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GRAIN BOUNDARY DESIGN FOR DESIRABLE MECHANICAL PROPERTIES

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Abstract - Grain boundary design for desirable mechanical properties of polycrystalline materials is discussed on the basis of the intrinsic structure-dependent mechanical properties, particularly deformation and fracture of grain boundaries. The importance of the grain boundary character distribution (GBCD) is emphasized which is a new microstructural parameter controlling the properties of polycrystals. The results of recent studies of GBCD are presented. The potential for grain boundary design for strong and ductile polycrystals through the control of GBCD and boundary inclination is discussed.

1. Introduction - The control of microstructure is a basic approach to materials development established by modern materials science which is based on the principle of structure-property-performance relationship (1-3). Grain boundaries (including interphase boundary) are important elements of the microstructure of polycrystalline materials. They can affect considerable effect on the properties and performance of the materials. So the design and control of grain boundaries are strongly required to develop high performance engineering materials with desirable properties (4,5). Recent rapid progress in our understanding of grain boundary structure and properties (6-9) appears to enable us to achieve a new approach to materials development. It has been revealed that the intrinsic grain boundary properties strongly depend on the type and structure of grain boundaries. It is very likely that structure-dependent grain boundary properties can be effectively utilized in materials development by manipulating grain boundaries to endow polycrystalline materials with desirable bulk properties.

2. Desirable Mechanical Properties for Structural or Functional Materials. Mechanical properties are the most important for structural materials which are normally required to meet the following requirements: high strength, high ductility, high fracture resistance, elastic and plastic homogeneity, low susceptibility to environmental effects, high stability of microstructure and properties and so on. These requirements are related more or less to the presence and effect of grain boundaries. For instance high strength and high ductility are often obtained by grain refinement, i.e. by incorporation of a high density of grain boundaries in polycrystalline materials. Normally almost all engineering materials whether structural or functional, must receive some kind of working and shaping before they are put in service. In the past there were a number of cases where materials could not be developed because of their undesirable mechanical properties, particularly brittleness or low ductility associated with intergranular fracture. It is well known that there is a general tendency for strong materials to become more sensitive to intergranular...
fracture and show a loss in ductility after some strengthening treatment. This is a long-pending problem which materials scientists and engineers must solve. There has been no general rule or working principle to obtain strong and ductile polycrystalline materials. This is a very challenging subject regarding the development of high performance materials with desirable and excellent mechanical properties.

3. Structural Effects on Intergranular Mechanical Properties

Basic knowledge of structural effects on intergranular mechanical properties is needed for later discussion on grain boundary design for desirable mechanical properties. In order to quantitatively analyse observed structural effects we need to characterize grain boundaries existing in real materials. In general, there are two groups of boundaries: low-energy boundaries and high-energy boundaries. We consider that low-angle boundaries and low $\Sigma$ coincidence boundaries belong to the first group and high angle general so-called random boundaries to the second. Throughout this paper we consider these three types of boundaries.

3.1 Deformation - The presence of grain boundaries can affect plastic deformation in crystalline solids through the generation or absorption of lattice dislocations, or by provision of barriers to dislocation motion (10,11). At high temperatures grain boundaries can play more roles in plastic deformation as sources or sinks of point defects, and more directly by grain boundary sliding. So we can expect that there will be more significant effects of grain boundary structure on high temperature deformation than low temperature deformation. In fact, it has been reported that the effect of grain boundary structure on low temperature deformation can be observed in a very early stage of deformation but not in later stage (12,13). Whereas at high temperature structural effects of grain boundary appear retainable over the whole range of plastic deformation up to the final fracture. So let us see the effect of grain boundary on high temperature deformation in zinc bicrystals.

Figure 1 shows creep curves of zinc bicrystals containing $<10\bar{1}0>$ twist boundaries at $45^\circ$ to the specimen axis. Tensile creep tests were conducted at constant shear stress for basal planes of the grains for all the bicrystal specimens irrespective of the twist angle. It is clear that a single grain boundary can drastically affect creep deformation with different magnitudes depending on the twist angle. Surprisingly grain boundary sliding hardly occurred in all the bicrystal specimens tested.

Of particular interest was that the specimens which contained $51^\circ$ or $54^\circ$ twist boundaries which are regarded as slightly off $29$ near coincidence boundaries showed more difficult creep deformation. The observed misorientation dependence on creep deformation may be related to the effectiveness of boundary as dislocation barrier dependent on the type and structure of boundary. The absorption or emmission of dislocations by grain boundary have been found to be strongly dependent on boundary structure (14). Regarding the structural effect on dislocation passage across grain boundary, Lim and Raj (15) have done excellent experimental work on nickel bicrystals with $<110>$ symmetric tilt boundaries. They quantitatively studied the effect of grain boundary structure on the continuity of slip across coincidence boundaries with different $\Sigma$ values. Figure 2 shows the observed effect of boundary $\Sigma$ on the continuity of slip. It is evident that the degree of slip continuity decreases with increasing $\Sigma$, in other words disorder of boundary structure for slip induced by screw dislocations, but not at all for slip by mixed dislocations. Their work clearly shows that the manner and the degree of interaction between grain boundary and dislocations depend on the type and structure of grain boundary and on the type of dislocation. Low slip continuity at high $\Sigma$ boundaries has been explained by the ease of absorption of lattice dislocations and a higher recombination energy for dissociation products of lattice dislocations.

