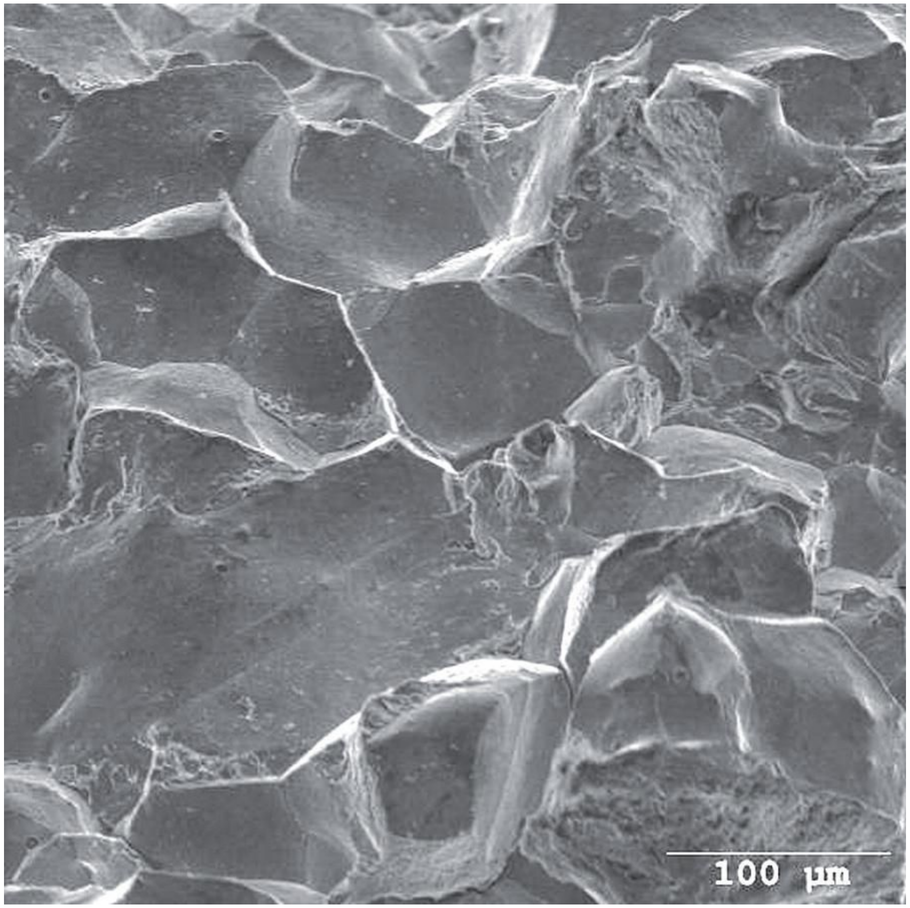

Fig. 2. SEM photo of intergranular temper embrittlement (Courtesy of Matco Associates Inc.)

steels tempered at low temperatures. Under tensile or bending stress, these higher-carbon steels are highly susceptible to intergranular fracture in both the as-quenched condition and after tempering at low temperatures generally considered to be safe from these embrittlement phenomena. In view of the fact that tempering is not required to render the microstructure susceptible to intergranular fracture, this type of embrittlement phenomenon is referred to as quench embrittlement. [2]

Why does it happen?

Tempered martensite embrittlement and temper embrittlement are examples of intergranular embrittlement. A common factor in such failures is the presence of elements that segregate to the grain boundaries. The chemical reaction rate or kinetics of segregation are such that they exhibit “C” curve behavior in the 350–550°C (660–1020°F) range. In other words, segregation does not occur uniformly. Both types of embrittlement are in part related to grain-boundary segregation of impurity elements (e.g. arsenic, antimony, phosphorus, and tin). Usually indicated by an upward shift in ductile-to-brittle transition temperature, both types of embrittlement develop during thermal processing after austenitizing and quenching to martensite.

Tempered martensite embrittlement is thought to result from the combined effects of cementite precipitation on prior austenite grain boundaries or interlath boundaries and the segregation of impurities at prior-austenite grain boundaries.

Temper embrittlement that occurs in the range of 375–575°C (705–1070°F) is believed to be due to segregation of impurity elements (P, Sn, As, Sb) to prior austenite grain boundaries. This causes decohesion of the boundaries, resulting in the tendency for low-energy intergranular fracture under certain loading conditions.

Which steels are affected?

All steels are susceptible, so the real question becomes how susceptible and what factors affect that susceptibility. For example, while plain-carbon steels may contain some of the impurity elements that will cause the embrittlement phenomenon to occur, the segregation of these elements is often enhanced by or caused by the presence of other alloying elements in substantial quantities. As a result, alloy steels generally have more susceptibility than carbon steels.

It is important to understand that the degree of embrittlement is affected by the prior austenite grain size and hardness. So, if we are dealing with a fine-grained plain-carbon steel of low hardness, it may not experience embrittlement symptoms despite its phosphorous content whereas a more highly alloyed Cr-Ni steel used at higher hardness is more susceptible to impurity content.

Widely used alloying elements such as chromium, nickel and manganese tend to promote temper embrittlement with the highest embrittlement effect observed in Cr-Ni and Cr-Mo steels. Small additions of molybdenum in Cr-Ni steels (0.2-0.3% in solution) can diminish temper embrittlement being caused by phosphorus. Temper embrittlement can be diminished by keeping silicon and phosphorus levels as low as possible, adding up to 0.15% molybdenum and avoiding the embrittlement heat-treating conditions.

Susceptibility also depends on impurity control, and here is where the steelmaking process is critical. For example, in plain carbon and Cr-Mo steels (those with no Ni) where phosphorous is the most important embrittlement element, the percentage can be controlled by the steelmaking process. In steels that contain significant amounts of nickel, antimony and tin are more potent embrittlement elements. Phosphorous has an effect, but not as large as it has in plain carbon and Cr-Mo steels. It should be noted, however, that antimony and tin in plain-carbon steels could cause other hotworking issues.

How can we correct it?

Tempered martensite embrittlement (TME) is irreversible and its effects are permanent. By contrast, the effects of temper embrittlement (TE) can be reversed. This is done by re-tempering above the critical temperature of 575°C (1070°F), then cooling rapidly, or by re-austenitizing and cooling rapidly. Impact toughness can be restored. If necessary, this process can be repeated.

A Simple Example

Alloy steel, which is susceptible to temper embrittlement, will exhibit a relationship in which one finds a lower temperature energy trough (Fig. 1) around 250 - 400ºC (480 - 750ºF), which is indicative of tempered martensite embrittlement (TME). At higher temperatures, 450 - 650ºC (840 - 1200ºF) the trough represents temper embrittlement (TE). The fracture surfaces (Fig. 2) show a "rock candy" type appearance.

Summary

The susceptibility of a given steel to temper embrittlement depends on a number of factors including grain size, hardness, steel grade and the impurity control in the steelmaking process itself. Not all steels and not all steelmaking processes are equal.

Finally, as heat treaters we must avoid the temptation to temper to a given hardness value without understanding the consequences of our actions. Since we do not have a simple embrittlement test that can be used on the shop floor, we must understand the phenomenon and question specifications that put us into temper embrittlement ranges.

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A Heat Treaters Guide to Hydrogen Embrittlement

Proper surface modification and heat treatment techniques offer many part performance benefits, but hydrogen absorption can cause unexpected problems. Hydrogen attack in the form of embrittlement is responsible for a surprising number of delayed failures and problems with heat treated parts, especially if they undergo secondary operations such as plating. Heat treaters need to better understand hydrogen embrittlement. Because it can affect both ferrous and nonferrous materials, hydrogen-bearing atmospheres for heat treating and brazing should be of particular interest. Let's learn more.

How Hydrogen Gets In

Hydrogen in atomic form can enter and diffuse through a metal surface whether at elevated temperatures (Table 1) or ambient temperature. Once absorbed, dissolved hydrogen may be present as either as atomic or molecular hydrogen or in combined molecular form (e.g., methane, or CH_4). Since these molecules are too large to diffuse through the metal, pressure builds at crystallographic defects (dislocations and vacancies) or discontinuities (voids, inclusion/matrix interfaces) causing minute cracks to form. Whether this absorbed

Table 1 Carbon and hydrogen diffusion rates [1]

	Micro-structural phase	Tempera-ture, ºF (ºC)	Diffusion rate, (cm^2/s)
Carbon	Austenite	1700 (925)	4.0×10^{-7}
Hydrogen	Austenite	1700 (925)	1.7×10^{-4}

Fig 1. Exfoliated steel sliver

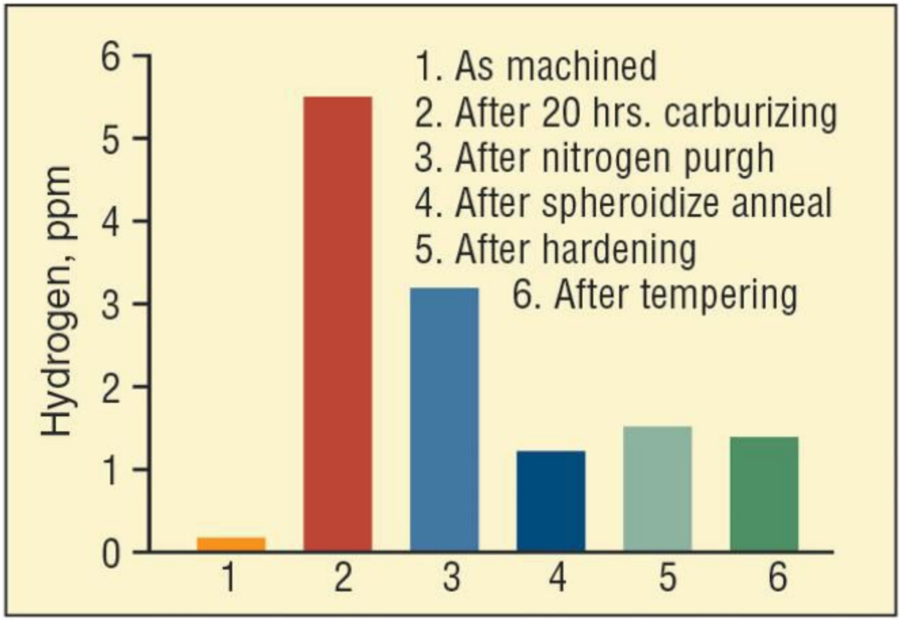

Fig 2. Hydrogen content after various stages of processing

hydrogen causes cracking or not is a complex interaction of material strength, external stresses and temperature.

Sources of hydrogen include heat-treating atmospheres, breakdown of organic lubricants, the steelmaking process (e.g., electric arc melting of damp scrap), the working environment, arc welding (damp electrodes), dissociation of high-pressure hydrogen gas and grinding in a wet environment. Parts undergoing electrochemical surface treatments such as etching, pickling, phosphate coating, corrosion removal, paint stripping and electroplating are especially susceptible.

For example, large deep carburized components can suffer corner exfoliation of large slivers of the case (Fig. 1), which are characterized by a central initiation coincident with the case core junction[1]. The initiation of the fracture is near the center of the fracture surface (burr etched square) corresponding to the case core junction. SEM reveals a region of intergranular fracture at the point of initiation. There was skepticism whether hydrogen absorbed during the carburizing process (not present originally in the steel) could be the culprit. An accurate analysis of the sources of hydrogen (Fig. 2) was performed to prove it. A nitrogen purge at the end of the carburizing cycle did not completely remove all the hydrogen. A significant proportion of the hydrogen was removed during a subsequent spheroidize anneal at 1150°F (620°C). However a nitrogen purge at the end of the carburizing cycle resolved the problem by lowering the hydrogen level at a critical period in the cycle.

Nature and Effect of Hydrogen Attack

Although the precise mechanism(s) of hydrogen embrittlement is actively investigated, the reality is that components fail due to this phenomenon (Table 2). It is believed that all steels above 30 HRC hardness, as well as copper (tough pitch or oxygen free) and titanium and nickel alloys are vulnerable.

A metallurgical interaction occurs between atomic hydrogen and the atomic structure inhibiting the ability of the material to deform or stretch under load, making the material "brittle" under stress or load resulting in fracture at a much lower load or stress than anticipated. This lower breaking strength makes hydrogen embrittlement so detrimental.

