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RECRYSTALLIZATION AND TEXTURE

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1. PERSONAL INTRODUCTION

It is, I hope, appropriate in a paper reviewing recent advances in the field of recrystallization at a meeting celebrating the contributions and achievements of a distinguished scientist to start with a few personal comments. My first contact with Robert Cahn was in my final year of graduate research at Oxford when he visited the Department of Metallurgy, then headed by Professor William Hume-Rothery. Robert Cahn was not the first person outside the department with whom I was able to discuss my research, but he was able to offer to me what I subsequently saw him offer to many other scientists: (i) real enthusiasm for new ideas, (ii) innovative suggestions for new experiments and (iii) very strong encouragement to a young investigator. He followed this by providing real help in that he obtained for me, from the publishers of a major conference on recrystallization,⁽¹⁾ the prepublication set of the conference papers that I critically needed for my doctorate thesis.⁽²⁾ Three years later he encouraged me to apply for, and he subsequently appointed me to, a lectureship at Sussex University where he was establishing the first undergraduate and graduate programme in Materials Science in the U.K. The opportunity this allowed, to be in at the start of an exciting if short-lived experience of founding a new subject in a new university, was perhaps the most stimulating teaching experience in my life. Robert Cahn and I wrote, in 1983, an account⁽³⁾ of the brief history of Materials Science at Sussex University from when it was founded in 1965 to its closure in 1982/3. There is no need here to say more about that experience other than to quote the following from the introduction: "Creating and building up a group which achieved a worldwide reputation was deeply rewarding for all of us, in spite of the emotional stresses of the final period".

In the early years at Sussex while Robert Cahn was continuing his interest in recrystallization with two excellent research students, Dr Manu Bhatia⁽⁴⁾ and Dr Edmundo Chojnowski,⁽⁵⁾ and producing several scholarly reviews of the topic,⁽⁶⁻⁹⁾ I was following

other research interests—mainly in solidification mechanisms. Robert strongly encouraged me to return to recrystallization research and he suggested that I might study the details of nucleation processes by using Kossel X-ray diffraction. This was done using a stage that was built at Sussex, that used the focused electron beam in our electron probe microanalyser. This study led initially to our first joint research paper⁽¹⁰⁾ with Dr Gustav Ferran, a new metallographic technique,^(10,11) a joint review⁽¹²⁾ and subsequently to a major part of my research at Sussex University.^(10–16) This research in recrystallization was largely facilitated by Robert Cahn's encouragement and generosity. Once he had succeeded in getting me involved again in recrystallization research he stood back and gave me the space to grow. Not only did he leave me the area while he moved onto new fields, notably that of highly metastable microstructures, but even more generously he directed towards me two outstanding doctorate students, Serge Bellier⁽¹¹⁾ and Paul Faivre,⁽¹³⁾ and two outstanding Post Doctoral Fellows, Dr Yukio Inokuti^(14,15) and Dr Alain Kreisler.⁽¹⁶⁾ These were scientists who had contacted Robert Cahn asking to work with him in recrystallization. However, instead of encouraging them to work with him in his new area of rapid solidification, he encouraged them instead to work with me. The research carried out by the group of investigators that Robert enabled me to build up, produced a range of insights into the subject which laid the foundation of my career. Such help and encouragement goes way beyond what is normally considered the appropriate "mentoring" of young academics by senior scientists and is, in my opinion, the hallmark of a really fine scientist. I was not the only person that I saw being similarly helped by Robert Cahn during the 16 years that it was my good fortune to be a colleague of his at Sussex.

2. NUCLEATION OF RECRYSTALLIZATION

The vast majority of structural transformations in materials take place by the mechanism of "nucleation and growth". This is a heterogeneous transformation that, as first recognized by Gibbs, is complete in localized regions of the structure, with the transformed regions separated by an atomically sharp interface from the remaining untransformed regions. Recrystallization, at least on the scale of optical microscopy, is such a transformation with the recrystallization interfaces being high-angle grain boundaries. Spinodal decomposition, spinodal ordering and notably recovery are, of course, important examples of transformations occurring by the alternative homogeneous type of process noted by Gibbs. These homogeneous reactions are ones which occur throughout the material, i.e. they are delocalized, but with the transformation initially incomplete everywhere. For heterogeneous reactions there are the two stages, those of nucleation and growth. Both of the stages require growth, i.e. the movement of the interface by atomic transfer, but the first stage, nucleation, requires additional features that determine how and why the transformation starts at particular locations and not everywhere.

One of the major developments in structural metallurgy was the application to physical metallurgy of the thermal fluctuation model of nucleation by Turnbull.⁽¹⁷⁾ This theory had been previously developed for physical chemistry.^(18,19) In order to form a nucleus of critical size, the interfacial energy between new and old structure, γ , in units of J m^{-2} , must be offset by the reduction in volume free energy, ΔG_v , in J m^{-3} . For homogeneous nucleation, in which the nucleus can start to form on any of the N_v atomic sites in a unit volume,

a critical local increase of free energy, ΔG^* , must be supplied by thermal activation. The density of critical-sized nuclei, n^* , is then given by the standard equations:

$$n^* = N_v \exp(-\Delta G^*/kT) \quad (1)$$

and

$$\Delta G^* = 16\pi\gamma^3/3\Delta G_v^2 \quad (2)$$

The critical radius, r^* , or more accurately the critical *radius of curvature* of the migrating part of the interface, is given by:

$$r^* = 2\gamma/G_v \quad (3)$$

Eq. (1), suitably modified to take account of various complications, notably the reduction in size of the barrier by use of the energy of different defects in heterogeneous nucleation, has provided the basis of the qualitative, and in a few cases quantitative, understanding of almost all of the nucleation phenomena studied in materials science.⁽¹⁷⁻¹⁹⁾ However, it has long been recognized—see, for example, Refs 6–9 and 20—that eqs (1) and (2) are not relevant to the topic of nucleation of recrystallization in deformed metals. Eq. (3) is, however, a thermodynamic requirement: a radius of curvature less than that given by eq. (3) leads to the small new grain disappearing, but this loss leads only to a very small increase in the mean sub-grain size. There are two reasons for the failure of eq. (1) to predict the density of nucleation sites in recrystallization. The first of these is the very low value of the stored energy of deformation⁽²¹⁾ which is normally only a few $\text{J g}^{-1} \text{mol}^{-1}$ ($\Delta G_v \approx 1 \text{ MJ m}^{-3}$) and the second is the high interfacial energy of the high-angle grain boundary, γ_{gb} , that surrounds the relevant part of the “nucleated” new grain. These values substituted in ⁽¹⁾ predict an impossibly small density of new grains. The magnitude of $\Delta G^*/kT$ is so large, $\approx 10^{-8}$, that even the most effective heterogeneous nucleation sites cannot reduce the barrier to nucleation to any *significant extent*.⁽²⁰⁾ The solution to this problem was first described fully by Robert Cahn^(22, 23) and later and independently by Beck.⁽²⁴⁾ The suggestion was that a new grain does not develop by nucleation of a totally new crystal with a new orientation but instead a recrystallized grain develops from *a recovered region of the existing deformed microstructure*, a cell or a sub-grain. The new grain then has an orientation which will be essentially that of the deformed region from which it grew. In other words, there is no true “nucleation” in the sense of the formation of a structure that did not previously exist. In other nucleation events seen—e.g. during the solidification of a supercooled liquid such as water, or in matrix phase changes such as that from fcc γ -Fe to bcc α -Fe, or the precipitation of a solute-rich zone or solute-rich precipitate from initially homogeneous solid solutions such as that of zinc in aluminium—a very different process occurs. In these cases the *new* structure—a crystal of ice, a crystal of α -Fe, a zinc-rich region with the fcc structure, or a zinc-rich cph precipitate—*did not previously exist* and it had to be built up, it is assumed, by atom-by-atom growth from its initial site. It is in these circumstances that eq. (1) applies. However, in even the most heavily deformed metal, and in almost all metallic alloys, the basic crystal structure still exists even if containing a high density of defects.

