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STRUCTURAL EFFECTS ON GRAIN BOUNDARY SEGREGATION, HARDENING AND FRACTURE

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Abstract - Recent experimental studies of structural effects on grain boundary segregation, hardening and fracture in metals and alloys are discussed.
It has been found that grain boundary segregation, hardening and fracture are difficult to occur at low angle boundaries and coincidence boundaries which are both low energy grain boundaries. On the other hand, high angle general (so called "random") boundaries can be preferential sites for these boundary phenomena. In polycrystals, their fracture mode and fracture behaviour have been found to depend on the grain boundary character distribution in them. Intergranular fracture will occur typically in a polycrystal with a high frequency of random boundaries. The grain boundary character distributions in real polycrystalline materials produced by different fabrication processes are discussed. A new idea " Grain Boundary Design " for strong and ductile polycrystals which has been proposed recently is mentioned briefly.

1. Introduction

The presence of grain boundaries and interphase boundaries affect significant influence on mechanical properties of polycrystalline metals and ceramics. It is well known that the introduction of a high density of grain boundaries by grain refinement enhances the strength and ductility of polycrystals. However, grain boundaries can be also potential sites for fracture. The occurrence of intergranular fracture causes a loss in ductility and embrittlement associated with grain boundary segregation and hardening in polycrystalline materials. It is a general tendency that strong polycrystalline materials become more sensitive to intergranular fracture and show a lower ductility. Therefore, it is of great interest to find some possibility to develop strong and ductile polycrystalline materials on the basis of our current knowledge of effects of grain boundary structure on intergranular fracture and its related grain boundary phenomena. The present paper is based on a recent paper by the present author (1) and unpublished work regarding structural effects on grain boundary segregation, hardening and fracture in metals and alloys.

2. Structure-Dependent Grain Boundary Segregation and Hardening

(a) Misorientation Dependence of Grain Boundary Segregation

Effects of grain boundary type and structure on grain boundary phenomena can be studied rather easily on bicrystal specimens with well-defined grain boundaries. As for the effect of grain boundary structure on segregation, Thomas and Chalmers are probably the first who made a quantitative study of misorientation dependence of grain boundary segregation. They studied the misorientation dependence of polonium segregation to <001> symmetric tilt boundaries in Pb-5%Bi alloy bicrystals by autoradiography (2). It was found that the extent of polonium segregation increased very slightly up to 15°, then an abrupt increase occurred above the misorientation angle. The concentration of polonium at the 25° tilt boundary was almost one order higher than that for the 15° boundary. After their work several workers have also observed by autoradiography that the extent of solute and impurity segregation is large at high angle boundaries but not so at low angle boundaries for sulfur segregation in alpha iron (3) and iron-silicon alloy (4).

Inspite of these experimental evidence, the structural effect on grain boundary segregation has been long ignored even in quantitative discussion of the phenomenon, until recently. Since the time of the advent of Auger electron spectroscopy (AES), several workers noticed again that the extent of segregation varies from boundary to boundary on intergranularly fractured surface of polycrystal specimens of Cu-Bi alloy (5-7) and a low alloy steel (8), for example. Hondros has mentioned that the extent of segregation should depend on the nature and crystallography of the grain boundary (7). So far, it has been shown that high energy grain boundaries are more preferential sites for segregation and absorb more segregants than low energy boundaries such as low angle boundary and coincidence boundary, through indirect observations on the boundary segregation (9-10). However, it is only recently that the effect of grain boundary structure has started to be discussed seriously on grain boundary segregation. Accordingly, there is still a lack of experimental data on structural effect on segregation, and our present understanding of the effect is still far from complete, as mentioned by Balluffi in his recent review on the present subject (11).