In general, a steel's susceptibility to hydrogen embrittlement increases with increasing steel strength. High strength steels with some degree of temper embrittlement are very susceptible to hydrogen embrittlement as are quenched and tempered and precipitation hardened steels

Nonferrous materials, such as tough-pitch coppers and even oxygen-free coppers are subject to a loss of (tensile) ductility when exposed to reducing atmospheres. Bright annealing in hydrogen bearing furnace atmospheres or torch/furnace brazing are typical processes that can induce embrittlement of these materials. Atomic hydrogen diffuses into the copper subsequently reducing cuprous oxide (Cu_2O) to produce water vapor and pure copper. Embrittled copper often can be identified by a characteristic surface blistering resulting from expansion of water vapor in voids near the surface. Purchasing oxygen-free copper is no guarantee against the occurrence of hydrogen embrittlement, but the degree of embrittlement depends on the amount

Table 2 Examples of hydrogen damage and ways to avoid it

Symptom	Solution
Internal cracking or blistering	Use steel with low levels of impurities (S and P) Modify the environment to reduce hydrogen charging Use surface coatings and effective inhibitors
Loss of ductility	Use lower strength (hardness) or high resistance alloys Select proper materials of construction and plating systems Heat treat (bake out) to remove absorbed hydrogen
High-temperature hydrogen attack	Select proper material (e.g., low and high alloy Cr-Mo steels) Limit temperature and H_2 partial pressure

Table 3 Hydrogen bake out requirements for high strength steels [3]

Tensile strength, ksi (MPa)	Hardness, HRC	Time, h (a)
247-261 (1,700-1,800)	49-51	22+
232-247 (1,600-1,700)	47-49	20+
218-232 (1,500-1,600)	45-47	18+
203-218 (1,400-1,500)	43-45	16+
189-203 (1,300-1,400)	39-43	14+
174-189 (1,200-1,300)	36-39	12+
160-174 (1,100-1,200)	33-36	10+
145-160 (1,000-1,100)	31-33	8+

Note: Per ASTM B 850-98 (2004). (a) Post plate bake out at 375-430°F (190-220°C) of oxygen present. For example, CDA 101 (oxygen free electronic) allows up to 5 ppm oxygen while CDA 102 (OFHC) permits up to 10 ppm. A simple bend test is often used to detect the presence of hydrogen embrittlement. Metallographic examination for the presence of voids at grain boundaries and of the near surface also is used.

How Hydrogen Gets Out

Hydrogen absorption need not be a permanent condition. If cracking does not occur and the environmental conditions are changed so no hydrogen is generated on the metal's surface, hydrogen can diffuse back out of the steel, and ductility is restored. Performing an embrittlement relief, or hydrogen bake out cycle (the term "bake-out" involves both diffusion within the metal and outgassing) is a powerful method to eliminate hydrogen before damage can occur. Key variables are temperature, time at temperature, and concentration gradient (atom movement).

For example, electroplating provides a source of hydrogen during cleaning and pickling cycles, but the most significant source is cathodic inefficiency. A simple hydrogen bake out cycle can reduce the risk of hydrogen damage (Table 3). Caution: over-tempering or softening of the steel can occur especially on carburized or induction hardened parts.

Influencing Factors for Hydrogen Embrittlement

The severity and mode of the hydrogen damage depends on:

- Source of hydrogen: external (gaseous) or internal (dissolved)
- Exposure time
- Temperature and pressure
- Presence of solutions/solvents (esp. acidic) that could react with metals
- Alloy type and production method
- Amount of discontinuities in the metal
- Treatment of exposed surfaces (barrier layers, e.g., oxide layers as hydrogen permeation barriers on metals)
- Final surface treatment (e.g., galvanic nickel plating)
- Heat treatment method
- Level of residual and applied stresses

Low Hydrogen Concentrations Too

Of concern today is embrittlement from very small quantities of hydrogen where traditional loss-of-ductility bend tests cannot detect the condition. This atomic level embrittlement manifests itself at levels as low as 10-ppm hydrogen and there are many documented cases of embrittlement failures with hydrogen levels this low (1 ppm hydrogen reportedly has been a problem in certain plating applications). This type of embrittlement occurs when hydrogen is concentrated or absorbed in certain areas of metallurgical instability. Both residual and applied stress causes this concentrating action, which tends to “sweep” through the atomic structure, moving the infiltrated hydrogen atoms along with it. These concentrated areas of atomic hydrogen can coalesce into molecular type hydrogen, resulting in the formation of highly localized partial pressures of the actual gas.

Where to Go For Help

A good but relatively unknown source of information for heat treaters about the effects of hydrogen is NACE International, The Corrosion Society (www.nace.org).

Summary

Although many of the most severe problems associated with hydrogen embrittlement have occurred with aircraft/aerospace parts, a simple motto to remember is that the part doesn’t have to “fly to die.” The insidious nature of hydrogen embrittlement continues to cause product failures during both processing and service; failures that often are catastrophic leading to injury or damage to adjacent structures. Hydrogen damage can and must be avoided.

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How to Conduct a Heat Treat Audit

Audits of the heat-treating department are a vital part of any good quality program - either as part of a self-assessment or ISO program for a captive shop or, of equal importance, as part of an evaluation of the capabilities of a commercial heat-treat supplier. In either case, the audit process needs to be formal in nature and follow specific guidelines. Let's learn more.

The audit process should be designed to ask basic questions: Who is performing the heat treatment and are they competent; what procedures are being used to carry out the heat-treating operation, and are they adequate to assure proper quality; where is the work being done, and is the shop performing the work capable of performing the required task(s); when was the last assessment, and was it representative of current practice; why is an assessment required now - in other words, did a quality issue or problem trigger the event, and if so, will the audit questions help resolve it; and finally, how will the audit be performed - that is, is the right personnel in place to reach meaningful conclusions?

What Constitutes a Good Audit?

Most audits that "fail" do so because they do not reveal the true nature of what is happening within the heat-treatment operation. Care must be taken to look at both the quality aspects (forms, instructions, compliance) and the performance aspects (process control, work handling, etc.). Too often audits focus their attention on the former and give a cursory look at the latter. This disconnect is the reason many organizations are confused as to why their departments or suppliers fail to achieve continuous improvement.

To be useful, heat-treat audits need to ask tough and realistic questions, not just be forms in which the auditor fills in the blanks. In other words, the true story is revealed only in the detail. It is critical that audits "drill down" to the level that the work is being done – a good heat-treat audit spends less time in the office than on the shop floor. Finally, auditors must reward well-run operations and not hesitate to give them top scores when it is deserved. Here's a look at some of the critical information necessary.

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Fig. 1. Typical commercial heat treat operation for auditing that serves the automotive and aerospace Industries (courtesy of ALD Thermal Treatment)

1. **General** (company/department profile)
    a. Date, supplier's name and plant location.
    b. Key contact information including corporate contacts (if appropriate), plant manager, quality manager, metallurgist, and 1st, 2nd and 3rd shift supervisors.
    c. Financial viability.

2. **Capabilities** (general requirements)
    a. List all part numbers, cross-indexing them to their corresponding engineering drawings, specifications (including all testing requirements) and special needs (e.g. distortion concerns, handling concerns, dimensional tolerances, etc.).
    b. List the types of materials that the heat treater is qualified to run.
    c. List the heat-treat processes capable of being run. Be sure to tie each heat-treat process with the specific equipment involved by part number.
    d. List each heat-treat cycle including the type of quench. Be sure to identify all process and equipment variables involved.

3. **Instructions** (for auditors)
    a. Clearly define what will be required in a heat-treat audit and communicate this information to the intended parties well in advance of the physical audit so that the necessary information can be gathered ahead of time.
    b. Create a consistent and fair rating guideline (see below) and adhere to the categories and questions selected.
    c. Be sure that both parties agree to corrective actions and completion dates and responsibilities are clearly delineated.
    d. Follow up personally within the specified time frame.

4. **Sample Rating Guidelines** (for audit questions)
    a. If a required activity is not being performed (rating = 0).
    b. If there is only rudimentary activity (rating = 1) or if the activity is being performed and documented but has minor deficiencies (rating = 2).
    c. If the activity is inadequate for the task required (rating = 3) or if the activity is properly documented but not properly performed (rating = 4).
    d. If the activity is being adequately performed and is documented (rating = 5) and if, in addition, includes evidence that the activity achieves the task(s) required (rating = 6).
    e. If the activity is well documented and is adequately performed (rating = 7) and if, in addition, continuous improvement is evident (rating = 8).
    f. If the activity is well documented and beyond expectations (rating = 9) and if continuous improvement is overwhelming (rating = 10).

5. **Continuous Improvement Program** (areas to review)
    a. Good-Better-Best practices related to heat treatment and testing.
    b. Process parameter variability is being controlled.
    c. Equipment variability is being controlled.
    d. Laboratory best practices are being used.
    e. Scrap-Reject-Rework plans and procedures being used.

f. Documented planned preventive maintenance.

6. **Audit Questions** (typical)
   a. Are heat treat part handling, processing and storage adequate to preserve product integrity and quality?
   b. Are adequate controls employed to ensure that the processing and inspection status of the product are known throughout the heat-treating operation, and are process/product monitoring and controls functions (and responsibilities) clearly defined?
   c. Is both the responsibility for and practice of heat-treat process (recipe) development, testing methods and quality planning clearly defined?
   d. Does the heat treater have available and use a procedure for reviewing part design and specifications in relation to method of loading as well as heat-treat process parameter and equipment selection?
   e. Are process verification and/or capability studies conducted on all new part numbers?
   f. Are control plans and process (FMEA's) used as a basis for establishing quality programs for heat-treat processes?
   g. What procedures are in place, and how does the heat treater react to customer concerns (internal or external indicators)?
   h. Are controls in place and being used on the shop floor to effectively monitor the process?
   i. If necessary, are statistical process control (SPC) methods utilized for key product parameters?
   j. Are written procedures/work instructions defining heat treat and quality functions available and in use on the shop floor (i.e., is the quality manual a living document)?
   k. Are adequate in-process monitoring and inspections/tests performed, and are there adequate records?
   l. If on-site, does the testing or metallurgical laboratory have the tools, procedures and expertise to accurately determine part quality? If off-site, is the testing laboratory

properly accredited?

- m. If part testing and/or PPAPs are performed, are records available with supporting documentation for the relevant heat-treated products?
- n. Are documented and verifiable heat-treating reject, reprocessing and/or scrap records available?
- o. Is there an effective preventive maintenance program in place for both the heat-treating and process-monitoring equipment?
- p. Does the heat treater have an effective system for ensuring the quality from his suppliers and service providers (instrumentation calibrations, quench oil checks, etc.)?
- q. Is plant cleanliness, housekeeping, environmental and working conditions conducive to a safe, efficient operation in which continuous improvement can take place?

7. **Non-Conformance** (document, in detail)
   - a. Major and minor non-conformances.
   - b. Pertinent general and specific observations.

8. **Corrective Action**
   (for each supplier location)
   a. Issue statement.
   b. Corrective action(s) required.
   c. Responsibility.
   d. Implementation Date.
   e. Root cause found.
   f. Follow-up plan (actions and dates).

A Look at CQI-9

An Automotive Industry Action Group (AIAG) heat treat audit guideline entitled "CQI-9, Special Process: Heat Treat System Assessment" was released in March 2006 and has been revised several times in the past few years. It is intended to help standardize the heat treat audit process. The HTSA supports the automotive process approach as described in ISO/TS 16949.

Within each audit area, the major sections covered are: (1) process and test equipment requirements; (2) pyrometry; (3) process monitoring frequencies; (4) in-process/final test frequencies; and (5) quenchant and solution test frequencies.

Summary

Heat-treat audits are so important that, in this writer's opinion, they need to be conducted by trained and certified heat-treat personnel, not just auditors skilled in the procedures involved. Both captive and commercial heat-treatment organizations should demand that this aspect of their business be given the attention and respect it deserves. A standardized audit guideline with fixed frequency of compliance is long overdue. With ever-increasing product performance from our customers, only continuous improvement will assure the heat treatment industry of continued growth and prosperity.

The Role of the Heat Treater in Reverse Engineering

Reverse engineering was the undercurrent of the 2003 movie thriller Paycheck starring Ben Affleck as a brilliant, yet brain-wiped engineer who had to solve the mystery of what he had spent the last few years of his life doing so right. One of the challenges that all engineers in this field face is how the components they are reverse engineering were heat treated. It's time to understand more about this technique and how our industry can contribute to its success. Let's learn more.

Reverse engineering is a true journey of discovery to uncover the fundamental principles of a component through analysis of its functionality, properties and microstructure. Such analysis can then be used to redesign the component usually in a reduced time frame, using such tools as computer-aided design modeling and rapid prototyping. This process typically involves a small number of highly specialized components. Speed and flexibility over traditional design and manufacturing methods are the hallmarks of reverse engineering.

Why Reverse Engineering?

Corporations often benchmark their products against those of competition by systematic disassembly and evaluation of the function of each component. Reverse engineering is also commonly used in the aerospace industry to produce replacement parts when the original source no longer exists, when the product has become obsolete or when the original product design documentation has been lost (or never existed). Reverse engineering can also correct previous mistakes or make a good feature into a great one. The goal of reverse engineering is to save cost by reducing analysis time and testing. Perhaps the biggest challenge faced by this process is the determination and duplication of the heat-treated microstructure.