A few, finely dispersed, two-phase alloys are now known that become amorphous during heavy deformation in “mechanical alloying”⁽²⁵⁾ but under these circumstances the structural changes on subsequent annealing are those of the *crystallization* of a glass and not the *recrystallization* of a plastically deformed polycrystalline material. The driving

force for crystallization of metallic glasses is much larger than the stored energy of plastically deformed crystalline metals.⁽²⁵⁾ Chemists, when they purify the product of a chemical synthesis by dissolution of the impure product in a solvent, usually at elevated temperatures, describe the formation of purified crystals from the supersaturated solution after cooling as “recrystallization”. However, most materials engineers would consider this type of crystal growth as a *precipitation* or a crystallization reaction. The former term appears most appropriate since the crystals being, in most cases, of a higher density do actually *fall* out of the solution. The rate of movement of most solid-state precipitates is rather slow at least under the influence only of earth’s gravity.

So for two reasons—(i) the size of the energy barrier to the formation of a new crystal, eq. (2), and (ii) the existence of a very high volume density of small regions of the structure with the lowest free energy (and the right chemistry) in the deformed material—nucleation in recrystallization of deformed metals occurs by an almost unique process: the rapid growth of a very small minority of the recovered cells which then become the new grains. The only other obvious example of this is the “nucleation” and growth of large grains in “abnormal” grain growth—a process sometimes called secondary recrystallization owing to its structural similarity to “primary” recrystallization of deformed metals.⁽⁶⁾ It has been often noted, see for example⁽²⁰⁾, that only a very small fraction of cells make the transition to a new grain. In a moderately cold-deformed sample of aluminium the sub-grains are typically about 1 μm in size while on recrystallization a grain size of at least 100 μm is quite common—giving a *volume* increase of about 10^6 . This means that only one sub-grain in a million makes the transition to become a rapidly growing nucleus capable of producing a recrystallized grain. For this reason it appears appropriate, in comparison with normal and abnormal grain growth,⁽⁶⁾ to describe nucleation as “abnormal” sub-grain growth.⁽²⁰⁾ However, since the growth of a very small minority of sub-grains to give recrystallized grains is the usual process of recrystallization, the use of the term abnormal sub-grain growth is perhaps rather misleading. Abnormal grain growth is, of course, much less common than normal grain growth.⁽⁶⁾ Normal grain growth occurs by the shrinkage of the 50% or so of the grains that are less than the average size.

The explanation of why only such a very small minority of sub-grains become recrystallized grains first became clear in a review by Cottrell⁽²⁶⁾ in 1953. Cottrell noted that the mobility of grain boundaries, their velocity under unit driving pressure, was very much lower for sub-grain boundaries with a low angle of misorientation than for high-angle grain boundaries. As a result of this mobility difference, only sub-grains that are *highly misoriented*, typically by more than about 15° with respect to at least part of their surroundings, can grow quickly and become recrystallizing grains. It is, in the opinion of this reviewer, appropriate that the mechanism of nucleation now known to dominate recrystallization of deformed metals is called the “Cahn–Cottrell” mechanism after the two scientists who provided the two critical insights into this process. All subsequent work in the field fully supports the ideas of the Cahn–Cottrell mechanism of nucleation.

The role of misorientation in controlling grain boundary mobility is clear from numerous qualitative studies that have shown that only high-angle grain boundaries migrate rapidly in recrystallization. Classic examples of this were provided by one of the students at Sussex.⁽¹¹⁾ His study found many new grains forming at the grain boundaries in a coarse-grained sample of pure aluminium compressed by 20%, see Fig. 1. The transmission X-ray pseudo-Kossel technique was used with Cu K_α X-rays generated by an

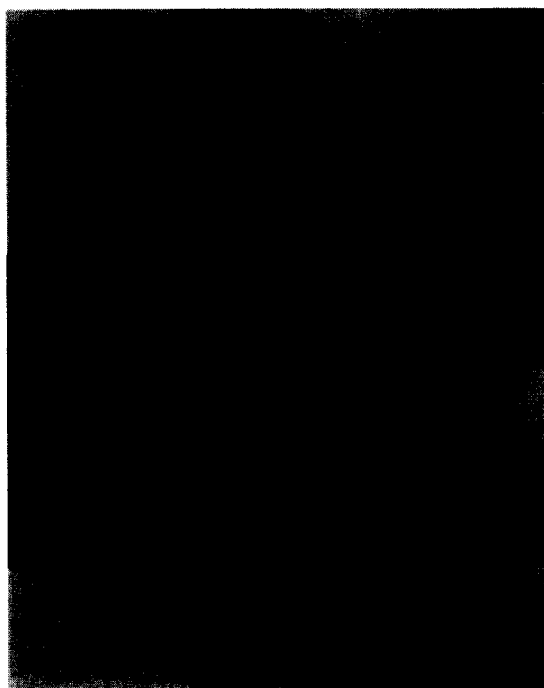


Fig. 1. Cluster of new grains seen at a prior grain boundary in high-purity aluminium compressed by 20% and annealed at 395°C for 11 min. The new grains 1, 2 and 3 came from within the orientation spread⁽⁴⁻⁹⁾ of the lower grain into which they did **not** grow. After Bellier and Doherty,⁽¹¹⁾ courtesy of *Acta Metallurgica*.

electron beam from a thin coating on the deformed and partially recrystallized samples of aluminium that had been thinned to a thickness of about 30 μm . By this means it was shown that the new grains had orientations within the 5–10° spread of *one* of the deformed grains at the boundary. The new crystals then grew only into the opposite grain—the one with which they had high-angle grain boundaries. Growth into the “parent” grain, with which the nucleus had only low-angle misorientations, did not occur. At some regions of the 20% deformed sample misorientations of 10–15° were found—these were incipient *transition bands* between regions deforming by different combinations of slip systems.^(11, 27-29) At these intermediate misorientations, new so-called “island” grains started to develop but they grew only slowly and were quickly overtaken and consumed by larger, grain-boundary nucleated, rapidly growing new grains with high misorientations.⁽¹¹⁾

Despite the overwhelming qualitative evidence, from nucleation studies, of the mobility advantage of high-angle compared with low-angle grain boundaries,⁽²⁹⁾ direct experimental measurements of these mobility differences are very rare. The experiment usually quoted to demonstrate the effect was the study by Viswanathan and Bauer⁽³⁰⁾ on very high purity copper bicrystals, Fig. 2. They found that low-angle boundaries, with misorientation of less than about 10°, migrated a thousand times more slowly than high-angle grain boundaries, misoriented by more than 18°, at temperatures close to the melting point. The low-angle grain boundaries migrated with activation energies of the order of bulk diffusion owing to, it is believed, the need for vacancy transmission through the perfect crystal to, from and between the different edge dislocations in the low-angle boundaries. High-angle