Watanabe et al.have recently studied the effect of boundary misorientation on grain boundary segregation of silicon in an iron-silicon alloy (12) and of tin in an iron-tin alloy (13) by applying AES technique to bicrystal specimens containing grain boundaries of mixed type (having tilt and twist components of the boundary misorientation). The result is shown in Fig.1 which demonstrates clearly that the amount of segregation of tin and silicon varies with tilt angle, increasing rapidly for tin segregation and slightly for silicon segregation. It should be noted that the tilt component of the boundary misorientation is responsible for structural effect on segregation but not the twist component. Figure 2 shows the result of misorientation dependence of segregation obtained by replotting the data of Fig.1 as a function of misorientation angle about <110> rotation axis. It is evident that the amount of tin segregation increases rapidly in the low angle regime and then shows a cusp around 70° which corresponds to Σ3 coincidence misorientation relationship. Recent experimetal work of Brosse and Biscondi on molybdenum bicrystals has shown that the amount of oxygen segregation to <110> tilt boundaries is small at the Σ3 and Σ11 coincidence orientations (14).

The effect of boundary orientation on grain boundary segregation has been observed by Suzuki et al.(15) on phosphorus segregation to grain boundaries in an iron-phosphorus binary alloy. However, Briant has shown that variation in the amount of segregation in a single grain boundary is not so great as the variation among grain boundaries (16). This may suggest that the effect of boundary inclination is not so

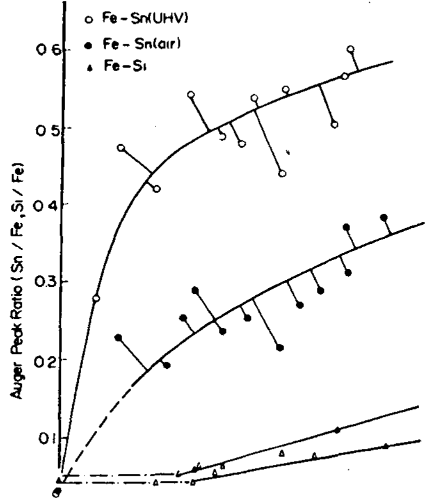

Fig.1. Misorientation dependence of the amount of segregation of tin and silicon in iron-tin and iron-silicon binary alloys (12,13).

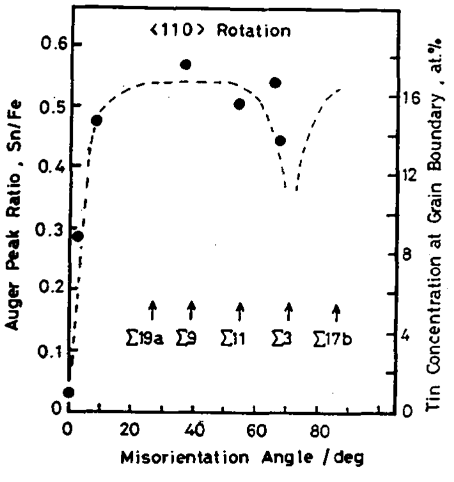

Fig.2. Misorientation dependence of tin segregation as a function of rotation angle about <110> axis in alpha iron-1.08 at.% tin alloy.

great as the effect of boundary misorientation, except for special cases such as twin (Σ3 coincidence) boundary and plane-matching boundary, although both boundary misorientation and inclination are important parameters to characterize the boundaries.

(b). Grain Boundary Segregation Diagram

In order to describe structure-dependent segregation, Watanabe et al.(13) have presented a " grain boundary segregation diagram " constructed on the basis of Seah-Hondros diagram describing the relationship between grain boundary enrichment ratio and the solid solubility of segregating element in the matrix (17) in connection with observations on misorientation dependence of segregation. Figure 3 shows a schematic representation of the diagram. An important feature of this diagram is that the more prominent misorientation dependence of segregation would be possible even for the grain boundaries with the same character and misorientation, as the solid solubility of segregating element in the matrix is decreased. For example, Cu-Bi system will give a strong misorientation dependence of segregation.

Quite recently, structural effect on grain boundary segregation has been discussed theoretically on the basis of computer calculations of atomistic structures of segregated grain boundaries (18,19). However, current discussions still remain qualitative presently. In order to test previous thoeries and to obtain our deeper understanding of structure-dependent segregation, further experimental work is much required regarding structural effect on segregation.
Moreover, another type of experimental work is also required which reveals the difference in structural effect on segregation at static and migrating grain boundaries. Recently it has been reported that more significant segregation can occur at migrating grain boundaries (20).

