12 Applications – Chemical Resistance

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ABSTRACT

Hansen solubility parameters (HSP) can correlate differences in physical behavior observed in chemical resistance testing of polymers and polymer-containing systems when a sufficiently large number of different organic solvents are included in a study. These correlations can then be used to predict the chemical attack expected in systems that have not yet been tested. Examples of HSP correlations included here are for solubility, degree of surface attack, tensile strength reduction, and simple evaluations of chemical resistance of the suitable-for-use or not type. Environmental stress cracking is discussed in more detail in Chapter 14. In each case, the molecular size of the liquids used can affect the result and should be considered in some way. Chapter 16 treats absorption and diffusion in polymers with this in mind. A common problem is that tests with larger molecular weight liquids have not reached equilibrium absorption within the timeframe of the exposure. HSP correlations are presented for chemical resistance studies of epoxy and zinc silicate tank coatings, PET, POM, PA6/66, PUR, PPS, PEI, Neoprene[®], etc.

INTRODUCTION

HSP are widely used in the coatings industry to select solvents for dissolving polymers and binders. This has been discussed in Chapter 8 and also in References 1 through 12, as well as elsewhere. The fact of solution is in itself clearly one simple form of chemical attack of the polymers they dissolve. This means that chemical resistance for some polymers can be partly inferred from HSP correlations of their solubility and/or swelling. HSP correlations of this type have been discussed in Chapter 5. An example is that if a chemical does not dissolve an epoxy component or the curing agent, then it is quite unlikely that it will attack a fully crosslinked epoxy coating or glue. The HSP correlations of surface phenomena, which have been discussed in Chapters 6 and 7 and elsewhere,13-18 can also provide some insight into chemical resistance. Liquids not wetting a surface are not as likely to attack it as those that do wet it, although there are no guarantees. A relation between spontaneous spreading and dewetting of liquids and environmental stress cracking has been found.¹⁹ This is discussed in more detail in Chapter 14. Liquids that spontaneously spread were found to induce environmental stress cracking at lower critical strains than those liquids that do not. Some surface studies may involve evaluation of a more direct form of chemical attack, such as the attack/whitening of PET coated with "amorphous" PET to improve weldability. This example is discussed in more detail later. Whatever is being correlated, the general considerations of the HSP characterizations discussed in earlier chapters are the same for the HSP correlations of chemical resistance reported here. However, there are certain additional pitfalls to be aware of when correlating chemical resistance. These include (lack of) attainment of equilibrium, the effects of molecular size of the test chemical, difference in local segments of polymers (even in homopolymers), and acid/base reactions.

Once a reliable HSP characterization of chemical resistance is available, it can be used to calculate the behavior of other systems that have not been tested. Obtaining a good HSP correlation of chemical resistance that allows reliable predictions depends very much on careful treatment of the available data or generation of data with such a correlation in mind. Unfortunately, very few

studies of chemical resistance have been designed with the purpose of generating HSP correlations. Also, it must be clear that the chemical attack discussed in this chapter does not include true chemical reactions leading to covalent bonding or destruction, such as with acids and bases, whether they are organic or inorganic. Chemical reactions forming new compounds are often found with amines and organic acids. These reactions often lead to discoloration in systematic solubility parameter testing with one or more of the amines used as test solvents. Discolored systems should simply be neglected in HSP correlations of physical (reversible) solubility. The products of reactions of well-defined organic bases with well-defined organic acids have actually been studied systematically from a solubility parameter point of view.²⁰ Some of the results of this study are discussed in Chapters 15 and 18.