The Process

The steps involved in this process include:

- Form-fit-function analysis
- Application review
- Material identification
- Mechanical-property determination
- Heat-treatment determination
- Confirmation testing

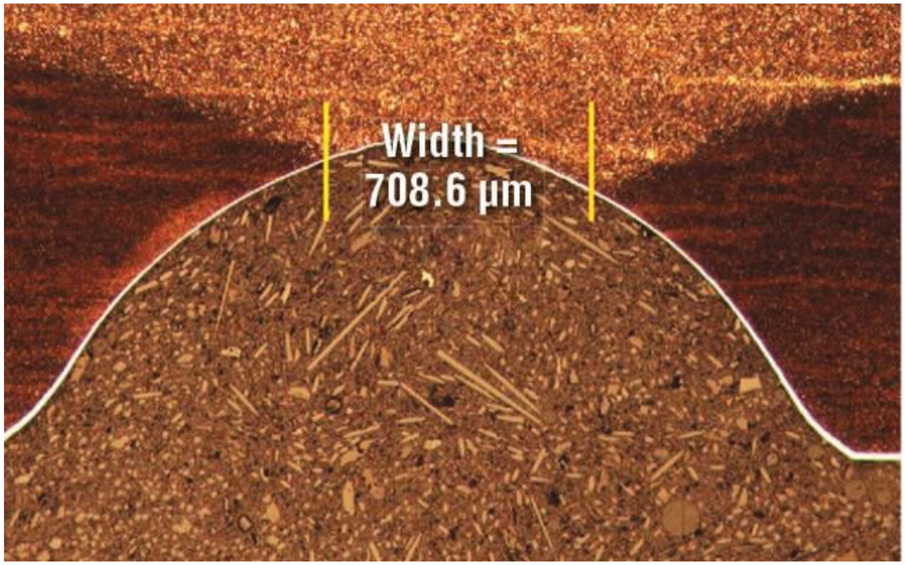

Fig. 1. 4150 ball screw

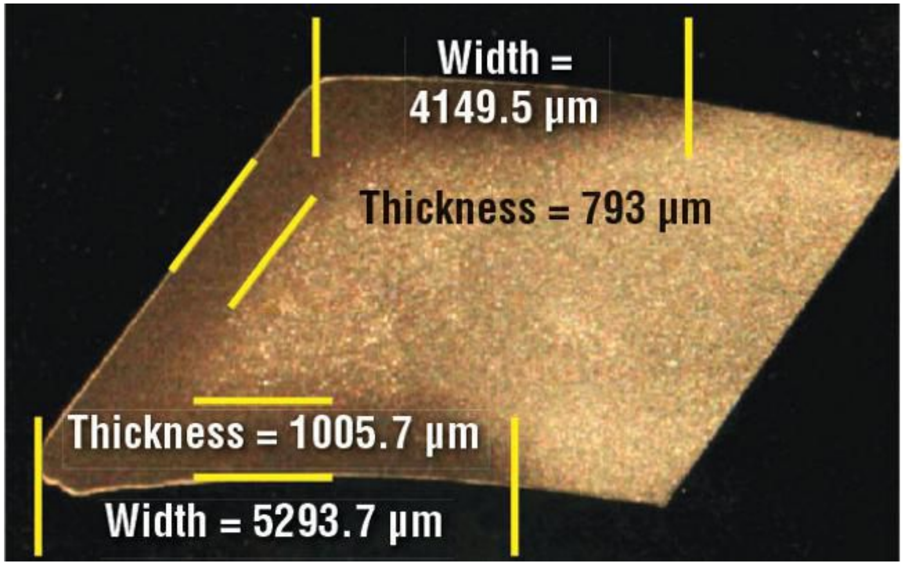

Fig. 3. 8620 deflector

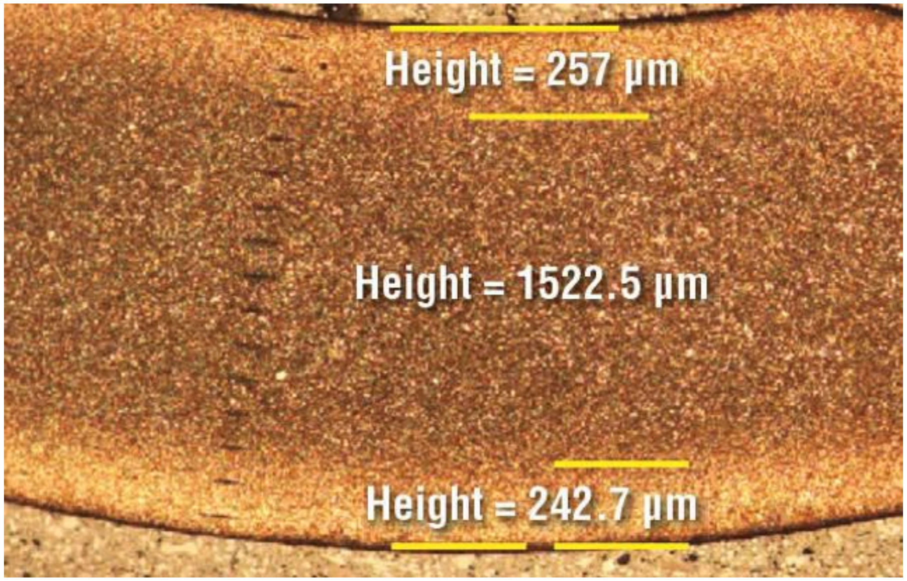

Fig. 2. 8620 pin

Fig. 4. 8620 ball screw

Typical steps in the process involve:

1. Examine the product. Understand (predict) how the product should work or how the product does work before disassembly.
2. Gather and analyze (interpret) all available information including customer requirements. With government projects, there may be overhaul manuals that define some or all of the critical dimensions and tolerances.
3. Acquire parts to use for dimensional data (e.g., size, shape, fits, interface considerations, etc.) and evaluate types of conditions to which the reverse-engineered component will be subjected. For wear issues, concerns should be part loading, seal surface condition, fluid erosion, etc.
4. Inspect and record all dimensions of the assembly.
5. Document the assembly with digital photographs.
6. Create a plan. Disassemble and document each part. Pay special attention to areas of wear, coatings, etc.
7. Develop a detailed understanding of the actual product function (structure) based on the disassembly process.
8. Establish primary engineering data.
9. Inspect each part visually and optically (via stereomicroscope). If appropriate, use other NDT methods.
10. Create a materials/components list (bill of materials).
11. Apply engineering analysis, simulations and build models (prototypes). Create preliminary drawings of each part and the assembly.
12. Establish tolerances from statistical analysis of measurements (if there are sufficient samples to inspect) or preferred standards.
13. Manufacturability review of drawing.
14. Perform chemical analysis to determine type(s) of material(s) in use.
15. Perform metallurgical, mechanical and physical testing to establish mechanical properties, thermal treatment and coatings. Results should include surface treatments and hardness, case depth (if applicable) and core hardness.
16. Deduce heat-treatment processes including pretreatments, localized effects, heat treating and mechanical working.
17. Deduce other manufacturing processes that will impact properties or end-use performance.
18. Examine metallurgical structure for any grain size, shape and grain flow.
19. Revise drawing with material chemistry, heat-treat recipes and specifications.
20. Produce finished components and perform functionality and life tests.

The engineering drawings created will provide dimensions, tolerances, finishes and other critical requirements. Function, however, is not normally specified on drawings or even referenced in specifications, so it must be determined by performance testing that simulates the mechanical, physical, metallurgical and environmental requirements of the end-use application. This requires an understanding of the materials, processes and heat treatments as well as gathering historical information, supplier records and material callouts.

General notes on drawings may help if they specify manufacturing operations. For example, drawings may call out method of cleaning, type of heat treatment, type of material, surface treatment (e.g., coatings, shot peening) surface finish, ancillary processes (normalizing, annealing, stress relief), inspection (penetrant or magnetic particle, etc.), process sequence (heat treat after welding, inspection after heat treatment), tooling (fixtures, etc.) and quality control tests (magnetic particle inspection, etc.) Preferred suppliers may also be called out indicating where to obtain specialized components and difficult-to-procure materials or where to go for specialized processing (casting, brazing, plating, straightening).

In the world of reverse engineering there is a simple cost equation:

$$\text{Cost} = \text{Material} + \text{Manufacturing} + \text{Inspection} + \text{Finishing} + \text{Rework} \quad (1)$$

The Approach

Using a sample component known to be of good quality is a luxury seldom afforded the reverse engineer. Many times the component delivered to us is worn, damaged or abused in some fashion. This makes the job much more difficult. Part dimensions determined by direct measurement must be altered to take into account these factors. Material must be determined by chemical analysis, but oftentimes subtle issues such as trace-element chemistry, grain size and prior microstructure must be factored into the analysis. Selection of the proper mechanical-test methods is a critical component of the process. One must also realize that results may vary, and validation is an important aspect of the final determination.

Mechanical testing helps to determine and/or confirm the heat-treatment methods used. Drawings, if available, typically call out hardness and perhaps key strength or ductility values as a range or a minimum. Indirect mechanical-test methods (preferred due to their cost) include hardness and conductivity testing,

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while direct methods include tensile testing, impact and torsion testing and corrosion tests.

Hardness alone cannot be relied upon to provide the complete picture to determine heat-treatment details. For example, SAE 4340 bar can be specified in accordance to MIL-S-5000 (air melted) or MIL-S-8844 (vacuum melted). Either form produces identical tensile properties and hardness. However, MIL-S-8844 has superior toughness and low temperature properties. Similarly, Inconel® 718 sheet can be purchased per AMS 5596 or AMS 5597. While both have nearly identical tensile properties, they require different heat treatments and demonstrate different creep properties.

Heat-Treatment Precautions

Heat treating requires both a process and a piece of equipment in order to be successful. Controlling both process and equipment variability is the key to ensure that the design engineer gets the product response he is expecting. One of the dangers encountered when reverse engineering a product is to not take into account the service history. Many projects fail due to a correct heat treatment to an incorrect specification. Finished effective case depth, for example, is not the same as effective case depth. Also, international standards, global material sourcing and advanced heat-treatment methods and equipment in use today may not have been in place when the component was originally manufactured.

Example 1: Ball Nut (Fig. 1)

This B1 Lancer slat-actuator ball screw is made from SAE 4150 material. Examination of this item revealed that it was induction hardened after preliminary groove cutting.

The scalloped pattern of the induction-hardened area resulted from the material removal that increased the distance to the coil.

Example 2: Floating Pin (Fig. 2)

This P3 Aileron control chain is a carburized component. The pin was carburized to provide a wear surface. After installation, the ends of the pin were swaged to retain them in the assembly. Subsequent analysis determined that the ends of the pin were induction annealed to allow the swaging without breaking or cracking of the edges.

Example 3: Deflector (Fig. 3)

The C5 Galaxy flap jack screw has carbon-enriched “tips” followed by an anneal from carburizing temperature. It is then induction hardened followed by edge tempering. The core was to remain very soft so that it conforms to a helical ball-screw groove in the nut and does not damage the groove in the screw.

Example 4: Ball Screw (Fig. 4)
From the C5 Galaxy, this slat-actuator ball screw is an example of an unexpected process – namely carburizing. The reverse-engineering assumption was induction hardening. However, the chemistry and metallurgy supports carburizing per AMS 2759/7. Examination of this item also revealed that it was partially machined before carburizing.

Confirmation Testing

A key question to ask yourself is, "Are you absolutely sure that you perform relevant functional tests?" If not, the part will fail to perform its intended function. Material and process characterization may be incomplete, perhaps melting practices have been overlooked or further inspections are necessary. Information about auxiliary processes, specifications, tolerances and manufacturing sequences may need to be revisited.

Summary

Always remember, making an identical part is either extremely difficult or impossible, so extreme care must be taken to ensure that the reverse-engineered product performs to expectation (and beyond).

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Setting Up a Planned Preventive Maintenance Program

"Pay me now, or pay me later" is a famous line from a TV commercial that aired several years ago. It featured an actor standing atop a smoldering car engine holding a FRAM® oil filter in one hand while pointing down at the destroyed engine with the other. His sad, thoughtful expression conveyed volumes. Sound familiar? This very phrase epitomizes equipment maintenance in the heat treat department. One of the reasons why we often pay later is a failure to present to management a comprehensive plan (not a list) for maintenance activities. Setting up a planned preventive maintenance program is the cornerstone of a successful heat treat operation. Let's learn more.