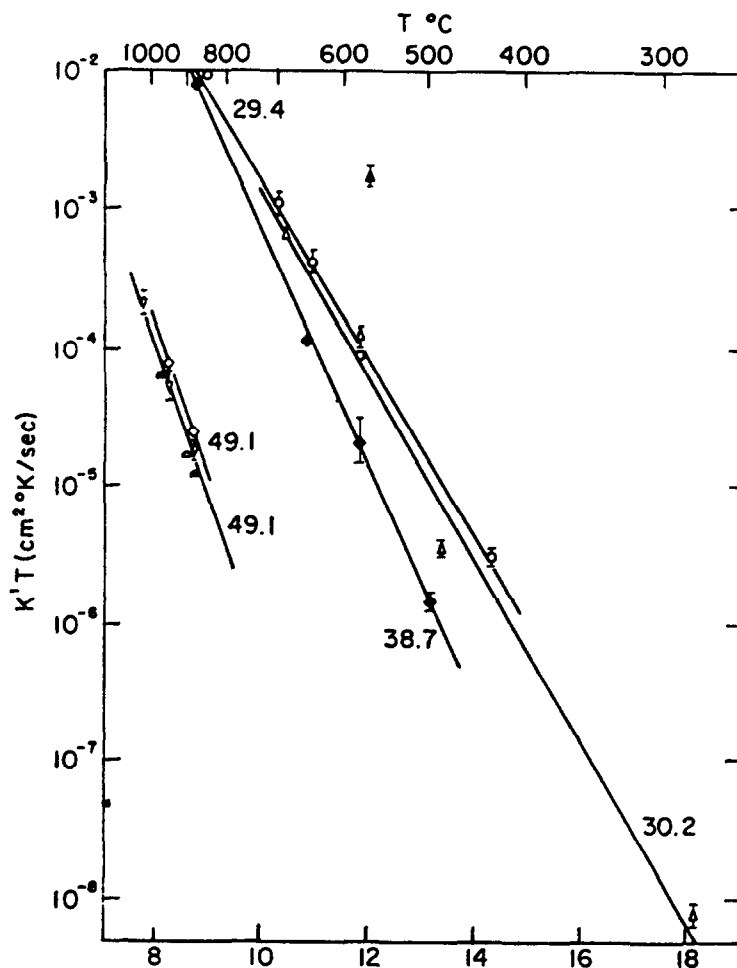


Fig. 2. Grain boundary mobility, K' , as a function of temperature, T , for low-angle boundaries of 2° (Δ), 5° (∇), 9° (\diamond), 18° (\circ) and 32° (\triangle). The apparent activation energies in kcal mol^{-1} are given. After Viswanathan and Bauer,⁽³⁰⁾ courtesy of *Acta Metallurgica*.

grain boundaries, however, migrate with low energies of boundary diffusion expected for very pure material with little solute drag effects.⁽³¹⁾ As a result of the different activation energies, extrapolation to the low temperatures where recrystallization of deformed pure copper occurs, $150\text{--}200^\circ\text{C}$,⁽³²⁾ shows that the mobility differences would then approach 10^6 . This result is fully satisfactory for very high purity materials but we have no *direct* evidence for the magnitudes of the mobility differences we might expect in lower purity metals, such as commercial aluminium. Ultra-pure aluminium can recrystallize close to room temperature⁽³³⁾ while additions of trace amounts of solute, particularly iron, raise the recrystallization temperature by 100 to 200°C . Haessner and Schmidt⁽³⁴⁾ showed that aluminium of 99.99 to 99.9999% purity, heavily deformed at low temperatures, recrystallized below room temperature—all their aluminium samples had, however, very low iron contents. Careful experiments on the orientation mobilities of low-, medium- and

high-angle boundaries in the solute drag region of 100 ppm impurity or so are critically needed. It is the recrystallization of such metals that is most important commercially. Almost all aluminium alloys used have about this level of iron in solution, and usually much more as-cast constituents, such as Al_6Fe and $\text{Al}_{12}\text{Fe}_3\text{Si}$.⁽³⁵⁾ Aluminium, unlike copper, is almost always used at a "commercial-purity" grade containing small amounts, 0.1–0.4 wt%, of iron and silicon.⁽³⁵⁾

Very recently, Ferry and Humphreys⁽³⁶⁾ reported a new method which shows promise for measuring the mobility differences of low- to medium-angle sub-grain boundaries in aluminium. They deformed Goss-oriented, $\{011\}\langle 100 \rangle$, single crystals of aluminium, with 500 ppm of Si and 3 ppm of other impurities, by channel die compression to strains of 0.5 to 3. They reported that there were no high-angle misorientations produced in this stable orientation, at least within the bulk of the samples, and, as a result, normal recrystallization did not occur provided the damaged surface regions were removed by electropolishing before annealing. In a few cases, highly misoriented regions from the surface did rapidly recrystallize the samples. The usual result of annealing at 250–350°C of the deformed Goss-oriented crystals was a combination of slow general sub-grain growth of most sub-grains, with typical misorientations of 2–3°, together with the development of small number of significantly larger or "abnormal" sub-grains which had larger misorientations, 6–10°. The more highly misoriented boundaries were shown to have higher mobility, by about 14 times, compared with the lower-angle boundaries.⁽³⁶⁾

The studies at Sussex^(10–16) on high-purity aluminium and iron deformed by 20–50% at room temperature and studied by X-ray pseudo-Kossel and by Kikuchi electron diffraction, together with many other studies as reviewed in 1978,⁽²⁹⁾ confirmed the second essential feature of the Cahn–Cottrell mechanism of nucleation. This is that the orientation of the new grains after nucleation can be traced back to *pre-existing orientations in the deformed microstructure*. This observation requires careful characterization of the deformed microstructures and must take account of the probability that a new grain may have grown from part of the sample that is not immediately visible.^(11, 29) In optical microscopy studies with nuclei larger than 10 μm there is a finite probability that the relevant part of the deformed structure will be invisible.⁽¹¹⁾ However, for transmission electron microscopy (TEM) studies with nuclei detectable when only 1–2 μm in size, the relevant deformed orientation is usually much easier to find.^(13, 16, 37) This is still true for samples that show multiple twinning during annealing as is commonly found in moderately deformed samples annealed as thin foils in the TEM.⁽³⁷⁾ All of these quoted studies confirm that small regions of the deformed material do develop into new grains, carrying the starting orientation with them, although twinning can occur⁽³⁷⁾ at least in special circumstances, such as with a very low stacking fault energy or on annealing even aluminium as thin foils. In most studies of recrystallization by annealing bulk materials of medium to high stacking fault energy, this twinning effect is rarely significant at the nucleation stage, when the critical cells are first starting to grow.

In earlier reviews of nucleation^(11, 20, 29) much of the work reviewed came from material studied at rather low levels of strain, 20–60% reduction, usually of high-purity metals in which the density of regions in the lattice with high misorientation was very low and the density of active nucleation sites even lower. At low reductions (less than 20%) in polycrystalline metals, the only high misorientations are found occur at prior grain boundaries. Even here, only very few of the sub-grains at the grain boundaries give rise to active nucleation events.^(11, 13–15, 37)