CHEMICAL RESISTANCE — ACCEPTABLE-OR-NOT DATA

Additional sources of data for solubility parameter characterizations include chemical resistance tables reported by raw material suppliers²¹⁻²⁶ or collected in books²⁷⁻²⁹ and other sources such as those supplied on electronic media by the Plastics Design Library.³⁰ Although these data are certainly valuable in themselves, it has been found that given data sets are not always as reliable/consistent/coherent as could be desired for solubility parameter correlations. Attainment of equilibrium may not have been achieved, and this effect is rarely confirmed or even considered. Solvents with low diffusion coefficients will appear to be less aggressive than they might become at longer exposure times or at higher concentrations. As discussed earlier, true chemical attack with acids and bases must sometimes be sorted out. Likewise, the data are often limited in number and scope, and the chemical reagents have not been chosen for the purpose of solubility parameter correlations. Nevertheless, with the use of due caution, it is often possible to find excellent solubility parameter correlations using chemical resistance data of the acceptable-or-not type, particularly when the list of agents is long. Additional precautions with regard to data of the acceptable-or-not type include whether a molecular size effect is present as discussed in the following. Also, it can be assumed that if a chemical attacks a polymer at, say, 20°C, then it will also attack it at, say, 70°C. It can then be included as data in a 70°C correlation, even though it may not have been tested at that temperature (and in principle the HSP are only valid at room temperature).

EFFECTS OF SOLVENT MOLECULAR SIZE

It has been emphasized in Chapters 1, 2, and 5 that the size of solvent molecules is important for polymer solubility. HSP correlations have confirmed that this effect is even more important when chemical resistance is being considered. Smaller molecules are expected to be better, that is, more aggressive from a thermodynamic point of view than larger ones, all else being equal. This is known from the theories of polymer solubility discussed in Chapters 1 through 4, and also from the discussion of barrier polymers and diffusion found in Chapter 13 and 16, respectively. So it is not surprising that solvent molecular size can be an important fourth parameter in correlations of chemical resistance. An appropriate way to check this is sorting output data from a computer (or other) HSP optimization according to the molecular volume of the test solvents. What appear to be errors in the correlation may become systematically arranged. It can easily be seen, if the top of the list includes the type of "error" where the smaller molecular species are "better" than expected by comparison with all the other solvents. This may take the form of unexpectedly dissolving, being more aggressive than expected, penetrating more rapidly, or reducing mechanical properties more severely. Larger molecular species which are "poorer" than expected by comparison with the data for the other solvents are often seen at the bottom of the list. One can focus upon the molecular size range of greatest interest in such cases and repeat the correlation, neglecting those species which are outside of this range of molecular volumes. The correlation is then strictly valid only

for the size range specified. Some indication of the behavior of the solvents with V larger than the upper limit is possible if their RED numbers are greater than 1.0. These would not be expected to attack under any circumstances. Likewise, the solvents with V less than the lower limit can be expected to attack if their RED numbers are less than 1.0. Larger numbers of solvents are needed in the study if this is to be done with any benefit.

As stated above, the size and shape of solvent molecules are very important for kinetic phenomena such as diffusion, permeation, and attainment of equilibrium. Chapters 13 and 16, respectively, discuss correlations of HSP and elaborate on diffusion phenomena in more detail. However, it will be repeated here that smaller and more linear molecules diffuse more rapidly than larger and more bulky ones. The diffusion coefficient may be so low that equilibrium is not attained for hundreds of years at room temperature in common solvent exposures of rigid polymers like polyphenylene sulfide (PPS) with thicknesses of several millimeters.³¹ Such effects lead to comparisons where some systems may have reached an equilibrium uptake, whereas others have not. Likewise, the second stage in the two-stage drying process in polymer film formation by solvent evaporation can last for many years.^{4,31} See Chapter 16. Polymer samples used for solubility parameter or chemical resistance testing may contain retained solvent or monomer for many years, and this may also affect the evaluations. However, the effects of water can be extremely rapid as discussed in the following.³²

Attempts to include the molecular volume into a new composite solubility parameter and size parameter have not been particularly successful.^{33,34} This may be because the size effects are not necessarily caused through the thermodynamic considerations on which the solubility parameters are based, but rather through a kinetic effect of diffusion rate.

CHEMICAL RESISTANCE — EXAMPLES

Chemical resistance means different things in different contexts. Various examples of HSP correlations of chemical resistance are included in the following. The HSP data for the correlations discussed are included in Table 12.1.