Accepting the Inevitable

Maintenance is a fact of life for industrial machinery, and is far more complex for heat treat equipment. In general, the cost of maintenance increases dramatically with increased operating temperature and/or the process severity (e.g., carburizing versus hardening). As with all equipment, some styles and designs require more attention than others. Construction of heat treat equipment can often be classified as heavy duty or light duty, and if operated outside its design limits, almost always translates to a need for more extensive maintenance.

A great deal of money can be spent (and wasted) if careful thought and clear understanding of the equipment design, as well as the extent of the repair, is not taken into account. Failure to determine the root cause of why a component failed can have disastrous bottom-line consequences.

Proper maintenance maximizes up-time productivity, and the use of planned preventive maintenance programs results not only in better equipment reliability, but also in improved process repeatability and control — essential to producing good parts with consistent metallurgical and mechanical properties.

Once management under-stands, accepts and budgets for maintenance expenditures, the operation of all heat treating equipment, especially high temperature furnaces and induction units, becomes far more reliable.

Recordkeeping

Good record-keeping is an important element of a planned preventive maintenance program. An operational, as well as a maintenance log, should be

Maintenance item (Location)	Type of maintenance (Action item)	Frequency (Operating hours)
☐ Oil circulating system	Functionality check (agitator rpm values)	24
☐ Elevator	Inspection (motion/timing)	24
☐ Quench oil level	Inspection	24
☐ Quench oil monitor	Test	24
☐ Load transfer drive(s)	Functionality (motion/speed)	24
☐ Quench oil filter	Condition	100
☐ Quench oil pump	Functionality	100
☐ Oiler and filter	Inspection	100
☐ Outer door	Alignment/fit	500
☐ Load transfer drive	Alignment/measurement	500
☐ Chain guide	Inspection (wear)	500
☐ Oil circulating system	Circulation pattern check	500
☐ Proximity switches	Adjustment	500
☐ Limit switches	Adjustment	500
☐ Quench oil	Property checks	1,500
☐ Elevator baffles	Inspection	3,000
☐ Oil circulating system	Velocity check (ft/sec through load)	3,000
☐ Load transfer drive	Stroke adjustment	3,000
☐ Oil circulating baffles	Inspection	3,000
☐ Agitator motor	Inspection	3,000
☐ Outer door	Seal replacement	6,000
☐ Oil agitator propeller	Inspection	6,000
☐ Limit switches	Replacement	9,000
☐ Chain guide	Replacement	12,000
☐ Load transfer drive	Replacement	18,000
☐ Agitator motor	Replacement	30,000

Fig. 1. Partial list of planned maintenance activities for an oil quench tank

kept, noting every action taken. A key aspect of such logs is that they should be reviewed periodically to look for trends, identify problem areas and gain a better understanding of the types of maintenance (and personnel skills) that are required.

Having the correct spare parts, detailed construction drawings and the right tools on hand minimizes downtime. Support by the plant engineering staff, the original equipment manufacturer (OEM) or a third party is often necessary too. Remember, successful planned maintenance is a team effort.

What happens if you buy a used furnace or inherit a piece of equipment from another location? First, gather as many facts as possible about its history, such as when and for whom it was built and what was the original intended application. Request spare-parts records from the OEM to identify type and frequency of parts sales—a good first indictor of places to look for problems. Obtain a copy of the instruction manual and obtain as many detailed prints as possible (there is often a fee for such items).

Next, completely inspect the unit inside and out. Look for the obvious, and ask yourself the reason for everything you see (and don't see). Last, don't rush the equipment into service. Measure and record such items as transfer times, speed of moving components such as doors or load transfer mechanisms and elevators. Once operational, record gas use, current draws and incoming voltage and power, and install hour meters, energy monitors and other reporting devices. In other words, learn as much about the normal operation of the equipment as possible before putting it in service, so you can monitor changes and quantify degradation that occurs over time.

The Plan

Divide and conquer. Begin by understanding the heat treat process(es) you will perform in the unit and compare these to the design ratings/limitations of the equipment. These items include:

- Temperature rating
    - Normal and maximum
    - Cyclic operating conditions
    - Idling conditions
- Loading
    - Volume or weight limitations
    - Part distribution
    - Maximum and minimum gross load weight as a function of temperature
- Atmosphere-process and heating
    - Type and function
    - Gas flowrate, pressures, etc.
- Special requirements

  - Baskets and fixturing
  - Quench restrictions
  - Access and site ports
- Design-specific features
  - Type of furnace (batch, continuous, pit, rotary retort, etc.)
  - Support items (heat exchangers, water circulating systems, etc.)

Understand the external constraints being placed on the equipment (use, budget, etc.). These factors are important in tailoring your plan to meet expectations.

Take the time to divide the equipment into logical sections so the maintenance on each of these areas focuses on those components or assemblies that are critical to their functionality (and ultimately that of the entire machine). Then inspect the exterior and interior. (Note: confined-entry training/permits may be required.) Observe how all components interact. This takes a surprisingly short time and yields a significant amount of information.

Put this information into a useable form (such as a spreadsheet), review with management and implement your planned preventive maintenance program. Remember that feedback and refinements to the plan will occur constantly. Make sure that the reasons for the changes are documented for later use, and make the system independent of changes that will inevitably occur in either the maintenance department, the heat treat department, or in management.

To realize the benefits of a planned preventive maintenance program irrespective of the type of equipment being used, a carefully structured, well-disciplined, rigorous plan must be created, implemented and followed.

Example

Figure 1 shows the planned maintenance program for an oil quench tank section of a continuous furnace with integral oil quench. The frequency of activities is based on operating hours. After identifying all key components, the list can be sorted by any category. For example, “maintenance item” or “frequency” sorts tell us what must be done and when it must be accomplished. A master list of all equipment sections can then be compiled.

A good maintenance program must include all of the necessary performance checks to be able to determine that the system remains operating at peak efficiency. The plan also reveals when an investigation should take place to explain a drop in performance values. Corrective action should follow.

The availability of computers in the office and on the shop floor allows administering preventive maintenance programs real time with benefits including cost containment and the ability to stay current with changes as they occur. Today, most heat treat furnaces can be supplied with an integrated planned preventive maintenance program, based on empirical data collected by the OEM's on mean time between failure (MTBF) of components and other historical/statistical information.

A simple, highly effective system I suggest is installing stack lights (red, yellow, green) atop the control panel. They can be integrated with the equipment controls to alert the operator when the equipment is operating within preset limits (green GO light); when a maintenance item (e.g., 10,000 cycles on a door cylinder) has reached a predetermined limit and needs attention, but is not so critical to the operation to force it to shut down (yellow CAUTION light); and when a failure has occurred (red STOP light).

Summary

What really matters is making the heat treating operation a transparent part of the manufacturing flow. The way to achieve this is by keeping equipment operating at peak efficiency and with as few "surprises" as possible. Planning for success is worth the time and effort. You deserve it, your heat treat equipment deserves it, and your wallet deserves it.

The Flame Curtain – Function, Adjustment and More

The Doctor has been making house calls and has found a disturbing trend – far too many heat treaters do not understand the function, design, adjustment or safe operating requirements for their flame curtains. It's time to review a subject too often taken for granted. Let's learn more.

Function

Flame curtains across furnace doors (Fig. 1) have multiple functions:

- To produce a vertical stream of combustion products across the full width and height of the door opening, thereby minimizing both the infiltration of room air into the furnace chamber and disruption of the furnace atmosphere inside.
- To serve as a positive ignition source of the combustible atmosphere that escapes through the door when it is open.

Design

Most flame-curtain systems are engineered to utilize your plant natural gas, propane or aerated propane supply regulated down in pressure to a stable 4–6-inch water column (2.30–3.45 psig). Air is supplied by either your plant high-pressure air supply adjusted to 80–100 psig, a small combustion-air blower or by tapping into the existing combustion-air blower if your system is gas fired.

For high-pressure systems, the air line should include a venturi-type inspirator, which converts a relatively small flow of high-pressure air to a much larger flow of air at low pressure. A globe-type valve in the air line, ahead of the solenoid valve, is the "shutoff valve" used when shutting down the furnace and flame curtain. A needle-type valve in the air line, downstream of the solenoid valve, is the "flow-adjusting valve." Having once been properly set at the time of initial start-up of the furnace, the setting of this valve normally will not be changed.

The flame-curtain gas- and air-solenoid valves are connected in parallel to the door position-sensing limit switch. Thus, as the door just begins to open, both solenoid valves open simultaneously and the curtain "lights-off" and continues to fire until the door has been closed. As the curtain is engineered with a very high firing rate, it generates considerable heat. To prevent possible damage from heat distortion to

Fig. 1. Properly adjusted furnace flame curtain

the doorframe and/or door plate, the door must never be held open any longer than required to turn on and initially adjust the flame curtain or to subsequently load or unload the furnace.

Whether you have a high- or low-pressure system, full coverage of the door opening must be routinely checked by operators, supervisors and maintenance personnel to determine if/when adjustments are needed. Variation in plant air pressure is the most likely reason flame curtains go out of adjustment.

Operating Safety Requirements

The key operating safety requirements for most furnaces with respect to the flame curtain are:

1. Whenever routinely starting up a furnace and before opening the door to turn on and verify adjustment of the flame curtain, you must first turn on the flame-curtain pilot, ignite it and verify it to be burning with a good, strong blue flame. Be sure that drafts or other sources of air do not move the pilot flame into a position where it cannot ignite the flame-curtain air/gas mixture when the door opens.
2. Whenever the furnace is in operation, you must never open the door without first verifying the flame-curtain pilot to be burning with a strong, stable blue or blue-yellow colored flame.

Table 1. Maximum flame-curtain gas and air demands

Flame-curtain length, inches	Natural gas, scfh	Propane, scfh	Aerated propane, scfh	Air, scfm	Air, cfm @ 80 psig
24	234	94	164	1.39	0.09
30	293	117	205	1.71	0.11
36	351	140	246	2.17	0.14
48	468	187	328	2.80	0.18
60	585	234	410	3.57	0.23

3. Whenever you wish to leave the furnace idling at temperature overnight or over a weekend with atmosphere flowing, you must verify – before you leave – that the flame-curtain pilot is burning and properly positioned.
4. Whenever you leave the furnace shut down and either still cooling or already cooled, you must make absolutely certain that the flame-curtain gas- and air-line shutoff valves have been fully closed. You must also do this if you leave the furnace idling at temperature with atmosphere not flowing.

Typical Initial Starting and Adjusting Procedures

The following procedure is typically required to initially turn on and adjust a high-pressure flame curtain:

1. Remove the protective cap over the gas-air ratio-adjusting screw in the gas-air mixer on the end of the air-line inspirator. Then, turn the screw in all the way, clockwise, until you feel it “bottom.”
2. With the furnace door fully closed, open the shutoff cock in the curtain gas line and the small cock in the pilot gas line and, without delay, light the pilot. Adjust the pilot so that it burns with a good, strong, stable blue flame. A blue flame without orange or

yellow tips indicates the pilot is burning at close to the required 10:1 air-gas ratio (perfect ratio) on natural gas, or 25:1 on propane or 14:1 on aerated propane. If the pilot is equipped with a supervising flame rod and flame-detection relay, the relay contacts are now closed.

3. Fully open the air-line shutoff valve ahead of the solenoid valve. Then, stand as far away from the furnace door and flame curtain as possible and open the door. As the door begins to open, the gas and air solenoid valves open but gas does not yet flow.
4. Set the air-line flow-adjusting valve downstream of the solenoid valve so that the air-pressure gauge reads 5–10 psig.
5. Verify that the pilot is still burning with a strong flame and start turning the gas-air ratio-adjusting screw in the gas-air mixer out, counterclockwise, to start gas flow to the curtain burner. Continue until the burner lights-off and the essentially raw-gas flame extends in height to approximately the top edge of the door opening (or the crown of the door vestibule arch). Note: If the flame curtain cannot cover the entire door opening, the system is undersized and must be upgraded before operating the furnace.
6. Without delay and only if required to make the flame transparent enough so that you can see through it, further open the curtain needle-type air-flow adjusting valve downstream of the solenoid valve to add more primary air to the mixture. As you add air, the height of the flame will lessen somewhat and the flame will become less orange or yellow in color.

   Proper air-gas ratio adjustment (Table 1) will leave the flame curtain burning very rich (at approximately a 1.2:1 ratio). If not enough air is added, the flame curtain will produce soot (carbon deposits) around the door, which is undesirable and indicates an adjustment to the air-gas ratio is needed.
7. Without delay, close the furnace door to turn off the flame curtain.
8. Verify that the flame-curtain pilot is still burning with a strong blue flame of adequate size. Make any adjustments required to obtain a proper flame.
9. Lock the gas-air ratio-adjusting screw in the mixer in position with the jam nut and replace the protective cap.