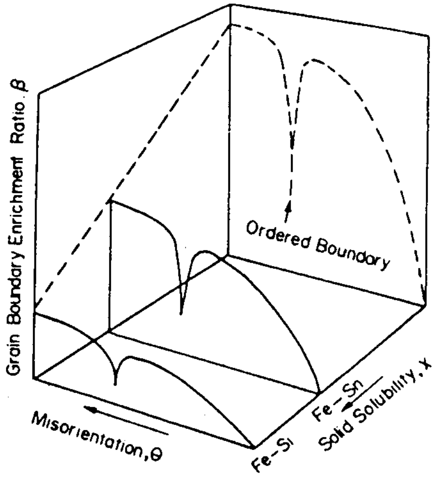

Fig.3. A schematic representation of the grain boundary segregation diagram (13).

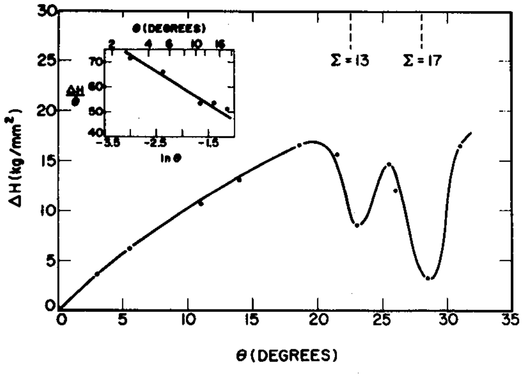

Fig.4. Misorientation dependence of grain boundary hardening for <100> symmetric tilt boundaries in niobium bicrystals (24).

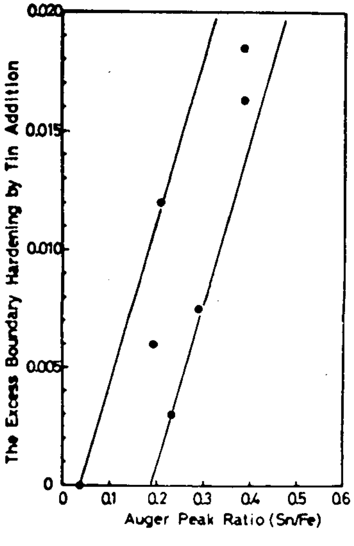

Fig.5. Relationship between the excess grain boundary hardening and the amount of segregation (13).

(c) Structure-Dependent Grain Boundary Hardening

Grain boundary hardening which is denoted by an increased hardness of grain boundary region has been believed to be attributed to grain boundary segregation (21,22). Westbrook and Aust have shown that the degree of grain boundary hardening, i.e. the excess hardening over the grain interior varies depending upon the type of grain boundaries in lead bicrystals (23). High angle random (general) boundary gave the greatest hardening, meadium degree of the hardening at 40°<110>/Σ9 coincidence boundary, and 15°<100> tilt boundary, incoherent and coherent twin boundaries showed a little or no grain boundary hardening.

Chou et al. have recently made a quantitative investigation into misorientation dependence of grain boundary hardening on niobium bicrystals containing <100>, <011> and <111> symmetric tilt boundaries with systematically controlled misorientation (24). Figure 4 shows the result for <100> tilt boundaries. The grain boundary hardening ΔH (defined as the difference in the hardness between the boundary and the grain) increases smoothly with the tilt angle up to 20°, then two cusps appear at the angles corresponding to 22.4°<100>/Σ13 and 28°<100>/Σ17 coincidence relationships. It is worth remembering that these coincidence boundaries gave rise to less significant grain boundary hardening than that for some low angle boundaries. Watanabe et al. have previously observed lesser extent of grain boundary hardening at Σ3 and Σ7 coincidence boundaries in the iron-tin alloy (13). The lesser extent of grain boundary hardening at coincidence boundaries and low angle boundaries may be related to the lesser extent of segregation at these boundaries, as mentioned soon later. Another interesting feature of observed structural effect on grain boundary hardening is that the twist angle of the boundary misorientation is not responsible for the hardening of mixed type of grain boundaries (13) and of pure <111> twist boundaries (22). The reason why only the tilt component is responsible for grain boundary hardening has not yet been well interpreted.