In other investigations, such as the classic study of Langford and Cohen⁽³⁸⁾ in heavily drawn steel wires, of Ray et al.⁽³⁹⁾ in heavily rolled copper and of Humphreys⁽⁴⁰⁾ in heavily rolled aluminium containing particles larger than 1 μm , the density of high local misorientations was shown to increase greatly with strain and, with the heterogeneities, “deformation zones” formed at the coarse second-phase particles.⁽⁴⁰⁾ Recently, there has been a great deal of detailed microstructural characterization of heavily deformed metals—see, for example,^(41–50)—by means of TEM, scanning electron microscopy (SEM) with special crystallographic etching⁽⁴⁶⁾ and by the use of backscattered Kikuchi diffraction (BKD).^(51–53) In all these studies it was reported that after heavy rolling reductions (80% and more) there was a very high density, particularly in the normal direction, of high-angle misorientations. After a reduction of thickness R , giving a strain ϵ of $\ln(1 - R)$, in a material with an equiaxed grain size of d_0 , the grain boundary area per unit volume—which was originally approximately $3/d_0$ —increases to $\exp(\epsilon)/d_0$. The spacing of the original high-angle grain boundaries, in the direction normal to the rolling plane (ND), falls to $d_0/\exp(\epsilon)$. However, it is often found, especially when d_0 is more than about 50 μm , that the grains are fragmented into several, $1 > n > 20$, regions by $(n - 1)$ transition bands^(11, 27, 28) so increasing by n the density of high-angle misorientations. The extent of this “transition” banding increased with grain size in cold-rolled copper.^(44, 47) In warm plane-strain deformed aluminium the value of n , with $d_0 = 140 \mu\text{m}$, increased from 3 for plane-strain extrusion to 7 for plane-strain compression in a channel die.⁽⁵⁰⁾ In these cases, the spacing of high-angle misorientations in the normal direction fell to only few μm after reductions of $R \geq 0.95$. Apart from the additional high misorientations found at second-phase particles with radii greater than about 1 μm in cold rolling,⁽⁴⁰⁾ high misorientations are also produced by the shear bands commonly seen with (i) deformation twinning in α -brass⁽⁵⁴⁾ and (ii) with shearable second-phase precipitates^(55, 56) or with high solute in aluminum alloys.^(57–59) So for heavily cold-rolled alloys, particularly those containing second-phase particles, there is a very high density of high misorientations, all of which could act as potential sites for nucleation of recrystallization. It should be noted that, for warm deformation, the misorientations introduced by particles⁽⁶⁰⁾ or by shear banding⁽⁶¹⁾ seem to vanish. Morii and Nakayama,⁽⁶²⁾ for example, showed that although pure aluminium single crystals, $\{211\}\langle 111 \rangle$, did not produce shear bands when rolled at room temperature they did so when rolled at only 77 K. It can then be expected that after warm rolling ($T \geq 0.6T_m$) only the misorientations at prior grain boundaries and at in-grain transition bands will remain as potential nucleation sites.⁽⁶¹⁾ However, recrystallization in hot- or warm-rolled alloys usually only takes place after very heavy strain, so there still remain very high densities of potential nucleation sites.

In all cases the possession of a high local misorientation is only a *necessary* condition for a potential nucleation site to become active—it is clearly not a *sufficient* condition since the vast majority of sub-grains at high-angle grain boundaries, and other high misorientation sites, do not become new grains. It has long been recognized^(12, 13, 63–65) that in addition to the mobility requirement for a sub-grain to grow, a successful sub-grain needs to have an *energy advantage* so that it grows rather than vanishing. The usual form of this energy advantage is having a significantly larger sub-grain size.^(12, 13, 41, 50) The size advantage may arise from the deformation process if a particular orientation on one side of a high-angle boundary has a larger sub-grain size and thus a lower stored energy. An example of this has been found in rolled iron in which grains with $\{111\}$ parallel to the rolling plane have been shown to have higher stored energy than other orientations.^(66, 67)

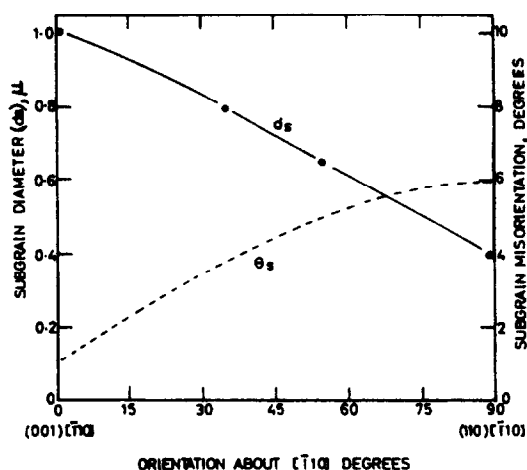


Fig. 3. Orientation dependence of stored energy as (i) mean sub-grain diameter, d_s , in μm and (ii) mean sub-grain misorientation, θ_s , in degrees in cold-rolled steel. After Dillamore *et al.*,⁽⁶⁶⁾ courtesy of *Metal Science Journal*.

Dillamore *et al.*⁽⁶⁶⁾ showed this effect by measurement of the mean sub-grain size, d_s , which was smallest in grains having $\{111\}$ parallel to the rolling plane, and by measurement of the mean sub-grain misorientation, θ_s , which was largest in grains with that orientation, Fig. 3. In pure iron cold-rolled only by 50%, Inokuti and Doherty⁽¹⁴⁾ found that nucleation occurred by invasion of $\{111\}$ grains by neighbouring grains with a larger sub-grain sizes. This is the process of "strain-induced boundary migration" (SIBM) originally identified by Beck and Sperry in 1950.⁽⁶⁴⁾ In compressed aluminium^(12, 13) and in rolled aluminium⁽³⁸⁾ cold deformed by only 40–50%, there were no significant sub-grain size differences seen in the deformed structure. However, the required size differences for SIBM nucleation appeared by the process of sub-grain coalescence in one of the grains, that can be considered the "parent" grain. The enlarged sub-grain then grew into the adjacent grain by migration of the existing high-angle grain boundary between the deformed grains. Detailed analysis of this sub-grain coalescence at grain boundaries was identified by Faivre and Doherty⁽¹³⁾ as requiring the presence, in the parent grain, of an *additional* high misorientation at a transition band in order that coalescence could take place.

At the time of the study⁽¹³⁾ the need for this other misorientation was not understood nor were the kinetics of the process, which were reported⁽¹³⁾ to be *too fast* to occur by the process modelled by Li.⁽⁶⁵⁾ A later analysis of sub-grain coalescence by Spzunar and Doherty⁽⁶⁸⁾ did, however, provide an explanation of these features. The revised model of sub-grain coalescence is shown in Fig. 4 using an idea originally proposed to the authors by the late Professor Verbraak. Verbraak had pointed out to the present author that the explanation offered in⁽¹³⁾, of coalescence only by screw dislocation glide, could not be valid! The dislocations in a sub-grain boundary cannot exist as isolated segments—the dislocations must continue into other boundaries and the movements of all parts of the dislocations must be connected. Figure 4, from the *subsequent* analysis,⁽⁶⁸⁾ shows a highly simplified model of a cube-shaped sub-grain with its misorientation axis shown normal to the twist portions of the interface. The dislocations are assumed to be one of two sets of loops. *Two results* then immediately follow from this model.⁽⁶⁸⁾ The first result concerns

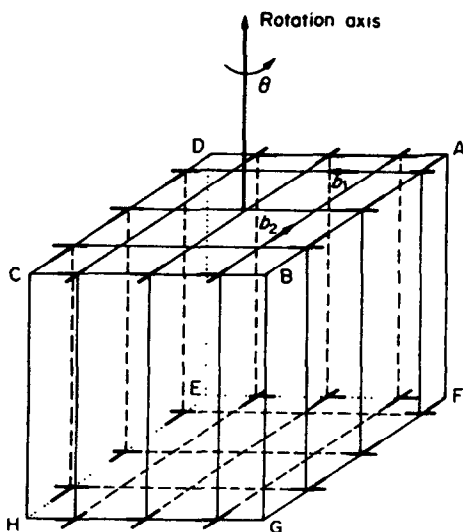


Fig. 4. Schematic idealized sub-grain, misoriented by a small angle, θ , normal to one of the interfaces, the twist boundary, ABCD, made up of a cross grid of screw dislocations of burgers vectors b_1 and b_2 . These continue as edge dislocations in the tilt boundaries, ABGF and BCHG, making a set of loops assumed to belong just to the one sub-grain. After Doherty and Szpunar,⁽⁶⁸⁾ courtesy of *Acta Metallurgica*.