Experimental data are always preferred over predicted behavior based on a correlation. However, a good HSP correlation can be used to find many chemicals that will clearly attack or that will clearly not attack. There are also situations where the attack is mild, and whether or not satisfactory results are found with a product depends on its use. Data in chemical resistance tables are often of the type +, +/-, -, or satisfactory/questionable/unsatisfactory, recommended/not recommended (R/NR), or something similar. The liquids which attack are clearly good solvents for the material in question and will be located within the HSP spheres with RED numbers being successively lower for more severe attack, all other things being equal. The correlations can include the solvents with mild attack (+/, questionable) either in the attacking (NR) group or in the nonattacking (R) group. They can also be neglected, not knowing which group to put them into. The treatment used in the individual correlations presented here is indicated in the following. Unless otherwise specified, the results are for room temperature.

TANK COATINGS

Chemical resistance is important for tank coatings used in the transport of bulk chemicals. The data in Table 12.1 include two older HSP correlations for chemical resistance for two types of tank coatings supplied by Hempel's Marine Paints. These are for a two-component epoxy type and for a zinc silicate type. The data and correlations are about 20 years old. They are included here for purposes of demonstration. Improvements in chemical resistance are known to have been implemented in a newer epoxy tank coating, but no HSP correlation has been made. A HSP correlation of the solubility of a lower molecular weight epoxy, Epon® 1001 (Shell Chemical Corp.), is included for comparison. The numbers are not too different from those of the HSP correlation for chemical

Transen solubility rarameter correlations for selected materials						
Material	δ	δ_{P}	$\boldsymbol{\delta}_{H}$	R _o	FIT	G/T
Epoxy tank coat (two component)	18.4	9.4	10.1	7.0	<u> </u>	<u> </u>
Epoxy solubility (Epon 1001)	18.1	11.4	9.0	9.1	a	<u>a</u>
Zinc silicate coating	23.5	17.5	16.8	15.6	<u> </u>	<u>a</u>
PET-amorphous coating	17.0	11.0	4.0	9.0	1.000	7/11
PET-CR (+/- R ^b)	18.2	6.4	6.6	5.0	0.865	7/34
PUR-CR $(+/-R^b)$	18.1	9.3	4.5	9.7	0.981	16/26
POMC/POMH (+/ Rb) (+/- R ^b)	17.1	3.1	10.7	5.2	0.955	2/28
POMC (+/- NR ^b)	17.9	5.9	8.3	6.6	0.609	11/28
PA6/PA66 (+/- NR ^b)	18.9	7.9	11.7	8.7	0.950	9/31
Halar 300 ECTFE 23°C	16.8	8.4	7.8	2.7	0.993	2/102
Halar 300 ECTFE 50°C	18.1	7.5	8.5	5.2	0.700	18/92
Halar 300 ECTFE 100°C	18.1	7.9	7.9	6.7	0.710	49/91
Halar 300 ECTFE 120/149°C	18.3	8.7	7.9	7.5	0.800	48/74
Neoprene-CR (+/- R ^b)	18.1	4.3	6.7	8.9	0.937	30/48
PPS tensile strength <60% 93°C	18.7	5.3	3.7	6.7	0.991	9/16
PEI ULTEM 1000 600 psi	17.3	5.3	4.7	3.3	1.000	3/20
PEI ULTEM 1000 1200 psi	17.0	6.0	4.0	4.0	1.000	4/20
PEI ULTEM 1000 2500 psi	17.4	4.6	9.0	7.2	0.967	9/20
PEI ULTEM 1000 solubility	19.6	7.6	9.0	6.0	0.952	8/45
PES mechanical properties	17.1	9.9	6.3	6.3	0.931	6/25
PES solubility	19.6	10.8	9.2	6.2	0.999	5/41

TABLE 12.1 Hansen Solubility Parameter Correlations for Selected Materials

Note: The symbols G for good solvents and T for total solvents are maintained, with the understanding that G solvents are within the HSP correlation spheres and are not recommended for use.

^a Data not available.