(d) Relationship between Grain Boundary Segregation and Hardening

Although the observed structural effect on grain boundary hardening appears to be consistent with an expectation of the hardening due to grain boundary segregation, there was no direct evidence for the connection between them, until recently. In order to confirm this, Watanabe et al.(13) made measurements of grain boundary hardness and the amount of segregation at the same grain boundaries in an iron-tin alloy. The amount of segregation of tin was determined by AES after measurement of the hardness at a given grain boundary in a bicrystal specimen. A relationship between the excess grain boundary hardening by tin addition and the amount of tin segregation is reasonably interrelated, as shown in Fig.5. Although the plots are scattered in a wide band, we can clearly recognize that the excess grain boundary hardening by tin addition is enhanced by tin segregation. We consider that this would directly prove the origin of grain boundary hardening associated with grain boundary segregation. However, we must be careful in discussing mechanism of grain boundary hardening because there is a large difference between the width of hardened grain boundary region (within 50 - 100 μm) and that of segregated boundary region (smaller than a few nanometers). This huge gap and mechanism of grain boundary hardening have not been well interpreted. Since grain boundary hardening is thought to play some important role in intergranular fracture, a study of structural effect on grain boundary hardening is much required in order to understand segregation-assisted intergranular fracture. Microscopic work of grain boundary hardening has been scarcely done so far.

3. Basis of Structure-Dependent Intergranular Fracture

When intergranular fracture occurs ideally in the absence of plastic deformation, the fracture energy γ^* is given by (25)

$$\gamma^* = 2\gamma_s - \gamma_b \qquad (1)$$

where γ_s is the surface energy of the exposed grain boundary and γ_b the pre-existing grain boundary energy. Since it is well established experimentally and theoretically that grain boundary energy γ_b depends on grain boundary structure (26), we can expect some dependence of the intergranular fracture energy γ^* on the boundary structure if

the surface energy γ_s does not depend on crystallographic orientation as much as γ_b. The value of γ^* for low-energy grain boundaries will be large and intergranular fracture will be more difficult than at high energy grain boundaries.
When plastic deformation is involved in intergranular fracture, the fracture energy γ^* is given by

$$\gamma^* = 2\gamma_s - \gamma_b + \gamma_p. \qquad (2)$$

where γ_p is plastic work associated with the propagation of the crack.
Here, we should remember that as the contribution of γ_p to the fracture energy γ^* decreases, intergranular fracture will occur more predominantly and ideally, and the effect of grain boundary structure would be increasingly important to the fracture process in polycrystals. We may say that intergranular fracture has a intrinsically strong dependence on grain boundary structure. Therefore, thorough understanding of the structural effect on intergranular fracture is indispensable to the control of brittleness of polycrystalline materials.

4. Structure-Dependent Intergranular Fracture in Bicrystals

(a). Low-Temperature Intergranular Fracture

Intergranular fracture stress can be determined by experiment on a bicrystal specimen as a measure of grain boundary cohesion. So far, several workers have made systematic investigations into the effects of boundary type and misorientation on intergranular fracture using bicrystal specimens of metals such as molybdenum (27-31) and tungsten (32,33), and of ceramics (34). According to the previous works, it has become evident that the fracture stress is low for high-angle boundaries with the misorientation above a certain angle, then it increases rapidly with decreasing the misorientation angle in the low angle regime. The critical misorientation angle at which a rapid increase in the fracture stress occurs,appears to depend on the material and the type of grain boundaries (27-33).