It has been well established that grain boundary sliding takes place by movement of lattice dislocations or their dissociation products along the boundary plane. Many investigators have shown by experiments that grain boundary sliding is a strongly structure-dependent intergranular phenomenon (16,17). An example of sliding behaviour strongly dependent on boundary type observed in aluminium (18) is shown in Fig.3. We can recognize a large difference in sliding behaviour between random boundaries (G.B.No 11 and 12) and coincidence boundaries (G.B.No 4 and 7). Sliding can takes place significantly with weak slide hardening at random boundaries, but it is difficult at coincidence boundaries because of prominent slide hardening. It is well accepted that grain boundary sliding is difficult at low-angle boundaries and low $\Sigma$.
Grain boundary sliding is a mechanism of high temperature deformation and superplasticity occurring in polycrystals. The structural effect on sliding may be related to the generation of the heterogeneity of deformation and of stress concentration at grain boundary irregularities or triple points. There is considerable evidence for important roles of sliding in intergranular fracture at high temperature, as shown later.

3.2 Fracture - Recently the effect of boundary structure on intergranular fracture has been extensively studied on orientation-controlled bicrystals of metals and alloys. The misorientation dependence of fracture stress and strain has been determined on molybdenum (20-22), zinc (23) and aluminium (24) bicrystals. It is commonly observed that low-angle boundaries and low Σ coincidence boundaries have higher fracture stress and strain than those for random boundaries. This is solid evidence that grain boundaries can be strong and ductile depending on the boundary type and misorientation. High temperature intergranular fracture is expected to have much stronger structural effect than low temperature intergranular fracture because intergranular fracture mechanisms involve more structure dependent boundary phenomena such as diffusion, sliding and migration.

So far several workers have made systematic investigations into the effect of boundary type on creep intergranular fracture (17,19,25-28). The propensity for creep intergranular fracture strongly depends on boundary type. As can be seen from Fig.4 random boundaries (denoted by R) were found to be preferential sites for fracture, but not for low-angle or low Σ coincidence boundaries (denoted by L or Σ plus numeral, respectively) irrespective of boundary inclination, in other words with or without involving grain boundary sliding in intergranular fracture.

Presently it is well accepted that sliding-induced creep intergranular fracture should have structure-dependence originated from sliding.

**Fig.1** Effect of boundary misorientation on creep deformation in <1010> twist zinc bicrystals.

**Fig.2** The effect of boundary Σ on the slip continuity in iso-axial <110> symmetric tilt bicrystals of nickel (Lim and Raj (15)).

**Fig.3** Sliding Curves obtained for random boundaries and coincidence boundaries in aluminium (18).
Until recently there was no experimental evidence that the boundary type can affect intergranular fracture occurring at the boundaries lying perpendicular to the stress axis, that is, in the situation in which any contribution of boundary sliding is expected. Quite recently Watanabe (28) has shown that creep intergranular fracture occurring at these boundaries is also dependent on the type of boundary. Table 1 presents statistical data of structure-dependent creep intergranular fracture observed on iron-0.8 mass% tin alloy polycrystals with different grain sizes. It is clear that most of fractured boundaries were random boundaries. Therefore it can be said that creep intergranular fracture is dependent on boundary type whether sliding-induced fracture mechanism or vacancy-condensation (diffusional) mechanism is operative. Don and Majumdar have found that the propensity for creep intergranular cavitation depends on the deviation from near coincidence orientation and Σ value in 304 stainless steel (26). Random boundaries which have the deviation angle Δθ exceeding the maximum value as coincidence boundary given by Brandon's criterion Δθ₀ = π/12(Σ)-1/2, tend to be cavitated severely, just similarly as was observed by Lim and Raj for cavitation in low-cycle fatigue at high temperature in nickel (29).

Figure 5 shows schematically structure-dependent intergranular fractures caused by sliding-assisted mechanism or by vacancy-condensation (diffusional) mechanism, on the basis of experimental observations that random boundaries can easily slide and fracture, but not low-angle boundaries and low Σ coincidence boundaries.

From the above discussion we are led to a conclusion that the presence of a high frequency of random boundaries will give rise to more intergranular fracture in polycrystals. Conversely an increase in the frequency of low-energy boundaries such as low-angle or low Σ coincidence boundaries will suppress the occurrence of intergranular fracture. This may be achieved by controlling the grain boundary character distribution in polycrystal as will be discussed in later section.

Now let us consider another important aspect of structural effects on high temperature intergranular fracture. In fact it has been increasingly recognized that the structure of grain boundary with a given misorientation can change from a low temperature structure to a high temperature one at transition temperature by increasing temperature. Such a grain boundary structural transformation may affect intergranular deformation and fracture at high temperature.