Routine Starting and Adjusting Procedure

The required procedures to routinely turn on the flame curtain are:

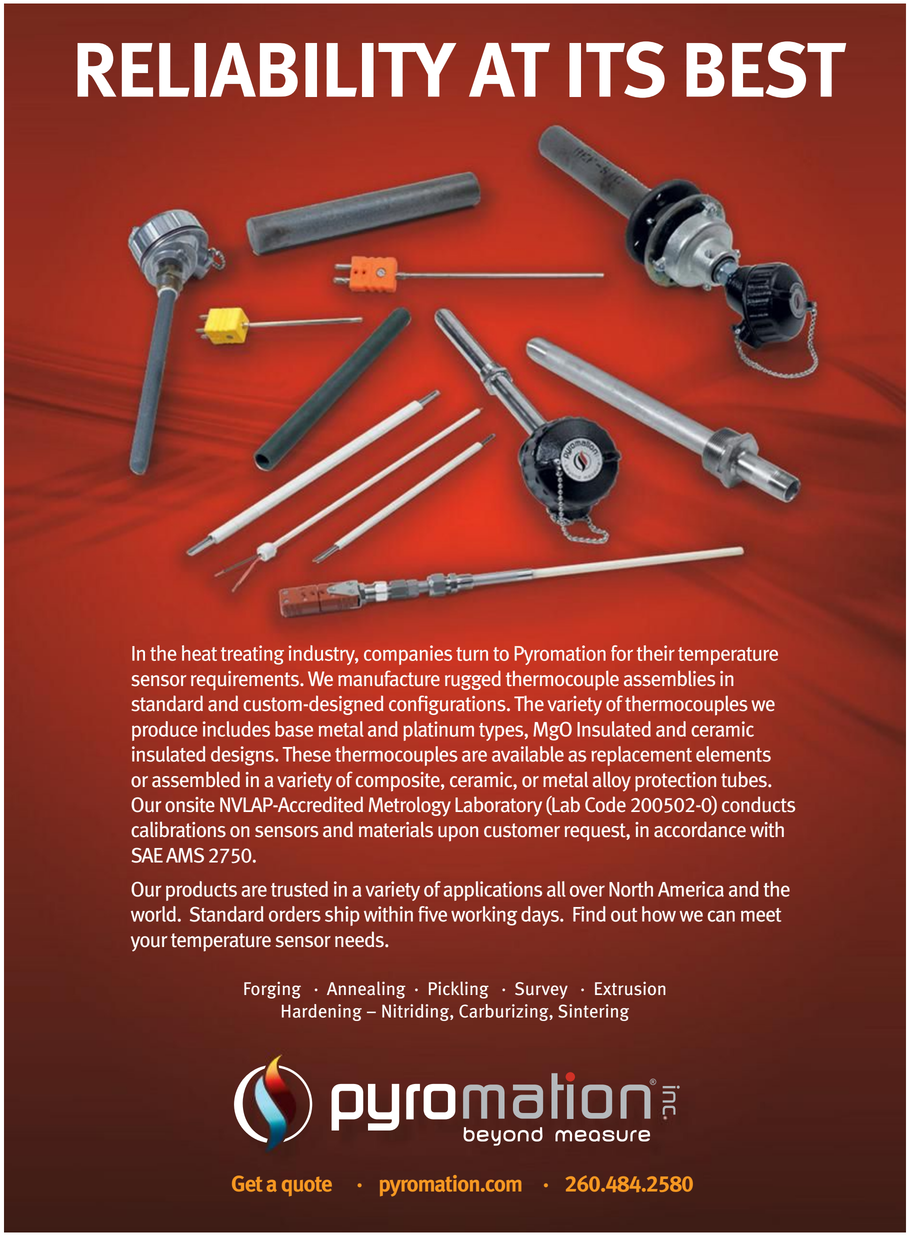
1. Verify that you have already brought the furnace to above 1400°F (760°C), that you have started

atmosphere flow into the furnace, that you have observed atmosphere to be burning into the furnace chamber off the end of the inlet pipe with a good, strong flame and that you have fully closed the furnace door.

2. Without delay, fully open the flame-curtain gas shutoff cock ahead of the solenoid valve and light both the flame-curtain pilot and (if supplied) the furnace-atmosphere burnoff-can pilot. Verify both pilots to be burning with strong, stable blue flames.
3. Fully open the curtain air-line shutoff valve ahead of the solenoid valve.
4. Stand as far away from the furnace door and flame curtain as possible and open the door. As the door begins to open, verify that the curtain lights-off with a flame of adequate height to cover the full width and height of the door opening and has proper transparency.
5. Without delay, close the furnace door to turn off the flame curtain.

Summary

The flame curtain is arguably one of the most important safety features on a heat-treat furnace. The information above will help keep your furnace running safely.



Experiences with Alloy Fan Problems

In his youth, the Heat Treat Doctor met a furnace designer who was concerned about the life of the horizontal fans he had selected for a pusher carburizer furnace. He convinced his boss to let him go out into the field to see for himself if the fans were operating properly. Upon arrival at the jobsite he was relieved to discover from the customer that the furnace was performing "trouble free." Knowing the fan design was marginal, he gave the furnace a closer inspection. Yes, indeed the fans were running, cooling water was flowing and there was little or no observable vibration. His curiosity peaked, he decided to look inside the unit despite the fact that it was at operating temperature and under atmosphere. Precariously perched at an odd angle and sweating profusely, he looked in only to discover that all the blades had been torn from their shafts and were embedded in the sidewalls of the furnace! The shafts however continued to spin freely. The engineer climbed down from his perch, complemented the customer on his excellent furnace performance and returned home thoroughly shaken and having adapted a new (but regrettable) attitude - what the customer doesn't know won't hurt him!!

Today, what we don't know *will* hurt us. Fans in all types of furnaces and ovens can be critical to the successful performance of the heat treatment operation. There are many styles and types, but all of them must be properly designed for the intended use and equally important, properly maintained. All fans face a variety of common problems, which must be overcome. Let's learn more.

Types of Fans

There are a number of different fan styles (Fig. 1 - 2) that are used throughout the heat-treating industry depending on application and include blowers, squirrel cage fans, radial blade fans and axial blade fans. These are typically divided into two general classes: centrifugal fans and propeller (axial flow) fans. This classification is functional; the fundamental difference between them involves the way in which air or atmosphere is moved.

Mechanical Problems

These types of problems typically involve such items as bearings, belts, motors, and wiring. Should these components fail, it is important to evaluate why they

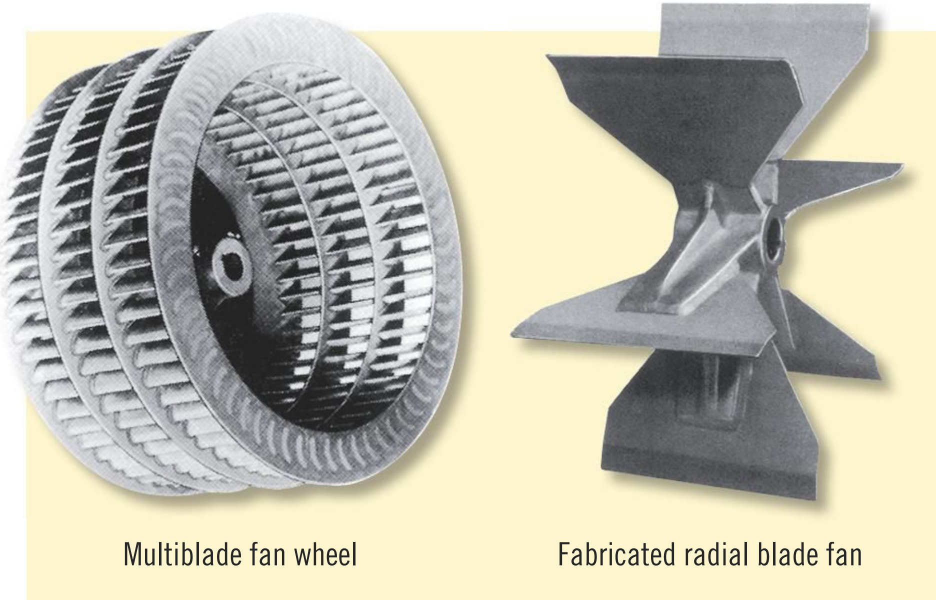


Fig. 1. A few common fan designs used in heat-treat furnaces.

failed or why they failed prematurely. Often it is a good idea to determine the MTBF (mean time between failure) of these components. Their failure may be a symptom of a much deeper problem – a design, material or performance issue.

One of the central mechanical issues often involves bearings. Many designs use special bearings or special lubricants, and substitution of a standard bearing may cause premature failure and unexpected downtime. In one instance a well-intentioned manufacturer modified standard bearings by substituting undersized balls and packed them with a special high-temperature grease but failed to inform his customers, who were baffled as to why when they purchased the same part number from a catalog, the bearings didn't perform as well. It is important to also remember that after a repair, fan performance must match the original equipment manufacturer's specifications. If not, either process or quality issues may arise after repairs are complete unrelated to the "mechanical" performance of the fan. Finally, it is not uncommon to find designs with top and bottom bearing spaced too far apart causing the fans to wobble or vibrate in service shortening their life.

Power Failures

A hidden problem in many fan applications, especially for fans operating at high temperature is that on restart after a power failure, the torque (stress) exerted on the fan blades can be enormous given the fans weakened strength at temperature. These starting forces can be great enough to literally tear the blades from the hub. This happens far too often. Backup power supplies or "soft start" features avoid many of these problems.

Fan Construction

Fan design is a function of the working environment, and often times how a fan is constructed makes a huge difference. Some fans are one-piece castings while others consist of two-piece assemblies. Cast blades pressed or welded onto a fabricated shaft are common. Still other fans are made only of fabricated components. The method of welding and the service environment should be carefully matched. It is common to find weld cracks in the area of the hub. Cracks grow rapidly and if discovered, the fan should be replaced immediately. Remember, fans should be inspected whenever the furnace is cooled down. Carburizing and high temperature applications are the worst.

Fan Speed

Many atmosphere control, uniformity and stability problems are directly attributed to fan performance. One interesting example involved a continuous carbonitriding furnace with an atmosphere stability problem so severe that carbon was observed dropping out of the atmosphere in the area of the furnace belt support rolls, which would lock up. The unit had to be shut down every 2 – 4 weeks for maintenance. In addition, the burn-off at the front end was erratic, alternately sucking air in and violently blowing out endothermic gas; variable case depths and case uniformity was also observed. The problems disappeared when it was discovered that the fans were not operating at the proper speed and the speed adjusted to the manufacturer's recommended settings.

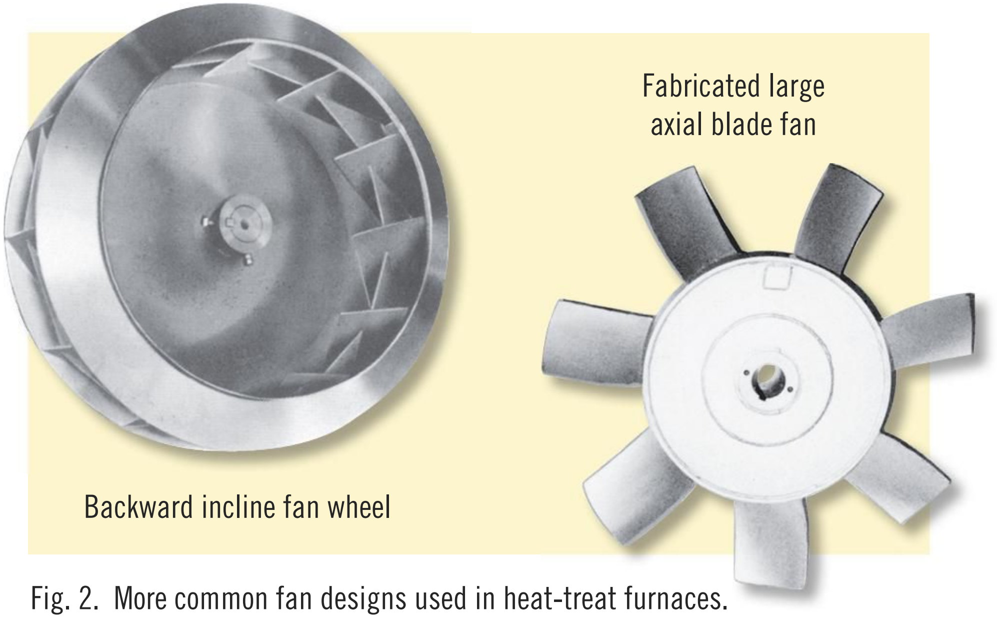


Fig. 2. More common fan designs used in heat-treat furnaces.