Figure 6 shows stress-strain curves for molybdenum bicrystals with <110> symmetric tilt boundary, obtained most recently by Kurishita et al. by four points bending test at 77K. It is clear that 10° low angle boundary and 73° high angle boundary have a large fracture stress and fracture strain, almost equal to the fracture stress and strain for single crystal. Other grain boundaries show rather small fracture stress and strain, particularly at 25°. The 73° high angle boundary is considered to be slightly off 70°<110>/$\Sigma 3$ coincidence (twin) boundary. Brosse and Biscondi have also found that high angle boundaries with coincidence orientations (50.5°<110>/$\Sigma 11$. and 109.5°<110>/$\Sigma 3$) have a large fracture stress and fracture strain on molybdenum

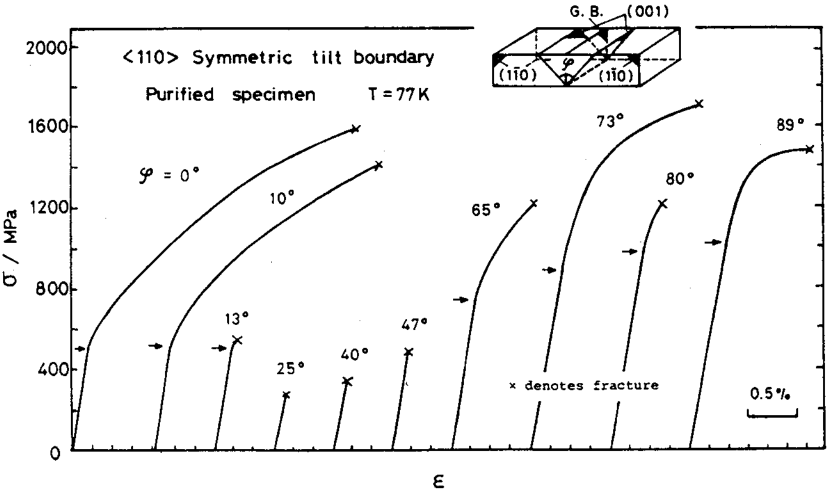

Fig.6. Stress-strain curves for molybdenum bicrystals with <110> symmetric tilt boundaries of different misorientations (31).

bicrystals at room temperature (29). Greater resistence to fracture of low-energy grain boundaries has been found by Roy et al. in copper-0.1 at.% bismuth alloy in which intergranular fracture is enhanced by grain boundary segregation of bismuth(35).

Figure 7 shows an interesting observation made by the present author on intergranular fracture occurring in a copper-bismuth alloy. The propagation of the intergranular cracks stopped completely at the portion A on the grain boundary which a twin meets. It is supposed that the structure of the grain boundary had been partly modified by the nucleation of the twin from the boundary. We can easily recognize how strongly intergranular fracture process depends on the type and structure of the grain boundary. Any modification of the original structure of the boundary by some mean will give rise to the change in grain boundary cohesion. Grain boundary segregation can modify the atomic structure of grain boundaries to result in the change in the boundary cohesion. Kumar and Eyre have studied the effect of grain boundary segregation on fracture of slightly off 154°<113>/Σ3 twist boundary in molybdenum (36). They found that the fracture stress was increased by segregation of carbon through its suppresion effect on oxygen segregation to the grain boundary.

Watanabe et al. have studied the effects of solute segregation and of third element addition on intergranular fracture in alpha iron-tin and iron-tin-molybdenum alloys (37), by three points bending test of bicrystal specimens. The result obtained is shown in Fig.8. The fracture stress decreased rapidly from some 120 MPa to 20 MPa with increasing the misorientation angle from 10° to 30°, being almost constant above 30°. The most important finding was that the addition of molybdenum to the iron-tin alloy shifted the fracture stress-misorientation curve to the right hand side and an increase in the fracture stress was observed at the misorientations below 40°. In this case the amount of tin segregation was found to decrease with increasing the amount of molybdenum segregation to the same grain boundary. Deembrittling effect of molybdenum on phosphorus-induced embrittlement has been reported (38). The deembrittling effect is considered to be due to strongly attractive Mo-P interaction which decreases the amount of segregation of phosphorus to grain boundaries.