the kinetics. If one set of loops is to be removed by absorption at a high-angle interface, where the energy per dislocation is less,^(11, 59) then the point defects needed for climb of one of the edge dislocations will be provided by the climb of the opposite dislocation (positive and negative edge dislocations in the same dislocation loop). At high temperatures, where bulk diffusion operates, then atom transport across the sub-grain is possible, giving kinetics⁽⁶⁸⁾ very similar to those suggested by Li.⁽⁶⁵⁾ At lower temperatures, such as those used by Faivre and Doherty⁽¹³⁾ or lower,⁽⁶⁸⁾ much faster climb is possible by the alternative transport path of *pipe diffusion* along the interconnecting screw dislocations. The second result concerns the need to operate on both sets of dislocations together. The process of removal of one set of dislocation by climb will, however, be quickly inhibited by the *elastic stress fields* of the twist boundaries. This arises since a twist boundary is only a low-energy configuration if there is an equal spacing, h , for each of the sets of screw dislocations. The equilibrium spacing is given by the scalar value of the burgers vector, b , and the sub-boundary misorientation, θ_s :

$$h = b/\theta_s \quad (4)$$

The need to maintain an equal spacing of the two sets of dislocation means that coalescence can only occur for sub-grains that have *two dislocation sinks* at higher than average misorientated boundaries. This requirement immediately accounts for the observation that the coalescence at a high-angle grain boundary only occurs if a further high-angle misorientation is present—the transition band in the parent grain seen by Faivre and Doherty⁽¹³⁾ for all the coalescence events they studied.

It is, of course, not true that such coalescence reactions are required for all nucleation events. The grain boundary coalescence model^(11–13, 63) merely provides one means by which a sub-grain size heterogeneity can be built up during annealing. This local sub-grain growth

will only be needed if suitable sub-grain heterogeneities are not present in the deformed microstructure. An important example of stored energy heterogeneities in rolled fcc metals, initially identified by Ridha and Hutchinson,⁽⁴¹⁾ will be discussed in more detail in the next section. This is the much lower stored energy found in cube-oriented regions after heavy rolling.

3. RECRYSTALLIZATION TEXTURE

It is one of the most striking features of recrystallization that the texture produced by heavy deformation can be completely modified by recrystallization.⁽⁶⁹⁻⁷¹⁾ There are at least three different types of recrystallization texture known to be produced by recrystallization: at one extreme the deformation texture components can be essentially retained, in other cases the texture can be strongly randomized and, finally, a component that is only a negligible fraction of the deformation can become an overwhelmingly dominant feature of the recrystallization texture. Figure 5⁽⁷⁰⁾ illustrates two of these features—heavily cold-rolled commercial-purity aluminium, containing a high density of iron-rich constituents, can give a heavy retained deformation texture while high-purity aluminium can give rise to a very strong “new” orientation, that of “cube” $\{001\}\langle 100 \rangle$. By use of the orientation distribution function technique, ODF (see ⁽⁶⁹⁾), the increase of random orientations in recrystallization can be easily shown.

There is a very major difference between the plastic anisotropy of an fcc metal with a strong deformation texture, both as-rolled and as a retained deformation texture after annealing, and that of the same metal with a strong “cube” texture. In the former case the deformation texture—usually described as a “tube” of components running from “brass” $\{011\}\langle 211 \rangle$, through “S” $\{123\}\langle 634 \rangle$ to “copper” $\{112\}\langle 111 \rangle$ —gives a lower yield stress for tensile strain applied at an angle of 45° to the prior rolling direction in a rolled sheet sample. If the rolled sample has a strong cube texture then the lowest yield stress is found in the directions that are 0° and 90° to the rolling direction. In the former case, drawn cups show strong “earring” in the 45° directions while the sheet samples with the cube texture give rise to 0 and 90° “ears”. Extreme plastic anisotropy is usually undesirable since for design purposes the lowest strength must be used. Also, in drawn cups the ears give wasted material, uneven thinning and the likelihood, at very high production rates such as those in making aluminium drink cans, that highly eared cans will jam the production line. For aluminium processing, two very different conditions apply. For sheet for many applications, for example in aerospace and in automobiles, the material is designed to have minimum anisotropy after recrystallization—so here a random texture is desired but difficult to produce. What is usually produced in a successful process is a “balanced” recrystallization texture with a mixture of the two opposite anisotropies: retained rolling and cube. However, for the dominant aluminum packaging product of the last 20 years, the drink can, a different recrystallization texture is needed—one with a strong cube component. In a major technological development, dating from about 20 years ago, the modern very thin walled can was produced from an Al-1%Mg alloy (AA3004) that has been strengthened *by heavy cold rolling before being drawn*. The trick to this process is a two-stage process in which the sheet is first directly deep drawn to a very shallow walled can; this is then redrawn with wall ironing to give a can with a thick base where more strength is required to abut with thinner walls (see ⁽⁶⁹⁾). To produce an low earring product in a heavily rolled sheet requires that the dominant rolling texture be