Fan Balancing

It is important to know if the fan you use has been statically balanced, dynamically balanced (and at what speed) or both. A number of problems with short fan life in integral quench and pusher furnaces have been directly traced to the failure of the manufacturer to balance the fan at full rated speed. In one noteworthy case, fan problems persisted for over seven years despite design and material changes until the retirement of the person in the balancing department, who as it turned out didn't see the necessity to balance the fans at speed although the instructions clearly indicated he should.

Fan Direction

Amazingly, some fans can be run backwards for most of their life while metallurgists and heat treaters question poor quality and equipment performance. Other fans literally unscrew if run in the wrong direction and have been found atop baskets of parts after their removal from the furnace. Few fans are designed to run in either direction, and a simple rotational check can avoid major headaches.

Materials of Construction

Cost and availability are important issues with respect to deciding which alloy and design to use. Not all fan alloys are created equal. A common tendency based on price, availability or familiarity is to universally use a standard alloy such as 330 (35% Ni, 15% Cr) and not consider other compositions. For example, sulfur present in some atmospheres will eat away nickel and leave a metallic sponge of porous material behind. Using a material with at least 25% Cr will increase the thickness of the oxide layer on the alloy fan surface. The increased chromium will also react during furnace (air) burnouts or to oxygen leaks by reforming these oxides. Also, having the right chemistry improves resistance in areas most susceptible to hot gaseous corrosion. The increased hot strength of a more suitable alloy grade will extend the performance life.

Type of Atmosphere

Hot gaseous corrosion and metal dusting are several of the phenomena that routinely occur with fans running in hostile environments such as carburizing. These problems often are more severe after repair or reinstallation of a fan due to damaged (or missing) refractory in the area immediately adjacent to the fan shaft or due to the introduction of a small air leak. Furnace roofs have been known to overheat and steel shells warp if insulation is missing or a furnace plug has been damaged causing bearing problems and excess vibration.

Cooling issues

Water cooled fan bearing housings are notorious for leak due to porous castings and can radically change the dew point inside the furnace. Air-cooled designs are becoming more popular for this reason.

Summary

Fans are a necessary evil in most heat-treating furnaces. They are often overlooked with disastrous consequences. Take a few minutes to understand them and you will be rewarded with hours and hours of time to do other things.

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Proper Care and Feeding of Furnace Mesh Belts

Mesh belts come in all shapes, sizes, materials and weaves and are used for such diverse applications as case hardening, brazing, sintering and glass-to-metal sealing to name a few. Belts run at temperatures from near ambient to several thousand degrees. They are expected not only to work in the furnaces but also for external part conveyance or in water, oil, brine, polymer and salt quench tanks. Mesh belts are exposed to a multitude of furnace atmospheres ranging from air to pure hydrogen and can be exposed to oxidation, sulfidation, carburization and nitriding. They operate in environments spanning dew points from below -73°C (-100°F) to above +38°C (+100°F). And most of us expect our belts to perform well beyond normal expectations. How do they survive, and how can we make them last even longer? Let's learn more.

When operating a mesh-belt conveyor furnace (Fig. 1), the goal should be to maintain a consistent temperature profile and uniform belt loading for any given part number over time. Premature or abnormally short belt life is often the sign of overloading, misapplication, abuse, neglect or ignorance. The vast majority of belt failures are either due to overloading or poor belt-system adjustments. Following some simple advice may extend your current belt life considerably.

Tips to Improve Belt Life

1. Know your belt speed by actually timing the belt movement. Do not assume it is what is stated in the instruction manual or what is displayed on a digital indicator. Calibrate the speed control (typically a magnetic or inductive pickup device) as often as your temperature instruments.
2. Determine your belt loading over time. Improperly adjusted vibratory feed systems and weigh scales that do not read correctly are two of the most common causes for improper loading of furnace belts. Load as uniformly as possibly to evenly distribute wear across the bottom of the belt and help to prevent camber and other belt distortion problems.
3. Avoid using side skid plates or belt guide rollers to help belt tracking. These items tend to do more harm than good. Where skid plates must be used, consider coating them with laminated plastic strips. Observe the motion of the belt – it should be smooth, not jerky, and maintain

Fig. 1. Typical mesh-belt-furnace part loading (Courtesy of Surface Combustion, Inc.)

a consistent speed and tracking over time. Remember, a metal mesh belt has flexibility along its length, semi-rigidity across its width and rigidity in its thickness.

4. Work with your equipment or belt suppliers to select a belt weave and alloy best suited for your process and parts. Be aware that some belts using heavier wire or larger crossrods may decrease furnace throughput. Avoid upturned-edge belts whenever possible.
5. Flip and/or reverse the belt at frequent intervals as signs of wear or camber become evident. Camber in a high-temperature belt may be either convex or concave in the direction of travel, or in some cases it may develop with one edge of the belt leading the other edge. It is frequently an indicator of other problems such as uneven loading, uneven cooling, improper drive tension (see #7 below) or a combination of these factors. Only flip and reverse the belt when it is sufficiently flexible to ensure that reversing of the belt does not create a fatigue problem.
6. Be sure that the belt is properly supported over its length. Watch for signs of premature failure such as abnormal distortion of the crossrods, flat spots, deterioration of belt edges, contamination/buildup of foreign residue and tracking problems.

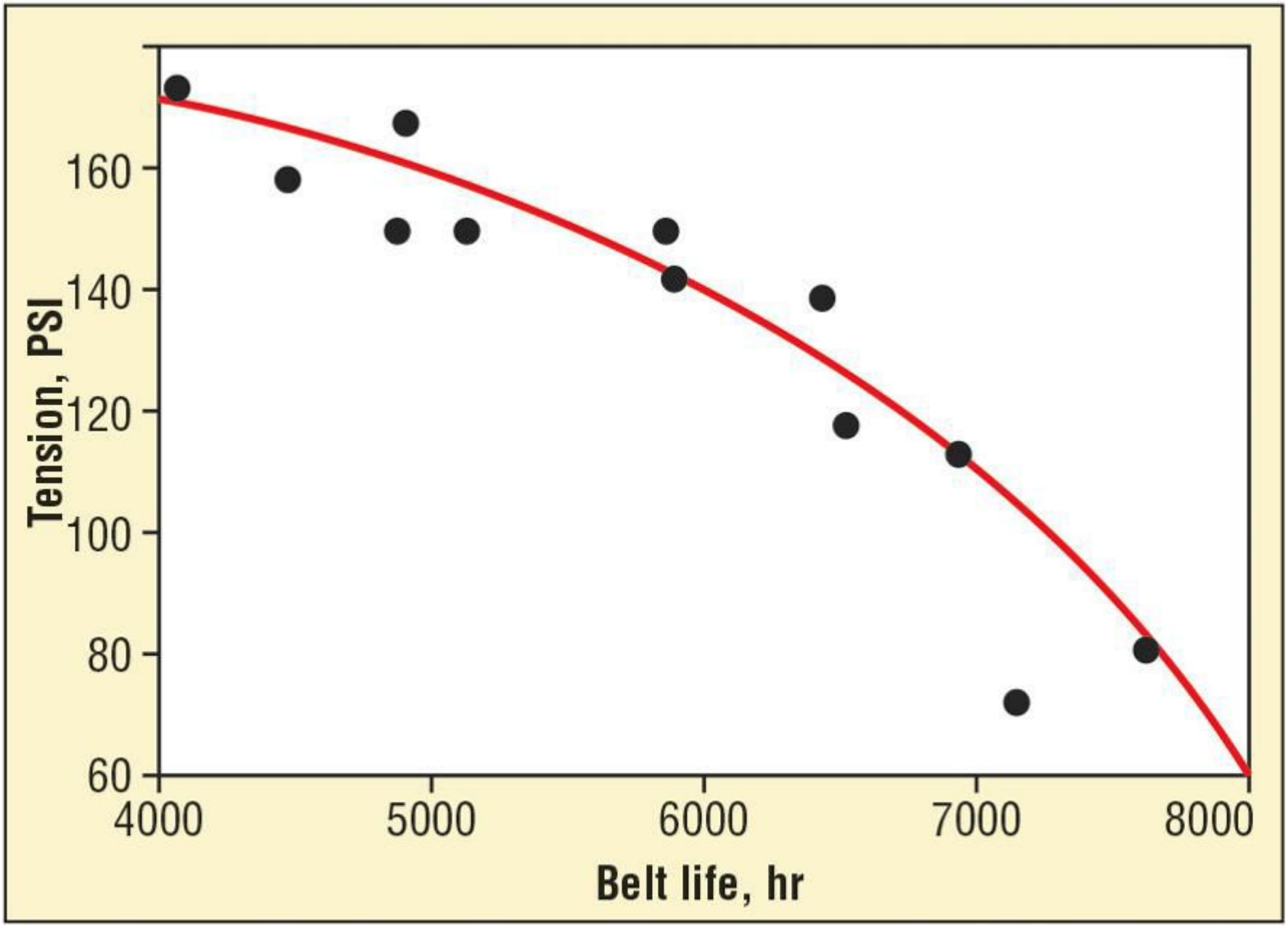

Fig. 2. Effect of belt tension on service life[2]

7. Check the tension on the belt frequently or as dictated by production usage. Some systems rely on springs to maintain tension, so check their length. Other systems rely on cylinders. Therefore, verify that plant air pressure does not fluctuate significantly. Remember, check the system for compliance to its design specification.
8. Have your belt drive system analyzed by a furnace manufacturer or belt company at least once a year.
9. Keep loading consistent. Many parts are loaded on the belt in such a way as to leave space along the edges to prevent parts from moving off the sides. This concentrates loading over a smaller square area and may affect belt selection or life.
10. Understand that maintenance is the difference between front-end-drive systems having pinch and tangential rolls (for tracking) and rear-end-drive systems having only pinch rolls.

Other Important Considerations

Run belts operating at or near ambient temperature or in quench tanks both unloaded and loaded for a minimum of three revolutions after final belt tracking adjustments and prior to use. Anticipate issues such as the environment (e.g., dirt, chips) or thermal expansion (e.g., austempering) before putting the belt into service.

For high-temperature belts, two frequently overlooked procedures are “break in” and “stress relief.” Take the time to do both. Though opinions differ on how to best perform break in, most agree that, if possible, the furnace should be brought up slowly <150°C (300°F) per hour to an operating temperature of 815-870°C (1500-1600°F). The belt should be allowed to complete 2-4 revolutions through the furnace (at least 4-6 hours at temperature with no load) at the lowest practical belt-speed setting. This will ensure proper seating of the spirals and allow for initial movement of the rods. Belt tracking adjustments should be made during and after this procedure.

Stress relief should be performed immediately after the break-in procedure is complete. For belts in furnaces operating below 900°C (1650°F), a stress relief at 10°C (50°F) above the normal operating temperature is required with the belt running at a speed to ensure that every portion of the belt reaches this temperature for at least one hour. For belts in furnaces operating above 900°C (1650°F), the furnace should be brought into the 925° - 955°C (1700 - 1750°F) range and allowed to complete 1-2 revolutions (at least 1-2 hours at temperature with no load) at the lowest practical belt-speed setting. This will help prevent excessive grain growth and embrittlement in service.

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After the stress-relief treatment, the furnace should be brought to normal operating temperature and the belt operated unloaded for as long as practical. Some manufacturer's recommend 4-6 hours, others up to 100 hours! The more time, the greater the improvement in creep strength.

Proper belt tension (Fig. 2) is also critical, as illustrated by a study conducted by a major belt manufacturer on copper brazing furnaces operating at 1120°C (2050°F). The results are reported to be applicable to all types of high-temperature belts and make the point that every effort should be made to maintain operating tension at the lowest practical level. Either the original equipment manufacturer or the belt supplier has usually predetermined the allowable tension range. If the furnace was not purchased new, it is even more important to know how the drive was sized, where the zero point of loading is and what the proper drum adjustments and belt tension settings are.

Finally, when possible, keep the belt clean (free of oxidation and scale) by running it under protective atmosphere above 760°C (1400°F). In certain situations, such as copper brazing, it has been reported that preoxidizing the belt can extend its service life where spills of copper or other braze alloys attempt to penetrate the surface of the belt.

Summary

The condition of your mesh belt is a clear indication of the health of your mesh-belt furnace. Insist on the extra time to properly take care of this vital component. You will save time and money!

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1. Mr. Robert Brodeur, Chief Engineer, C. I. Hayes, private correspondence.
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Health and Safety in the Heat-Treat Shop

When most of us think about health and safety in the heat-treat shop, we tend to focus on things that are hot, heavy or dangling overhead. The threats we can see, smell, hear or perhaps even taste are most often dealt with quickly. The old adage, "what you can't see, won't hurt you," doesn't apply in our industry. Let's learn more.