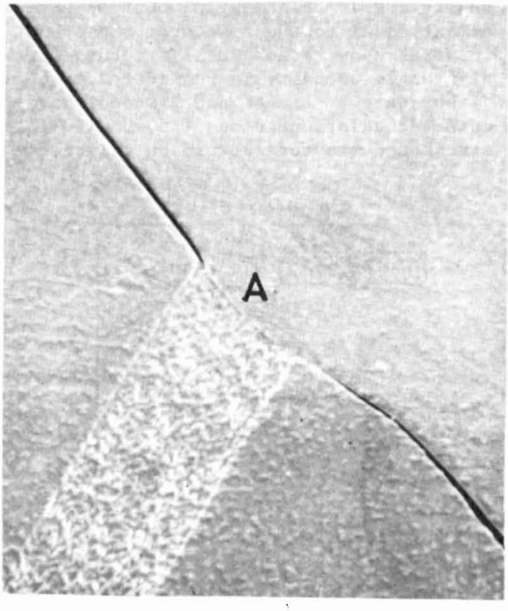

Fig.7. An observation of the stoppage of crack propagation at the part of the grain boundary which a twin meets.

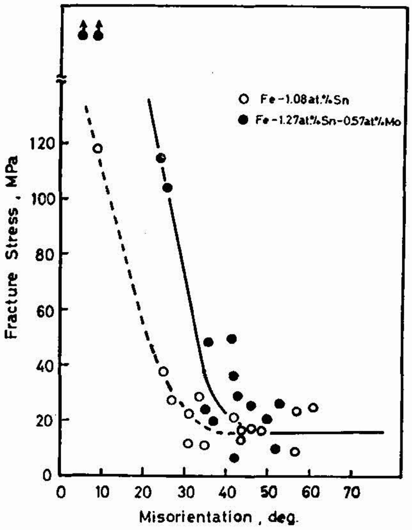

Fig.8. The effect of molybdenum upon misorientation dependence of fracture stress in alpha iron-tin alloy.

(b). High Temperature Intergranular Fracture

So far, there has been a few experimental studies of the effect of grain boundary structure on intergranular fracture in metals and alloys at high temperatures. The present author and coworkers have made systematic investigations of high temperature intergranular fracture associated with grain boundary sliding. The author has recently written several overview papers (1, 39, 40) in which he discusses effects of grain boundary type and structure on high temperature intergranular fracture in some detail. It has been found that high temperature creep fracture strongly depends on the type and structure of the grain boundaries, beeing associated with grain boundary sliding which is much dependent on grain boundary structure. Low angle boundaries and coincidence boundaries are difficult to slide and fracture while high angle random boundaries slide and break easily.

(c). Liquid-Metal-Induced Intergranular Fracture

Liquid-metal-induced intergranular fracture is known to occur in a brittle manner. Kargol and Albright (41) have made an excellent study of structural effect on intergranular fracture induced by liquid Hg-3at.%Ga for <110> symmetric tilt aluminium grain boundaries. They found that the crack extension force at a given velocity strongly depends on the boundary misorientation, having peaks at 70° and 130°tilt angles which correspond $\Sigma 3$ and $\Sigma 11$ coincidence orientations. Their experiments have shown unambiguously the presence of special grain boundaries having the high crack propagation resistance.

Watanabe et al. have also studied the structural effect on liquid-metal-induced intergranular fracture on $\langle 10\bar{1}0\rangle$ tilt and twist zinc bicrystalsin the presence of liquid gallium (42). As shown in Fig.9 the fracture stress depends strongly on the misorientation angle and has a peak at 55° which corresponds to the $56.6^\circ\langle 10\bar{1}0\rangle/\Sigma 9$ near-coincidence orientation in zinc with HCP structure. Again, low angle boundaries and coincidence boundary with lower Σ value show high fracture stress and high crack propagation resistance.

5. Structure-Dependent Intergranular Fracture in Polycrystals

In comparison with bicrystal specimens, more complicated stress situations may exist at or near the boundaries in polycrystal specimens. Therefore, it is supposed that if the structural effect was not strong enough, it would be masked by other factors such as stress condition at the boundary. In order to make this point clear, structural effects on three types of intergranular fracture (low temperature fracture, high temperature creep fracture and liquid-metal-induced fracture)have been studied on polycrystals (1, 40, 43).