^b See text: NR — not resistant, R — resistant.

resistance for the two component epoxy tank coating. These three correlations have been reported earlier.⁸

The fact of a successful HSP correlation for a completely inorganic type of coating like zinc silicate is surprising. This is still another demonstration of the universality of the applications possible with the HSP concept. Although the data fit numbers were not recorded at the time, the two chemical resistance correlations reported here were clearly considered reliable.

PET FILM COATING

Another example of chemical resistance, or lack of the same, is the attack of the amorphous, modified PET coating on PET films to improve their weldability. This correlation is based on only 11 well-chosen data points but clearly shows that attack for many chemicals can be expected. Among those chemicals not attacking are hydrocarbons, glycols, and glycol ethers and higher alcohols which have a reasonably high hydrogen bonding character.

ACCEPTABLE OR NOT — PLASTICS

Several examples of HSP correlations of data reported in the form acceptable-or-not are included in Table 12.1. The data for these are all found in Reference 21. Other data sources for these are also available. HSP correlations of this type are included for PET, PUR, POMH, POMC, and PA6/PA66 using data from Reference 21. The first three correlations of this type consider reported evaluations of minor attack which will require further evaluation (+/–) as if these systems were suitable for use (resistant). The last two correlations consider this condition as not suitable for use (not resistant). This is to demonstrate that differences are found, depending on how the data are considered and that outliers are often found when correlating this type of data. It is for this reason that more extensive tables of HSP correlations of chemical resistance are not reported here. Too much space is required to try to explain why given results are outliers. However, some reasons have been given earlier and others are in the following.

The HSP values for PET based on chemical resistance are somewhat different from those of the amorphous PET coating, which is readily attacked by far more solvents. The compositions are also different. The POM correlations have typical problems in that the correlation considering all minor attack as negligible is based on only two severely attacking solvents among the 28 solvents tested. When the solvents demonstrating minor attack are considered as being in the attacking group, the data fit shows that there are many outliers. A systematic analysis of why this is found will not be attempted for reasons of space, even if all the outliers could be explained in one way or another.

The HSP found for a polymer in this type of correlation may not be representative of that polymer in all aspects of its behavior. There is a question as to coverage over the whole range of solvent HSP possible by the test solvents. Additional solvents may be required to make an improved correlation based on the improved coverage possible. There is also a question as to which segments of the polymer may be subject to attack by which solvents. Block copolymers may demonstrate two separate (overlapping) correlations that cannot be reasonably force fitted into a single HSP correlation. Viton[®] is an example of this. The most severe attack or swelling may occur in one region or another of the polymer or maybe even on a third component, such as a cross-linking segment. Viton[®] is discussed in Chapter 5 and Chapter 13 in more detail.

In spite of these pitfalls, it is strongly suggested that those generating this type of resistance data should try HSP correlations to evaluate the consistency of the data before reporting it. Outliers can be reconsidered; whether or not equilibrium has been attained can be inferred, and the probable effects of solvent molecular size may become apparent.

The effects of temperature on the chemical resistance of poly(ethylene co-chlorotrifluoroethylene) (ECTFE) Halar[®] 300 can be seen in Figure 12.1. The data on which these correlations are based are of the recommended-or-not type and were found in Reference 26. This figure has affectionately been dubbed a "bullseye," as there appears to be symmetry about a central point, although this is not strictly true as the HSP data confirm. The radius of the chemical resistance spheres increases with increasing temperature, as expected, as more solvents then become more severe in their attack. The HSP data for these correlations are also included in Table 12.1. The data fits are not particularly good at the higher temperatures.

To complete this section, a correlation of chemical resistance data for Neoprene[®] rubber (Du Pont)²² is included. Solvents in the intermediate category, i.e., that of a questionable-for-use recommendation, are considered as being in the nonattacking group for this correlation.

Previously it was indicated why HSP correlations of this type lead most often to guidance rather than to a firm recommendation. There are many pitfalls to be aware of both in generating such correlations as well as in using them, but their usefulness becomes clearer with some experience.