Lesson Learned

I recall as a young engineer that when called upon to inspect the inside of a cold, well-ventilated batch integral-quench or pusher-type furnace that had been running endothermic or exothermic gas atmosphere, I would often experience mild headaches after an hour or so inside. I remember carrying aspirin with me for just such an occasion. Little did I realize at the time that furnace atmosphere was slowly being released from the brickwork and that, in this confined space, I was experiencing the early symptoms of gas poisoning.

Dangers of Carbon Monoxide

Carbon monoxide (CO) is an odorless, colorless, toxic gas present in many heat-treat shops from furnace atmospheres and products of (incomplete) combustion. The symptoms of carbon-monoxide-gas poisoning, defined as levels of 10% or higher absorbed gas in the bloodstream, include in descending order of danger:

- Headaches
- Dizziness
- Irritability
- Confusion/memory loss
- Disorientation
- Muscle aches/poor reaction time
- Balance/coordination problems
- Nausea and/or vomiting
- Difficulty breathing
- Chest pain
- Swelling of the brain (cerebral edema)
- Convulsions/seizures
- Coma
- Death

Carbon monoxide has powerful effects (Table 1) even at very low-dosage exposures and often begins with "flu-like" symptoms such as being tired, having achy muscles, a headache that just won't go away, eye strain and even a runny nose.

Table 1. Physiological effects of carbon monoxide[1]

Concentration, ppm	Physiological Effect
100	Allowable limit for an exposure of several hours
400	One-hour exposure at this level will be without appreciable effect.
600	Low dosage symptoms begin after one-hour exposure.
1,000	Severe side effects but often no permanent damage after an exposure of one hour
1,500	Dangerous health consequences for exposures of one hour or longer
4,000	Fatal for exposure of less than one hour

When carbon monoxide enters the body, it is absorbed into the bloodstream, preventing oxygen absorption and the transfer of oxygen to vital organs such as the heart, central nervous system and brain. The heart responds by beating more rapidly and irregularly and by decreasing blood pressure. In extreme exposure conditions, a life-threatening neurological condition results due to the destruction of brain cells.

Monitoring the heat-treat environment coupled with proper fresh-air ventilation is the key to combating this problem. One of the most important considerations in the treatment for carbon-monoxide exposure is the immediate recognition of the problem. That's why CO detectors should be installed in every heat-treat shop. Does yours have one?

Once detected or suspected, the following actions can be taken:

1. Move the victim(s) to fresh air.
2. If the victim(s) are experiencing any gas poisoning symptoms call for trained paramedics and activate your company's emergency plan.
3. Warn others and ventilate the affected area.
4. Monitor the victim(s) for respiratory problems.
5. Try to ascertain the source and shutdown suspect equipment until trained professionals can assess what is wrong.

Dangers of NOx

NOx (pronounced "knocks") emissions is the generic term for a group of highly reactive gases that contain combinations of nitrogen and oxygen in varying amounts – nitrogen monoxide (NO) and nitrogen dioxide (NO_2) being prime examples. NOx is responsible for a wide variety of long-term health and environmental problems, so it is important for the heat treater to understand that most combustion processes, like those that take place in our gas-fired furnaces (Fig. 1), can be a source for NOx emissions. These pollutants spread out over great distances, which means that problems associated with

NOx are not confined to just the heat-treat shop. These effects include:

- Smog (ozone) – formed when NOx and volatile organic compounds (VOCs) react in the presence of sunlight.
- Dust (particulate material) – NOx reacts with ammonia, moisture and other compounds to form solid particles that permeate the air we breathe.
- Acid rain – NOx and sulfur dioxide react with other substances in the air to form sulfuric, nitric and other acids that fall back to earth as rain, fog, snow or dry particles.
- Oxygen-depleted bodies of water – NOx emissions increase the absorbed nitrogen level in water, upsetting the chemical balance of nutrients used to support life.
- Toxic compounds – NOx reacts in the air with common organic chemicals and ozone to form a wide variety of toxic chemicals and by-products.
- Greenhouse gases – Nitrous oxide (N_2O), a member of the NOx family, is a greenhouse gas contributing to climate change.
- Global dimming – Nitrate particles and nitrogen dioxide block sunlight.

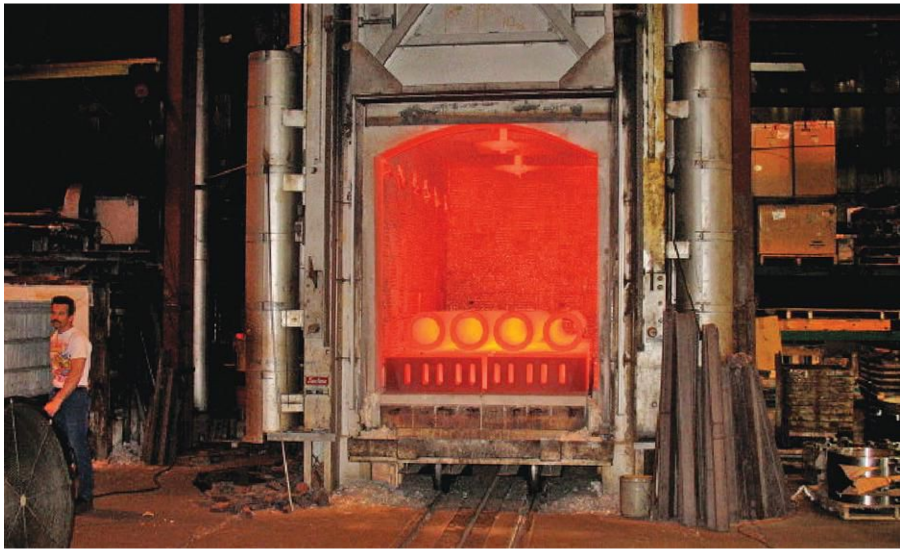

Fig. 1. Typical gas-fired furnace with low-NOx burners at a commercial heat treater

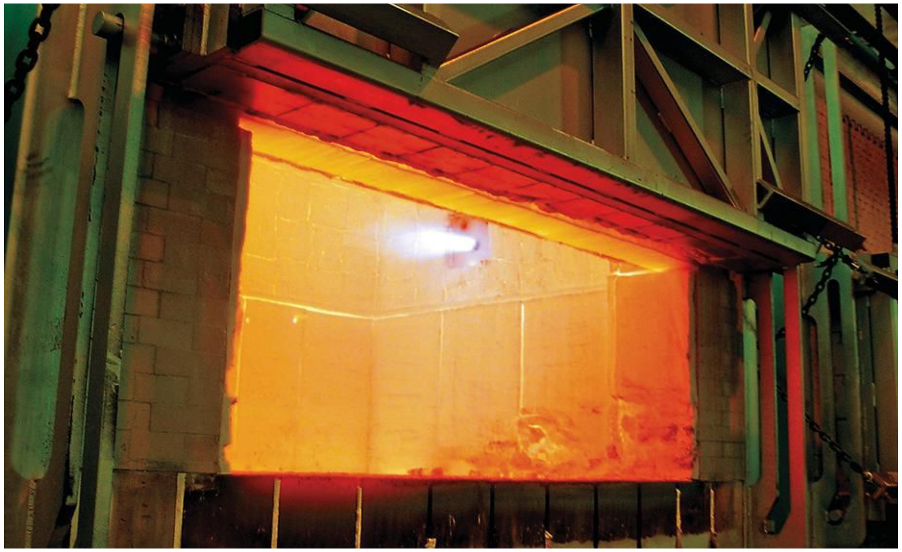

Fig. 2. Low-NOx burner in operation

Table 2. Physiological effects of oxygen deficiency

Concentration (%)	Physiological Effect
19.5	Legal minimum concentration for humans per OSHA
15 - 19.5	Decreased ability to do work; induce early symptoms in persons with coronary, pulmonary or circulation problems
12 - 14	Increased pulse rate and respiration, impaired perception and judgment
10 - 12	Further increase in pulse and respiration, giddiness, poor judgment, blue lips
8 - 10	Mental failure, nausea, fainting, vomiting, unconsciousness
6 - 8	4- to 5-minute exposure, full recovery; 6-minute exposure, 50% fatal; 8-minute exposure, 100% fatal
4	Coma in 40 seconds, convulsions, breathing stops, death

Reducing NOx Emissions From Furnaces

The amount of NOx emissions is a function of the type (composition) of the fuel being burned, combustion conditions, burner design and flame temperature. "Thermal NOx" is the result of the conversion of atmospheric nitrogen and oxygen and is dependent on the reaction temperature, residence time and gas chemistry (stoichiometry). Long residence times at high temperature contribute to thermal NOx formation as does rapid mixing of combustion air (oxygen) with the fuel. "Fuel NOx," by contrast, is the conversion of chemically bound nitrogen in the fuel with oxygen. It also increases with rapid mixing.

Incomplete combustion products can usually be held to industry minimums by the proper operation of modern burner equipment (Fig. 2). There are also several strategies for reducing NOx that rely on either post-combustion treatment of the gas or upgrading to different types of burners. Do you know your shop's total NOx emissions? You should. Here are some methods to better control how much NOx is being emitted:

- Flue gas recirculation (FGR)
- Selective catalytic reduction (SCR)
- Selective noncatalytic reduction (SNCR)

Upgrading burner designs is yet another way to maintain control over NOx emissions. Options include the use of:

- Staged air burners
- Staged fuel burners
- Ultra-low NOx burners
- Low excess-air burners

Other Safety Threats

When dealing with any gas (including nitrogen), always be aware of its inherent dangers and understand the causes of asphyxia. For example, oxygen may be displaced to a level of around 16% of the gas/air mixture before symptoms begin to appear (Table 2). Health and safety are everyone's responsibility and avoiding accidents in the heat-treat shop will make it a better place for all of us.

References

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2. Garg, Ashutosh, "Trimming NOx from Furnaces," Chemical Engineering, November. 1992.
3. Various websites (www.carbonmonoxidekills.com), (www.ccohs.ca/oshanswers/safety_haz/metalworking/heat_treating.html)

Identifying Sources of Oil Quench Fires

Oil quenching presents some of the greatest safety hazards in heat treating, and often results in personal injury, equipment damage, business interruption, environmental clean-up, and possibly litigation expenses. The heat treater needs to know some of the most common sources of a potential explosion or fire. Let's learn more.

Presence of water

One of the most dangerous types of industrial fires can occur when the quench oil contains water. Even minute amounts of water (0.10%) are cause for concern. Quench oil should be continuously monitored and routinely checked for the presence of water, and contaminated tanks should be pumped and separation techniques used. Because water will accumulate in the bottom of the quench tank, the sampling method must take this into account.

Integral quench and pusher furnaces are especially vulnerable to this problem, but it is also seen in open quench tanks. The source of water contamination may come from a leaking water-to-oil heat exchanger, from a leaking jacket in the atmosphere cooling section if this is water cooled and any other area that is water cooled. Furnaces down for maintenance can easily be contaminated if the quench tank area is open.

Oil drag out

Most furnace loads are allowed to drain over the oil prior to being removed from the quench tank. However, insufficient drain time or part/workload geometry may be such that a significant quantity of oil remains on the parts. This is called drag out. When the load passes through the door flame curtain it could ignite, and the resultant fire is extremely dangerous to personnel trying to extinguish the fire and close the front door. In rare instances, insufficient quench time results in a load that is still above the self-ignition point, and upon removal of the furnace, a fire will result.

Load hang-up

One of the most dangerous types of furnace related fires occurs when a load being transferred to the quench "hangs up," or becomes stuck partially in and partially out of the oil. The oil at the interface between the hot load and the surface generates

Fig. 1. Quenching into a polymer tank

huge amounts of oil vapor, and in many furnaces these fumes are carried out with the furnace atmosphere and ignited by a pilot. This results in a large flame, or torch, condition that exists while the workload slowly cools. This type of phenomena can also occur when the quench elevator jams, or in other words, when its travel is restricted due to a critical failure of plant air pressure, hydraulics, or when mechanical malfunction occurs. In some designs, low air pressure can cause the elevator to slowly lower into the quench oil resulting in an unsafe condition.

Even the presence of a nitrogen safety purge system does not guarantee that this situation can be controlled. In one instance, an integral quench furnace was responsible for burning down an entire plant when the initial fire spread to the adjacent plating department, igniting its fiberglass ventilation system. The operator was told to turn the furnace and nitrogen purge system off because the fire would burn itself out, an action that resulted in a catastrophe.

Exhaust Stacks

Fires in exhaust stacks are all too common occurrences. This may be due to improper design of the stack itself, improper airflow and improper maintenance. Oil fumes carried out of the furnace accumulate in the exhaust stacks. Several ignition sources, such as the rapid evolution of furnace gases, can trigger a fire that is not easily controlled because of its ability to spread quickly in areas that are difficult to access with fire suppressant materials. Awareness of the potential for a fire and adequate maintenance steps are important in avoiding this situation.