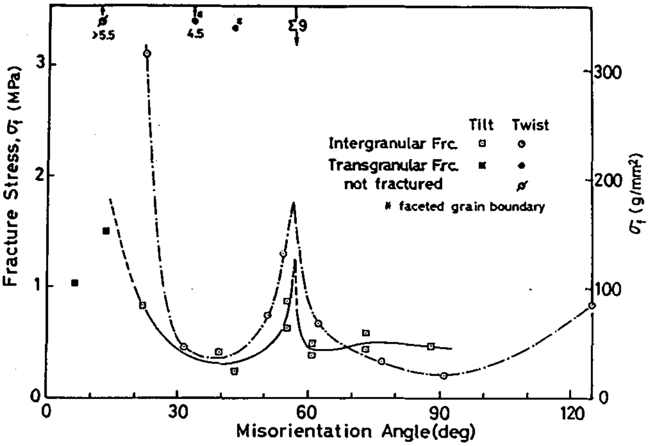

Fig.9. Misorientation dependence of fracture stress for $\langle 10\bar{1}0\rangle$ tilt and twist bicrystals in zinc (43).

The characterization of all the grain boundaries in each polycrystal specimen was made by the electron channelling pattern (ECP) technique. Figure 10 shows an example of the characterization of grain boundaries in a beta brass polycrystal specimen for the test of liquid-gallium-induced intergranular fracture. Letters,R, Σ and L denote random boundary, coincidence boundary and low angle boundary, respectively.
It was found that intergranular fracture preferentially occurred at high angle random boundaries, but coincidence and low angle boundaries were difficult to break, as just observed on bicrystal specimens. Now we may say that the effect of grain boundary structure on intergranular fracture must be strong enough not be masked by complicated stress situations in polycrystals.

Another important finding on fracture process in polycrystals is that the fracture mode changed from intergranular to transgranular or vice versa during crack propagation (43). SEM in-situ observations have revealed that the change in fracture mode occurs, depending on the type of grain boundaries which the propagating crack meets in beta brass polycrystals.
Fracture processes in a polycrystal are shown schematically in Fig.11 which illustrates two fracture paths (A and B) started from the bottom of the figure. In the case of Path A, a crack nucleated at a random boundary propagates to a connecting coincidence boundary, but it is prevented from further propagation along the boundary because of high propagation resistance of the coincidence boundary, and then enters the grain interior, changing the fracture mode. On the other hand, in the case B, the crack nucleated at a random boundary keeps propagating, selecting random boundaries at every triple point. Consequently, intergranular fracture can occur ideally and no change in the fracture mode is involved in the fracture process.

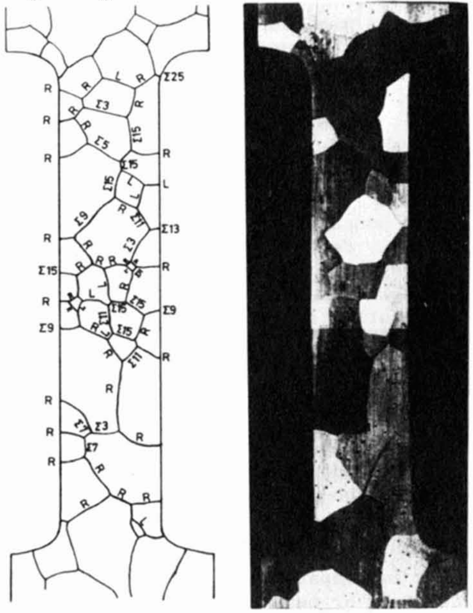

Fig.10. The characterization of all the grain boundaries in a beta brass polycrystal specimen (43).