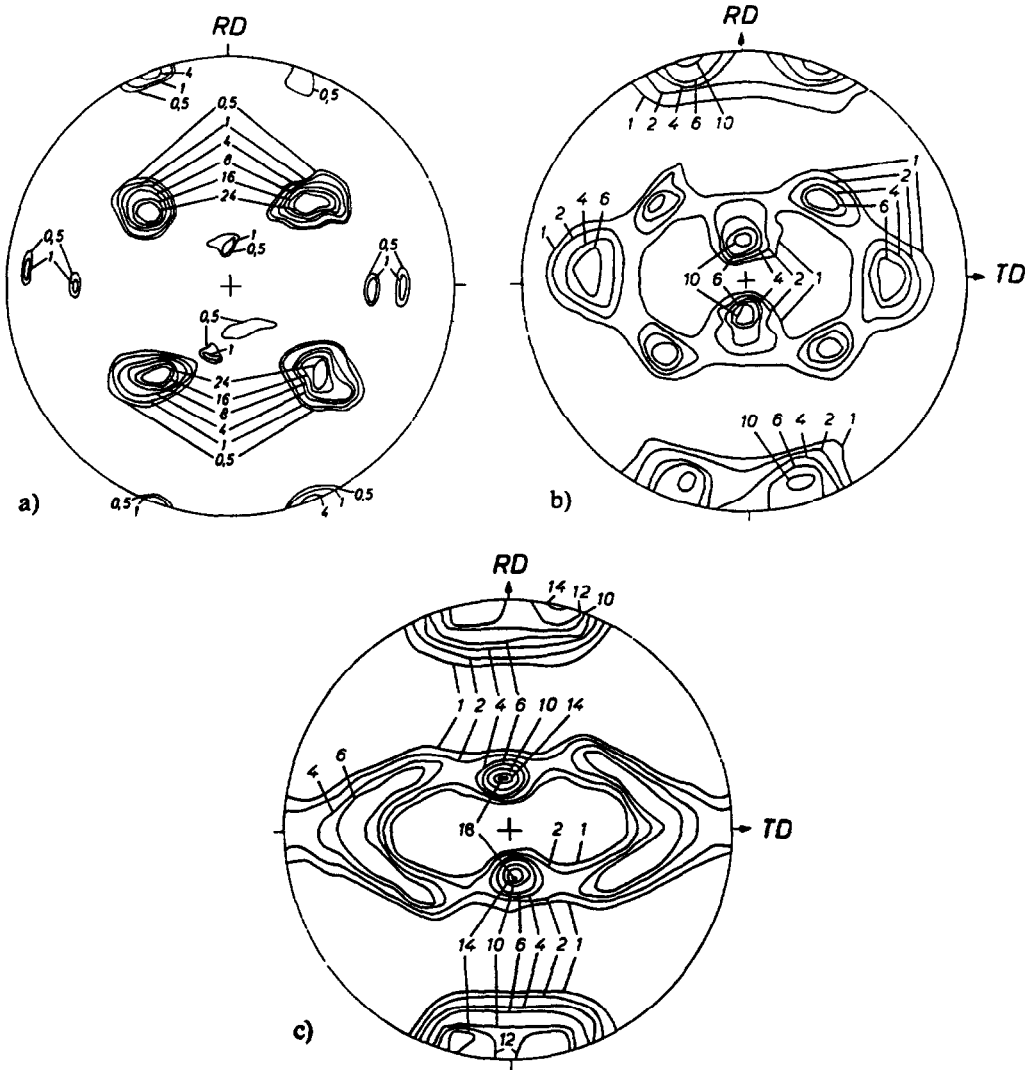


Fig. 5. $\{111\}$ Pole figures of heavily deformed and recrystallized aluminum alloys: (a) 99.99% Al, (b) Al-0.15% Fe-0.34% Si and (c) Al-0.54% Fe. After Grewen and Huber,⁽⁷⁰⁾ courtesy of Dr. Riederer-Verlag.

balanced by a very strong recrystallized cube component, Fig. 6.⁽⁷²⁾ Given the industrial significance of this problem, the topic of the origin of cube texture in rolled fcc metals has been given a great deal of study recently and the major results of this will now be briefly outlined and related to the ideas of nucleation developed above.

4. ORIENTED NUCLEATION OR ORIENTED GROWTH?

As considered in a panel discussion in 1988,⁽⁷⁰⁾ there has been a lengthy dispute over the origin of strong recrystallization textures. Two alternative models have been proposed. One

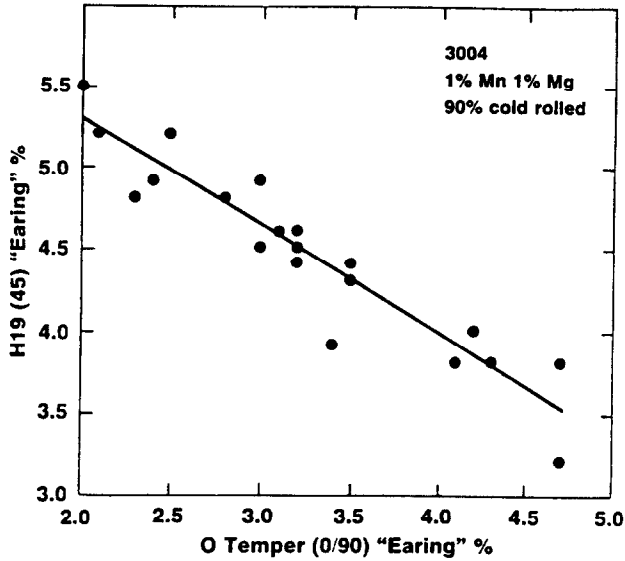


Fig. 6. Decrease of 45° earing in heavily rolled aluminium sheet by high cube texture (90°) earing after recrystallization of warm-rolled aluminium alloy. After Doherty *et al.*,⁽⁷²⁾ courtesy of the Institute of Materials

model is oriented nucleation, in which the orientations that dominate the final texture do so by nucleating new grains more frequently than other orientations. The other model, oriented growth, is that the dominant orientation is achieved by grains of the required orientation growing faster or at least bigger. These ideas are readily expressed mathematically.⁽⁷³⁾

Oriented growth requires that the frequency of grain nucleation of the special orientation such as cube, α_c , exceeds the frequency expected for random nucleation, α_r .

$$\alpha = \alpha_c / \alpha_r \gg 1 \quad (5)$$

The value of α_r depends on the allowed angular deviation from the exact orientation. For a deviation of 10° $\alpha_r = 0.006$, for 15° $\alpha_r = 0.025$ and for 20° $\alpha_r = 0.047$.⁽⁷⁴⁾ For oriented growth, it is required that grains with the special orientation grow *faster* than grains of other orientation to give grains with larger mean grain size, d_c , than the mean grain size of randomly oriented grains, d_r :

$$\beta = d_c / d_r \gg 1 \quad (6)$$

The advantage of this formulation is twofold. First, it leads to an immediate way of determining the relative importance of the two mechanisms which are perhaps best described as either a frequency advantage or a size advantage. These measurements of α and β are now readily carried out using the BKD technique^(43, 45, 48, 50, 75) or other local orientation techniques^(46, 76) in fully or partially recrystallized structures. The other advantage of the formulation of the oriented nucleation/oriented growth argument in the form of the α and β parameters is that it decouples the measurements of the effects from any assumed models for oriented nucleation or growth—such as the enhanced mobility of particular grain boundaries, e.g. those with a 40° misorientation about $\langle 111 \rangle$ for

oriented growth,⁽⁷¹⁾ or particular models e.g. that of Dillamore–Kato for enhanced cube nucleation.⁽⁷⁷⁾ Measurement of enhanced frequency, $\alpha \gg 1$, or larger grain size, $\beta \gg 1$, establishes oriented nucleation or growth independently of the mechanism causing the effect, which needs to be determined independently. The effect and the mechanism can be different in different cases given the complexity of the deformation of single and polycrystals.^(61, 69, 71)

For the formation of cube texture in rolled fcc metals, two publications^(69, 71) concluded that for recrystallization in many deformed single crystals there was strong evidence for oriented growth and that it was unlikely that “nuclei of random orientations ever occur”⁽⁶⁹⁾—that is, in non-random recrystallization textures, α is always > 1 . It appears to remain a matter of dispute as to what the relative importance is of α and β in general, particularly in polycrystalline material. However, recent studies of very strong cube recrystallization texture in heavily cold-rolled high-purity copper,^(32, 44, 46) heavily cold-rolled high-purity aluminium⁽⁴³⁾ and heavily *warm* plane-strain deformed commercial-purity aluminium^(42, 45, 48–50, 61, 78–80) have all showed for these polycrystalline materials a dominance of oriented nucleation with $\alpha \gg 1$ and $\beta \approx 1$. These recent studies, and older studies⁽⁶⁹⁾ for copper, have shown that the final recrystallized cube texture is greatly strengthened by:

1. increased prior strain;
2. decreased prior grain size;
3. for commercial-purity aluminum alloys, a higher deformation temperature; and
4. a higher starting cube texture.

These observations, following recent investigations, particularly Refs 47, 49, 50 and 61, have become much clearer. Simple models^(47, 50, 61) now appear to be able to give, for the first time, successful quantitative predictions of the resulting cube intensity based solely on oriented nucleation. The major recent discovery reported by many of the investigators is that, in contrast with the prior models of rolling deformation (e.g. ^(72, 77)), at least during warm rolling of aluminium and in the cold rolling of copper, the cube orientation is rather stable and at higher strains there is a tendency to rotate towards the exact cube, Fig. 7. As a result of this, the spacing, λ_c , between adjacent deformed cube bands falls. In one study⁽⁵⁰⁾ it was found that this spacing could be successfully predicted on the basis that each initial cube grain gave rise to a deformed cube band, Fig. 8, and as the strain increased this spacing (measured in the normal direction) approached the size of the recrystallized grain thickness, d_R , measured in the same direction. The form of the successful prediction was:

$$\lambda_c = d_c(0)(1 - R) = d_c(0)/\exp(\epsilon) \quad (7)$$

Here $d_c(0)$ is the initial spacing between cube grains before deformation.