Inner Door Open During Quench

To speed up transfer time from the heating chamber

Fig. 2. Quenching into an oil tank

to the quench, especially for thin parts that are prone to heat loss, some furnaces allow quenching to take place while the inner door between the heating chamber and the quench tank is still open (or is being closed simultaneously with the lowering of the furnace elevator). Some of the most devastating plant fires have occurred in just such a manner. The evolution of gas during the transfer in combination with the oil volatilization on quenching can combine to shoot flames up and out of the furnace.

High Surface Area Loads

In certain circumstances, it is impossible to avoid running a workload having high surface area. Fasteners and other small components that tend to “nest” very densely present the potential to volatilize a large amount of oil when quenched. These oil fumes can be carried out of the furnace and ignited. Even when precautions, such as positioning and manning fire extinguishers, have been taken, the unexpected can happen. In one particular industrial fire, flames shot over 30 feet in the air, igniting the supposedly fire retardant roof insulation and overwhelming the fire suppression equipment on hand.

Oil Characteristics

Depending on the heat-treating requirements of the parts, quench oils are used over a wide range of process variables including temperature, agitation, viscosity, quench speed and contamination. All of these variables affect oil vaporization, oil drag out and oil-quench characteristics. In extreme cases where the variables are not properly controlled or are allowed to change over time, what was a normal, safe quenching operation may turn into a potentially hazardous operation.

Inadvertent Oil Discharge

When "burning in," or introducing furnace atmosphere, into the quench areas of an integral quench or pusher furnace, pockets of air may remain, and as they suddenly ignite, result in a pressure buildup on the surface of the oil. This presents the real danger of an oil "burp," or discharge, out of the quench tank. Even overflow pipes that discharge to containment drums may suddenly be over capacity and spill oil out into the pit area. In rare cases, the pressure buildup is so severe that oil is dumped out the front door often igniting if a pilot has been lit or another source of ignition is present.

One particularly damaging plant fire was due to a relatively small floor fire spreading into a pit area that contained an estimated 3 to 4 inches (75 to 100 mm) of oil from repeated (and ignored) oil discharges from the quench tank. Extensive equipment and building damage resulted.

Irregular Parts

When quenching irregularly shaped parts or loads, such as long tubes having hollow internal diameters, from pit furnaces into open quench tanks, oil "spouts"

Fig. 3. Smoke generated from oily loads being tempered

and flames can literally shoot up and out from the inside of the tubes forming an oil fountain in a spectacular, albeit dangerous, display. Factory ceilings have been known to catch fire due to the height and internal pressure developed. Extreme precautions are needed, paying particular attention to areas immediately adjacent to the quench tank.

Oily Parts In Washers

Successful cleaning involves the choice of the correct cleaning solution chemistry together with the correct use of time, temperature and energy. Equipment design, age and use, and ancillary devices such as oil skimming are important to ensure that a minimal amount of oil will remain on the parts when they are transferred from the washer into the tempering furnace. In some cases, such as with powder metallurgy parts, due to their inherent porosity, they can retain up to 3% (by weight) oil despite the best washing practices available.

Oily Parts In Tempers

If the tempering furnace or oven is operating above the flash point of the oil, introduction of an oily load can cause an immediate and uncontrolled explosion. Even if the unit is operated below the flash point, oil can accumulate over time, and raising the operating temperature for another process or attempting to perform an elevated temperature “bake out” can result in a fire or explosion. This is one of the most common hidden dangers of oil quenching.

While these dangers exist in oil quenching practice, the potential hazardous conditions can be minimized through clear, concise safety training.

Summary

Identifying sources of problems with quenchants, especially those related to oil quenching, will help avoid unplanned downtime in the heat-treat shop.

It's Howdy Doody Time!

It was September 21, and the sun was shining on a bright and seasonably cool day in 1971 as a young, enthusiastic and incredibly naïve 21-year-old metallurgical engineering graduate from the University of Illinois walked toward his first full-time employer's manufacturing plant in Chicago (a furnace manufacturer no less) full of hopes, dreams and aspirations. Curiously enough, as he crossed the parking lot toward the entrance, he remembers saying to himself, "Well, only 45 years to go!" Let's learn more.

Enter Howdy Doody

For those of you too young to remember, Howdy Doody was an American children's television program (with circus and Western frontier themes) that was created and produced by E. Roger Muir and telecast on the NBC network in the U.S. from Dec. 27, 1947, until Sept. 24, 1960 (Fig. 1). It was a pioneer in children's television programming and set the pattern for many similar shows. One of the first television series produced at NBC in Rockefeller Center, in Studio 3A, it was also a pioneer in early color production, as NBC (at the time owned by TV maker RCA) used the show in part to sell color television sets in the 1950s.

What I recall most vividly as a very young child watching the show was that each episode began with Buffalo Bob (Howdy's creator) asking, "Say kids, what time is it?" and the kids yelling in unison, "It's Howdy Doody Time!" Then the kids all sang the show's theme song (set to the tune of "Ta-ra-ra Boom-de-ay"):

> It's Howdy Doody time,
> It's Howdy Doody time,
> Bob Smith and Howdy too
> Say "Howdy do" to you!
> Let's give a rousing cheer,
> 'Cause Howdy Doody's here.
> It's time to start the show
> So kids, let's go!

And indeed it was. Howdy Doody was a puppet (but no dummy!) with a whimsical smile and a larger than life personality. He dispensed cowboy wisdom and his own rather philosophical views on life. They left a lasting impression on everyone who tuned in. The show quietly ended with a roll of credits over an empty, darkened set as "Auld Lang Syne" was played on a celeste. Ironically, one of the thoughts that kept entering my mind in later years thinking about that credit roll was that the words to the enthusiastic entrance tune would be fitting when it came time for me to ride off into the sunset.

A Career of Service to the Heat-Treat Industry

Fundamentally, I enjoy helping people. I have never been driven by money (but know it is a necessity of life), or ego, or a need for prestige or accolades. I am not the best, the brightest or the most talented person you will ever meet, but I am one of the most persistent. If I don't know the answer, I will find it, either in my (huge) technical library or from an incredible number of people who I know are willing to share their hard-earned knowledge.

And I am a pack rat (haven't thrown much away since first starting – much to the chagrin of my lovely wife) with a near-photographic memory for heat-treat-related detail, things so trivial at times that one wonders how (and why) it can be recalled. That's simple: There is a story behind each piece of paper, each challenge and each problem solved, and I love stories. Finally, if I say I will do it, I will. If I fail or can't fulfill a promise, I will try until I die to help find someone who can.

If I were to offer any advice to our industry after nearly 45 years it would be to share technical knowledge openly (there are few secrets out there; really, there aren't) and never, ever settle for "good enough." Accept that the quality of the products we produce can never be compromised. Somewhere, sometime, somehow someone's life will depend on what we have done.

Mathematics and the Metallurgist

Did you ever think to yourself that you might be in the wrong profession? I haven't. The "chemistry of metals" as metallurgy was first described to me appealed to my inner self. My father was a machinist for 45 years (ironic, isn't it), and I was in the machine shop with him surrounded by metal and humming screw machines as early as age 6 (yes, Virginia, it was a different era back then).

After switching majors, I quickly discovered (surprise, surprise) that metallurgical engineering is the most fascinating of all the engineering disciplines! Being able to predict what will happen inside a metal component during manufacturing (including thermal processing) without always being able to "peek inside" (metallurgical terminology for cutting up parts) is the type of challenge I embraced. Highly motivated professors and interesting real-world problems kept this day-dreaming boy focused during my wild, wacky and often turbulent college years in the 1960s.

Fig. 1. The iconic Howdy Doody with Buffalo Bob

Of course the long commutes to and from work on public transportation back in the early '70s quickly found me reading books on a wide variety of subjects – from The Lord of the Rings to Quantum Chemistry. One of the books I read early on was, aptly titled A Survey of Modern Algebra by Garrett Birkhoff and Saunders Mac Lane, the latter being a professor at the University of Chicago. This book introduced me to the field of abstract algebra and Galois Theory in particular (if you don't know the story of Évariste Galois, you should Google it).

Like most engineers and scientists, I have always been better than average at mathematics. Until reading this book, I had always focused on applied rather than abstract mathematics (although ironically I did have a half year introduction to the subject in high school). This cover-to-cover read set me up for graduate school "just to prove I could." Children and life interrupted the dream of teaching but could not quench the fire within.

Just to put things in perspective, today I own slightly over 300 metallurgical books and slightly over 3,000 mathematics texts. Need I say more as to where my true passion currently lies?

Where Do We Go From Here?

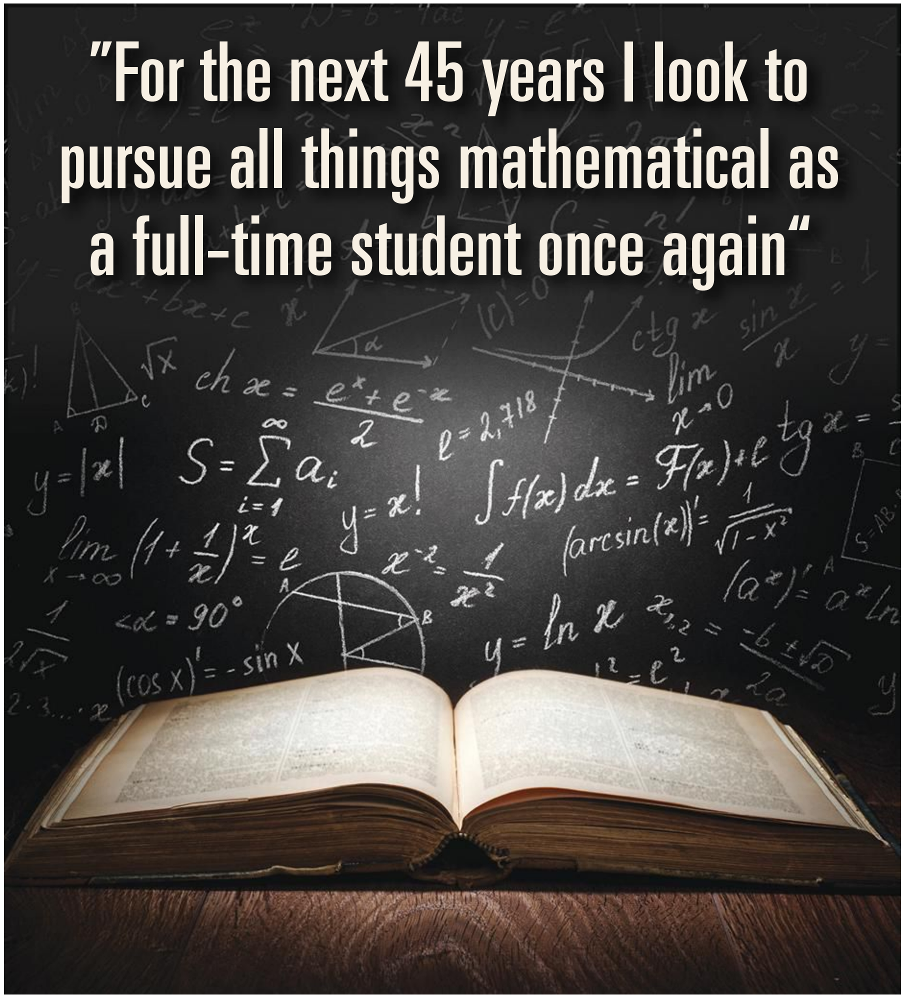
My relationship with Industrial Heating extends back to the early '80s when then-editor, Stan Lasday, asked me to contribute an article to the magazine. What fun! Over the years I have worked closely with Stan, Ed Kubel and Doug Glenn and now Reed Miller and Darrell Dal Pazzo – all fantastic individuals passionate about helping the industry achieve its full potential.

As such, I cannot and will not abandon you, the readers, and so The Heat Treat Doctor column will continue to appear monthly.

However, it is indeed "Howdy Doody Time!" For the next 45 years I look to pursue all things mathematical as a full-time student once again so that I can gain deeper insights into my second true love. I am neither completely closing up shop as a consultant nor stopping my association with Industrial Heating. I am simply becoming very, very selective as to the projects I take on.

The Heat Treat Doctor will be there for you for years to come, and while I have no dreams of being one of the 25 greatest mathematicians of all time (as Évariste Galois, who died at 20, was), it is time for me to take Howdy's advice and nudge my horse down a new path. I leave you with these simple words from a chorus of Auld Lang Syne.

> "And there's a hand, my trusty fiere!
> and gie's a hand o' thine!
> And we'll tak' a right gude-willie waught,
> for auld lang syne."
>
> *- Robert Burns, 1799*



"For the next 45 years I look to pursue all things mathematical as a full-time student once again"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