A suitable goal for a future project is to determine the effective HSP for various media frequently encountered in resistance lists, such as mustard, certain detergents and oils, etc. This could perhaps be done by composition in some cases. In other cases, one could see whether behavior paralleled that of a known chemical. A third method is to determine these parameters by recognizing a similarity to all materials attacked and a difference from those not attacked. This approach has been used to assign HSP to some liquids when calculations were uncertain. A computer program was developed similar to the SPHERE program as described in Chapter 1, but working in the

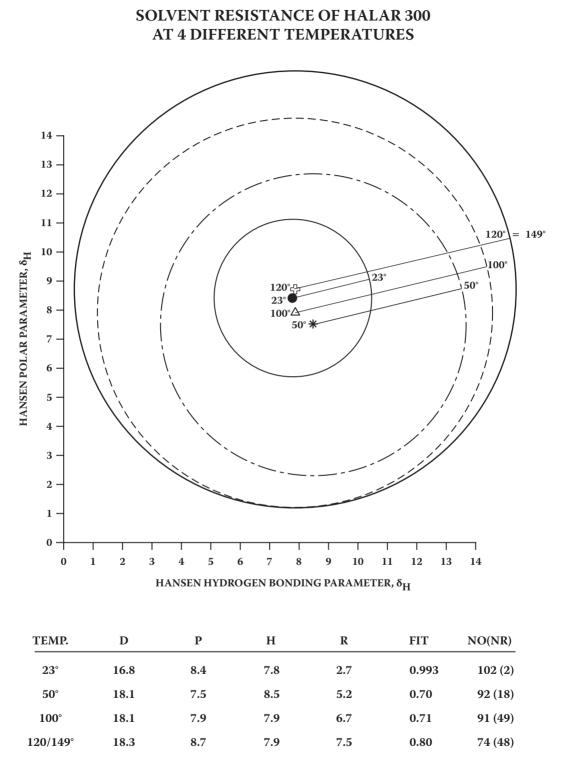


FIGURE 12.1 Chemical resistance of Halar[®] 300 ECTFE at various temperatures. Liquids within the spheres (circles) are not recommended at the given temperatures. HSP data given in Table 12.1.

opposite manner. The solubility of a number of polymers was evaluated in the solvent. The solvent parameters were then systematically varied by the program to reduce the collective error, that is, to locate the best possible set of HSP for the solvent. In general, the data fits for this procedure were comparable to those found for polymer solubility using the SPHERE program. In other words, not all the predictions based on the HSP thus assigned to the solvent agreed with the experimental data, but the errors were small.

TENSILE STRENGTH

The long-term exposure of polymers or polymer composites to solvents normally leads to changes in mechanical properties. One of the more direct techniques to measure such effects is to determine the tensile strength. The tensile strength reduction for glass fiber reinforced polyphenylene sulfide (PPS) after exposure to a number of solvents at 93°C for 12 months has been reported.²³ A HSP correlation of these data using the "good" solvents as those which reduce tensile strength under these conditions to less than 60% of the initial value is found in Table 12.1 and Figure 12.2. More extensive correlations for PPS are found in Reference 31.

Additional HSP tensile strength correlations have been generated for polyetherimide, ULTEM[®] 1000, using data reported by General Electric.²⁴ It is clear that the chemical resistance is dependent on the stress level. Higher stress levels lead to more severe attack by a larger number of chemicals. The solvents considered as being those which attack led to cracking during the study, which lasted 336 h. Some led to earlier cracking than others, which could be treated in a separate correlation, but this has not been done. A more rapid attack is expected from the better solvents with the smallest size and shape. The correlations all have high data fits.