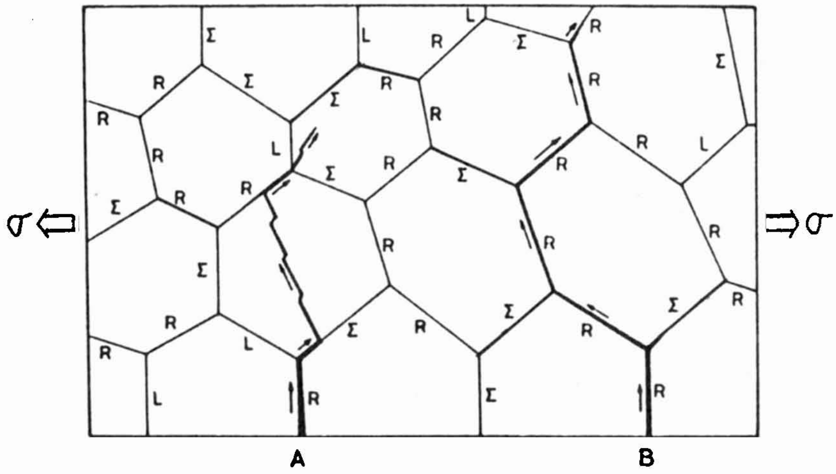

Fig.11. Schematic respresentation of grain boundary structure-dependent fracture processes in polycrystal. Path A: combined process of intergranular and transgranular fractures.

6. Grain Boundary Character Distributions for Strong and Ductile Polycrystals

It has been already shown that the character of a grain boundary and its distributions are important parameters controlling the propensity of intergranular brittle fracture and the ductility of polycrystals. Therefore, it is indispensable to study the grain boundary character distributions in polycrystals. Recently we have made a systematic investigation into the grain boundary character distributions in polycrystals produced by different fabrication processes; aluminium polycrystals produced by annealing single crystals compressively deformed (44), ordinary rolled and annealed beta brass (43),iron-3mass% silicon and other iron-base alloy (46), and sintered powder iron polycrystals (47).

In the case of aluminium polycrystals produced from single crystals, it has been found that the frequency of coincidence boundaries with Σ values smaller than 29 ranged from 20% to 30%, depending on the original orientation of single crystal and the amount of prestrain (43). Belluz and Aust found that the frequency of coincidence boundaries in aluminium polycrystals produced from single crystals was increased up to 50% by addition of small amount of tin (10 ppm)

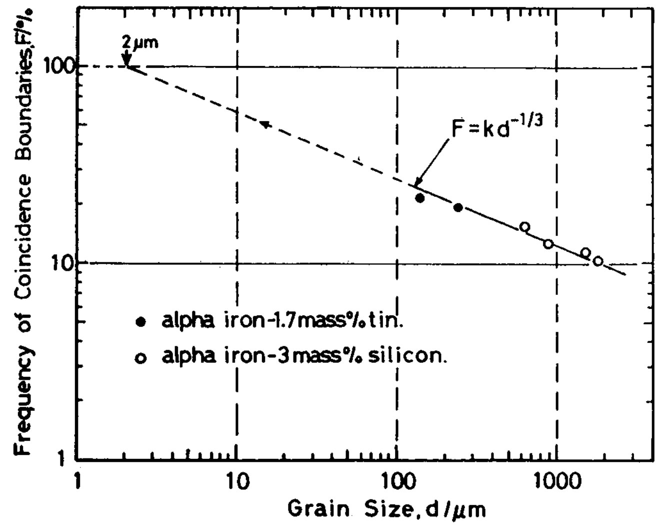

Fig.12 Relationship between the frequency of coincidence boundaries and grain size for recrystallized polycrystals of alpha iron alloys.

Most recently, a relationship between the frequency of coincidence boundaries and grain size for alpha iron base alloys has been studied by the present author and his coworkers. The result is shown in Fig.12, which plotted the frequency of coincidence boundaries as a function of grain size.

It is clear that the frequency increases with decreasing grain size. Furthermore, an interesting finding has been obtained:

When extrapolated to a range of grain size below 10 μm, the relationship gives the frequency of coincidence boundaries over 60%, and 100% for the grain size of 2 μm. This result gives us a new insight into the origin of high ductility of untrafine grained polycrystals. It seems very possible that high ductility of polycrystals is attributed to a high frequency of coincidence boundaries which are now known to be very resistent to intergranular fracture.

It is of our great interest to know how we can increase the frequency of the grain boundaries which have high resistance to intergranular fracture,in order to obtain strong and ductile polycrystals. This approach seems to the author very promising as a new approach to materials design based on current knowledge of grain boundary structure and properties, so called " Grain Boundary Design ".

Acknowledgements

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