Other studies,^(49, 61) while giving similar trends, find a less perfect agreement with this simple model. The full cube stability found in Fig. 8 appears to depend on the starting grain size and the deformation temperature. The recent studies^(48–50, 61) also support a result reported earlier for heavily cold-rolled copper⁽⁴¹⁾—that the deformed cube bands have significantly lower stored energies, larger sub-grain sizes and smaller sub-boundary misorientations than in other orientations. It seems likely that this arises at least in part owing to easier recovery in cube-oriented regions; according to ⁽⁴¹⁾ from the fact that the four main slip systems in this orientation all have orthogonal burgers vectors which have

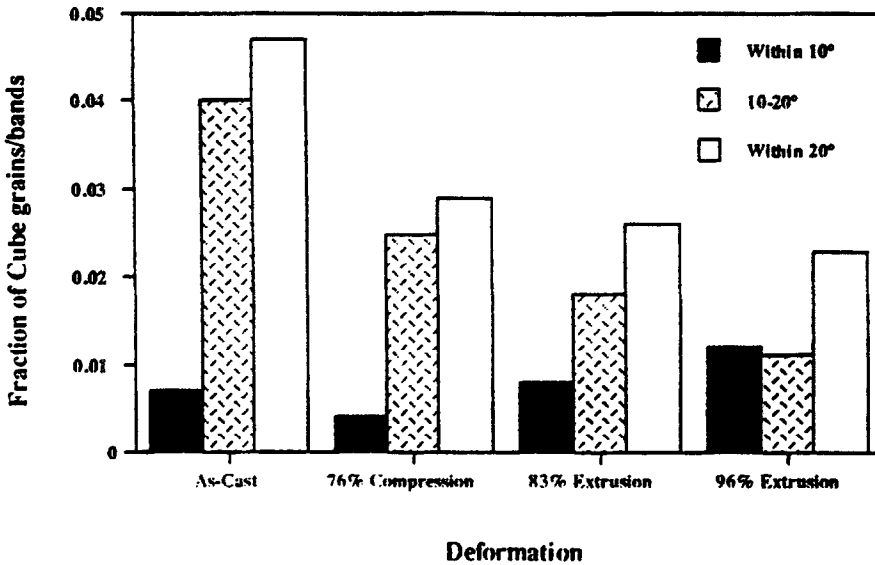


Fig. 7. Aluminium plane-strain deformed at 320°C at a strain rate of 0.05 s⁻¹ to three reductions, showing the initial decline of near-cube-oriented regions but a rotation towards cube at the highest strains. Frequency of orientations within 10° to 20° of exact cube measured by BKD measurements. After Samajdar and Doherty.⁽⁵⁰⁾

minimum elastic interactions. Irrespective of its origin, the combination of thin deformed cube bands, only a few sub-grains in thickness, containing large cube-oriented sub-grains abutting with high-angle boundaries onto other orientations with higher stored energies causes many cube nuclei to develop from these bands and grow in all directions, but the growth of particular importance is in the normal direction giving resulting grains of a thickness d_R , see Fig. 8.

Duggan and Chung⁽⁴⁷⁾ proposed a simple model for the frequency of cube grains:

$$\alpha_c = 2d_R/\lambda_c \quad (8)$$

Indradev and Doherty modified the equation in two ways: the number of cube nuclei per band, N_c , was allowed to vary between 1 and 2, depending on the detailed structure of the deformed cube band,⁽⁵⁰⁾ and they included the prediction of λ_c given in eq. (7). Their form of eq. (8) was:

$$\alpha_c = N_c d_R / \lambda_c = N_c d_R \exp(\epsilon) / d_c(0) \quad (8a)$$

The initial cube grain separation, $d_c(0)$, is determined by the initial grain size, d_0 , and the initial cube grain frequency, $\alpha_c(0)$:

$$d_c(0) = d_0 / \alpha_c(0) \quad (9)$$

So:

$$\alpha_c = [N_c d_R \exp(\epsilon) \alpha_c(0)] / d_0 \quad (8b)$$

This model matches qualitatively all experimentally observed trends in cube texture (items 1 to 4) noted above, it fitted quantitatively the data in one study⁽⁵⁰⁾ and it showed close agreement⁽⁶¹⁾ with a wide range of experimental data which were obtained

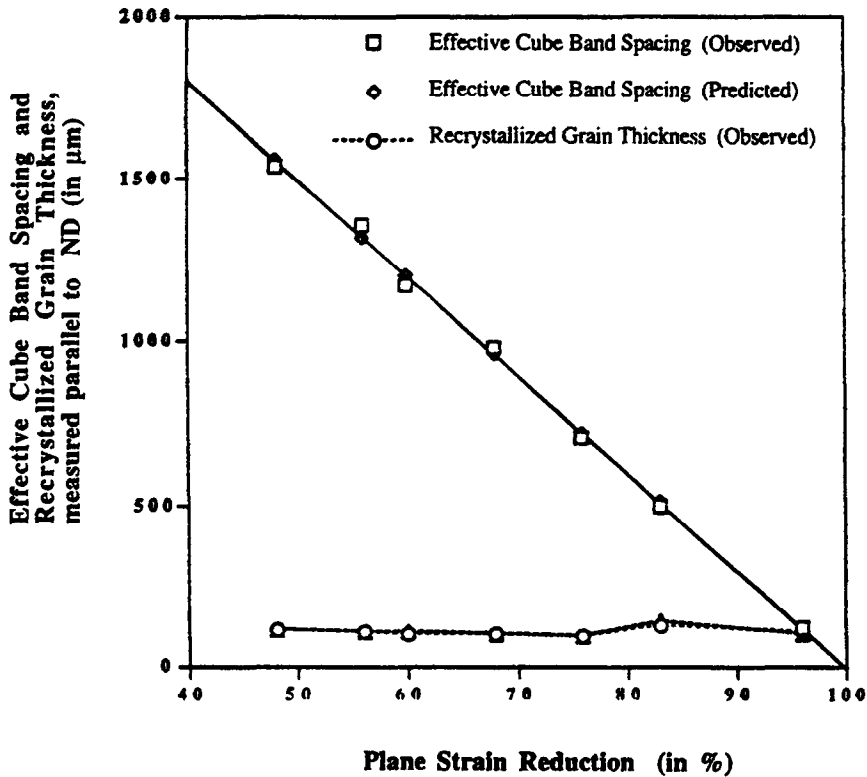


Fig. 8. Experimental spacing, λ_c , between cube bands (square symbols), predicted values from eq. (7) (full line) and the experimental value of the recrystallized grain thickness, d_R , all measurements in the normal direction, ND. After Samajdar and Doherty.⁽⁵⁰⁾

independently.⁽⁷⁸⁻⁸⁰⁾ It was particularly striking that the role of deformation temperature in promoting cube texture⁽⁶¹⁾ appeared to come from its effect on reducing the frequency of competing non-cube nucleation sites. That is, higher deformation temperatures lead to much higher values of the recrystallized grain thickness, d_R , while having little apparent impact on N_c . A full development of the ideas will require experimental insight into the features that determine the cube grain stability, eq. (7), and the parameters that control the recrystallized grain thickness, d_R .