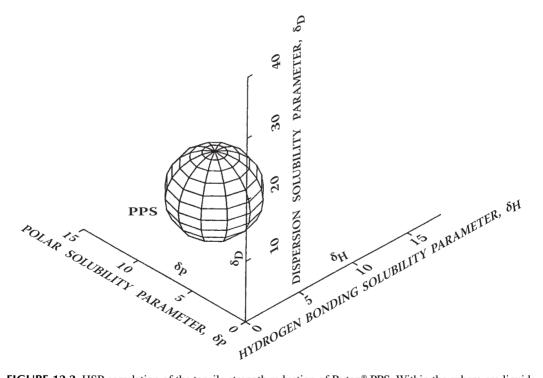


FIGURE 12.2 HSP correlation of the tensile strength reduction of Ryton[®] PPS. Within the sphere are liquids which reduce the tensile strength to less than 60% of the original value after exposure for 1 year at 93°C. (From FORCE Institute, Solvent Resistance of Polymer Composites, Glass Fibre Reinforced Polyether Sulphone (PES), 1st ed., Center for Polymer Composites, 1994, 31. With permission.)

The next entry in Table 12.1 is for true solubility of ULTEM 1000. These data were generated in a standard solubility parameter study. This correlation is not directly comparable with the previous ones for ULTEM 1000 as the number and range of solubility parameters included in the test solvents are different. The parameters for the polymer found in this correlation are those expected to reflect its (thermodynamic) affinities most closely. The previous study²⁴ did not include a sufficient number of solvents having widely different HSP to give a true total picture of the ULTEM 1000.

A final HSP correlation of the suitable-for-use or not type is presented in Table 12.1. This is for polyethersulfone (PES) based on mechanical properties after exposure to various liquids. The HSP correlation for the recommendation from the supplier²⁵ for glass fiber reinforced PES (Ultrason[®] E, BASF) can be compared with the HSP correlation for simple solubility of the polymer in standard test liquids, also given in Table 12.1. There is a difference, but it is not large.

SPECIAL EFFECTS WITH WATER

As stated elsewhere in this book (Chapter 1 and Chapter 15, in particular), the seemingly unpredictable behavior of water has often led to its being an outlier in HSP correlations. For this reason, it is suggested that data for water used as a test solvent not be included in HSP correlations. Water can be a very aggressive chemical. Water uptake in most polymers increases with increased temperature. This is because the solubility parameters of the water and polymer are closer at higher temperatures. The very high δ_H parameter for water decreases more rapidly with increasing temperature than the δ_H parameter for most polymers. This has been discussed in Chapter 1 and Chapter 8, but it is repeated here with examples for those interested in chemical resistance.

Water is an exceptionally good plasticizer because of its small molecular size. The presence of water not only softens (reduces the glass transition temperature) a polymer as such, but it also means diffusion rates of other species will be increased. Therefore, the presence of water in a film can influence the uptake of other materials, with hydrophilic materials, in particular, being more prone to enter the film.

The increase of water uptake with increased temperature can cause special problems with blistering if the temperature of a water-saturated polymer falls rapidly to a lower temperature. The previously soluble water can no longer be truly dissolved. Some of the water already in the polymer is now in excess and suddenly appears as small clusters or droplets of freed liquid water within the polymer itself (see Chapter 8, Figure 8.3). These droplets can quickly collect into blisters, especially if there are hydrophilic sites in the polymer or at an interface to which water will rapidly diffuse. This special type of failure has been discussed in more detail elsewhere³² (see also Chapter 1 and Chapter 8). The phase separated water has been called SWEAT (soluble water exuded at lowered temperatures). The author has observed this phenomenon as a mechanism of failure for epoxies, polyesters, alkyds, polyethersulfone (PES), polyphenylene sulfide (PPS), and even EPDM rubber. This mechanism can be confirmed experimentally by cycling samples continually exposed to water between two relevant temperatures using a quench from the higher one to the lower one. One follows weight gain by rapidly weighing samples that are surface dry. Typical results for the SWEAT phenomena for EPDM are seen in Figure 12.3 and for PPS in Figure 12.4. Control samples that are not cycled reach equilibrium and stay there, whereas the cycled samples suddenly begin to gain weight well beyond the equilibrium value. The extra weight is phase-separated water within the samples. This has been discussed in detail in Reference 35 for PPS and PES.

A related problem can be encountered in chemical resistant coatings for tanks that have been in contact with methanol. If a coated tank has been used to store methanol, and perhaps hot methanol in particular, the coating is more than likely saturated with methanol. It may take several days of exposure to fresh air (to reduce the amount of methanol to acceptable levels) before subsequent direct contact with water or seawater can be tolerated. If there is too much methanol retained in the coating, the water diffusing into the coating will associate with the methanol. The increasing

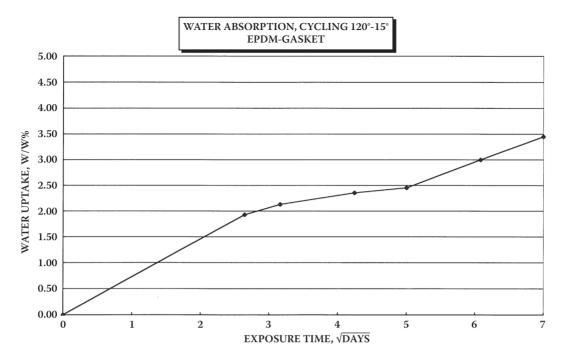


FIGURE 12.3 A rapid quench to a lower temperature can free water already dissolved in a polymer in the form of SWEAT. SWEAT can lead to blistering, cracking, and delamination. The data in the figure are weight gain for EPDM with cycling in water between 120 and 15°C. Water in excess of the equilibrium value at longer cycling times is SWEAT.

water content in the mixture of methanol and water will ultimately cause the solubility parameters of the mixture to be sufficiently high so that it becomes incompatible with the coating. Blisters form and total delamination can occur. These blisters are often near the substrate, as this is where the retained methanol will be found at highest concentrations.

CONCLUSION

HSP can correlate differences in physical behavior observed in chemical resistance testing of polymers and polymer containing systems when a sufficiently large number of different organic solvents are included in a study. HSP correlations including systematic consideration of the solvent molar volume (or other suitable size parameter[s]) should be an inherent part of all future studies of chemical resistance. These correlations aid in the determination of whether equilibrium has been attained, as well as provide insight into the behavior expected from untested solvents whose HSP are stored in a solvent database or can be calculated.

Examples include HSP correlations of true solubility and swelling, degree of surface attack, tensile strength reduction, and correlations for simple evaluations of chemical resistance of the suitable-or-not type. It is reemphasized that, in each case, the molecular size of the liquids evaluated will affect the result, and this should be considered in some way.

Data for true acidic or basic chemical attack must not be included in HSP correlations, as HSP correlations reflect physical attack and not chemical attack. It is strongly suggested that data for water not be included in these correlations as well. Its behavior is too unpredictable compared with other test liquids, and if it is included as an outlier, this fact will force a correlation with less predictive ability than had it been neglected.

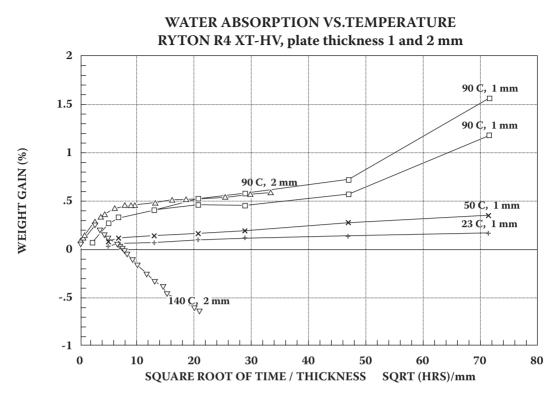


FIGURE 12.4 A rapid quench to a lower temperature can free water already dissolved in a polymer in the form of SWEAT. SWEAT can lead to blistering, cracking, and delamination. The data in the figure are weight gain for PPS with cycling in water between 90 and 23°C. Water in excess of the equilibrium value is SWEAT. (From Hansen, C.M., The Resistance of PPS, PES and PA Polymer Composites to Temperature Cycling During Water Exposure, Center for Polymer Composites (Denmark), Danish Technological Institute, Taastrup, 1994, 11. With permission.)

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