5. VARIANT INHIBITION-ORIENTATION PINNING

In an recent review exploring these ideas,⁽⁶¹⁾ despite much success, a striking conflict was found with *one experiment* reported in ⁽⁸⁰⁾. The conflict was for an aluminium alloy with a very fine grain size, $d_0 = 50 \mu\text{m}$, a very high starting cube fraction, $\alpha_c(0) = 0.35$ and high strain $\epsilon = 2$, that yielded a much finer predicted cube spacing, $\lambda_c = 20 \mu\text{m}$, than the experimental recrystallized grain size, $d_R = 150 \mu\text{m}$. However, in conflict with the prediction of eq. 8b, this sample gave a *very low recrystallized cube fraction*. The fit then required a very small cube nucleation frequency per deformed cube band, a value of N_c of only 0.02, rather than N_c of ≈ 1 . The solution offered for this failure⁽⁶¹⁾ was an idea based

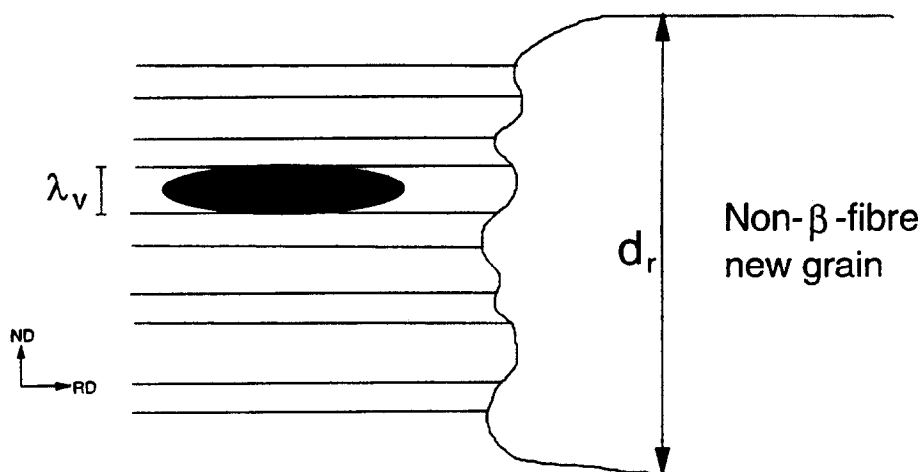


Fig. 9. *Variant inhibition*. Restricted growth of a new grain of an orientation that has a fine interband spacing, λ_v , between deformation texture variants of the same orientation. Grains of orientations that are only minority components of the deformation texture can grow to a much larger thickness. For most warm-deformed aluminium samples cube is a minority component but not for samples with a fine initial grain size and a very high initial cube grain fraction, so variant inhibition of cube can occur under these circumstances. Inhibition of detained rolling components is expected for warm deformation since the deformed bands are then continuous—this is unlikely after cold rolling, see Fig. 5. After Doherty *et al.*,⁽⁶¹⁾ courtesy of Riso National Laboratory.

on the *low mobility of low-angle grain boundaries*. A cube nucleus growing out of one cube band will have its growth inhibited if it meets another continuous deformed band of similar orientation, i.e. as in the “rogue” sample if $\lambda_c \ll d_R$. The same idea accounts for the lack of any retained deformation texture in *heavily warm-rolled* aluminium alloys^(45, 49, 50) since there will be exactly this inhibition for any new grain with an orientation from within the deformation texture, brass, S or copper, since $\lambda_v \ll d_R$, Fig. 9. λ_v is the spacing between the same variant of the deformation texture component. This hypothesis was named “variant inhibition”. At the conference where this idea was proposed,⁽⁶¹⁾ Juul-Jensen presented, independently at the same conference, a very similar idea⁽⁶¹⁾ which she called orientation inhibition, Fig. 10. The idea here is the same: a recrystallized grain will have its growth inhibited by contact with regions of the deformed microstructure with a similar orientation—the recrystallization front will have low mobility boundaries with these regions. Juul-Jensen^(81, 82) reported that, in partially recrystallized regions in both aluminium and copper, new grains with either rolling texture or random orientations had 10–25% of low-angle grain boundaries while cube grains had only 3–10% of such low-mobility boundaries. These measurements fit with the observed slower growth rates of non-cube grains—so accounting for the $\beta > 1$ values reported in the studies.^(75, 81, 82)

There is, see Fig. 5, clear evidence for strong retained rolling texture in commercial-purity aluminium alloys heavily cold-rolled at room temperature. Other evidence for this is given, for example, by^(45, 75, 76). Given that the commercial-purity alloys contain large iron-rich constituents of a diameter of 1–2 μm that will have misoriented deformation zones of twice this size, then these deformation zones will be thicker than the deformed bands of the deformation texture variants. These regions will disrupt the

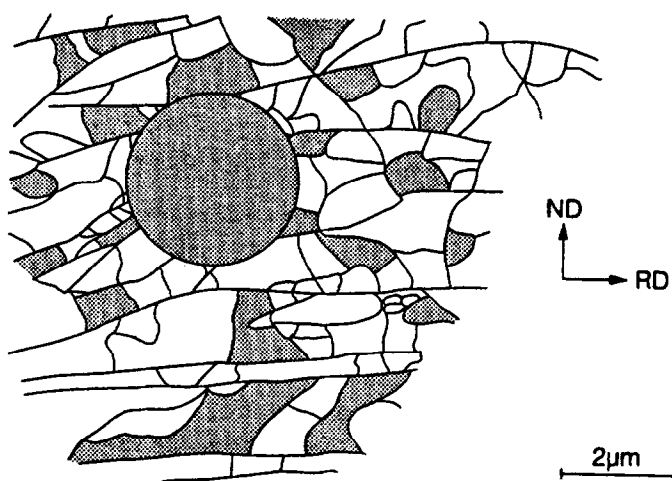


Fig. 10. *Orientation pinning.* A spherical nucleus is seen growing into a heavily deformed microstructure. When the nucleus grows into areas with orientations close to that of the nucleus (shaded in grey), the motion will be hindered by the low mobility of the low-angle boundaries. After Juul-Jensen,⁽⁸¹⁾ courtesy of Riso National Laboratory.

continuous bands of deformation texture orientation and allow the growth of new grains of the retained rolling orientation to grow through the deformed bands. As a result, the variant inhibition of Fig. 9 will be much weaker in cold-rolled commercial-purity aluminium than in the warm-deformed aluminium^(45, 50) so accounting for the strong retained rolling texture seen in cold-rolled commercial-purity aluminium.^(45, 70, 75) These ideas are presently rather speculative but can be easily tested by detailed microstructural examination of heavily cold-rolled commercial-purity aluminium, if examined in the partially recrystallized state.^(81, 82)

6. CONCLUSIONS

The ideas developed nearly 45 years ago by Robert Cahn^(22, 23) on the nucleation mechanism in recrystallization, extended by the observation by Cottrell⁽²⁶⁾ of low mobility of low-angle grain boundaries, have formed the basis of all successful understanding of nucleation of recrystallization. These ideas are now leading to some real understanding and predictive capacity for controlling recrystallization texture in high-purity and commercial fcc alloys. Lack of time, and lack of expertise by the reviewer, did not allow consideration of the equivalent success being achieved for the understanding of recrystallization texture in steel sheet after cold rolling. This subject has been reviewed^(67, 69) recently and the interested reader is directed to these publications. The ideas expressed there appear to be as closely in agreement with the Cahn–Cottrell mechanism as are the studies of fcc metals described here.

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