Figure 6 shows the temperature dependence of the average sliding rate on <1010> tilt zinc bicrystals recently reported (30). The temperature dependence was found to change at certain transition temperature Tc which varies with the tilt angle. The highest Tc was obtained on a 54.2° tilt boundary which is slightly off 56.6°<1010>/29 near coincidence orientation relationship. Moreover a 16.5° low-angle tilt boundary showed no change in the temperature dependence over the entire temperature. The observed change in temperature has been attributed to a grain boundary structural transformation.

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Fig. 4 Structure-dependent creep intergranular fracture observed in alpha iron–tin alloy.
Quite recently Shvindlerman and Straumal (31) have examined reported TEM observations on grain boundary dislocations at various temperatures. They found that the temperature above which dislocation image disappeared at coincidence boundaries supposedly by a boundary structural transformation, decreases with increasing C. From their result it is led that the coincidence boundaries with C smaller than about 3 can be stable at temperatures below 0.6Tm, Tm is the melting temperature. Conversely we expect that those coincidence boundaries may lose their special properties above 0.6 Tm. High C coincidence boundaries are considered to have lower thermal stability. It is reasonable to consider that temperature-induced structural transformation will affect high temperature intergranular deformation and fracture in polycrystals depending on the frequency of structurally stable or unstable boundaries. Unfortunately no consideration has been taken into this aspect. Structural modifications of grain boundaries may also be induced by segregation (32,33) and by incorporation of lattice dislocations (34,35).

4. Bridging between Bulk Properties of Polycrystal and Intrinsic Boundary Properties.

4.1 Importance of Grain Boundary Character Distribution, GBCD.

Up to now, when we want to predict the bulk properties of polycrystals from available basic knowledge of intrinsic boundary properties, there is no way because so far no consideration was taken into in discussing bulk properties of polycrystals. There is a gap between polycrystals and bicrystals. Accordingly useful informations on intrinsic structure-dependent properties of grain boundaries have not been fully utilized in materials development. Recent studies have revealed that low-energy boundaries and high-energy boundaries behave very differently, but very systematically depending on their boundary character which can be determined experimentally. Now what we need to know is statistical data concerning the distribution of low-energy or high-energy boundaries in real polycrystalline materials. This kind of statistical information has been called the grain boundary character distribution (5,36-38). Although it is still premature, the bulk properties are expected to be controlled by the grain boundary character distribution, GBCD. Furthermore basic research which reveals the relationship between GBCD and bulk properties of polycrystals is needed. GBCD is considered a new microstructural parameter which is necessary for explaining and predicting the bulk properties. The GBCD can bridge between the intrinsic properties of grain boundaries and the bulk properties of polycrystals.

4.2 Recent Studies of GBCD in Polycrystals.

More recently systematic investigations into GBCD for polycrystalline materials produced by different fabrication methods, as partly reviewed by the present author (5,36,37).

Fig. 5 Schematic representation of structure-dependent intergranular fracture caused by (a) sliding-induced mechanism and (b) vacancy condensation-diffusional mechanism.

Fig. 6 Temperature dependence of the average sliding rate on <110> tilt zinc bicrystals (30).
Regarding GBCD the following aspects need to be clarified. (i) the type of boundary actually existing in real polycrystals, (ii) the frequency of occurrence for individual types of boundaries, (iii) factors affecting GBCD and (iv) relationship between GBCD and bulk properties of polycrystals produced by well defined and controlled processing.

So far we have studied the GBCD on aluminium polycrystals produced by annealing compressed single crystals, iron and its alloys conventionally cast, rolled and annealed (Fe, Fe-Si, Fe-Sn and Fe-Co), cast iron-chromium alloy with columnar grain structure, rapidly solidified and annealed iron-6.5mass% silicon alloy ribbons, iron-cobalt alloy polycrystals annealed in magnetic field. The electron channelling pattern (ECP) technique for orientation analysis has been effectively used for the characterization of grain boundaries in polycrystals having a wide range of grain size (5μm-10mm).

Here we look at some important findings obtained from our studies and those by others. From the work on aluminium polycrystals produced from single crystals (38), it was found that the frequency of specific types of boundary (low-angle, low Σ coincidence with Σ=3-29, and random boundaries depends on the amount of prestrain (60%, 10%, 80%) and the initial orientation of single crystal sheet, as shown by Fig.7. The frequency of coincidence boundaries with Σ=3-29 tends to increase from about 20% to 30% with increasing compressive prestrain. It is surprising that the polycrystals produced from the single crystals with the initial orientations of 3 and 4 near (112) showed a drastic decrease in the frequency of low-angle boundaries with increasing prestrain. The observed high frequency of low-angle boundaries was found to be associated with the presence of (110) texture. Conversely the frequency of random boundaries increased from about 25% to 50% with increasing prestrain from 60% to 80%. There was not a significant change in GBCD for the polycrystals produced from single crystals with the orientation 2 near (110).

Rybin et al. (39) have also studied the GBCD on molybdenum polycrystals produced by annealing rolled or hydroextruded single crystals, or rolled polycrystals. It is of particular interest that an extremely high frequency (≈65%) of low-angle boundaries was observed on the polycrystal specimen produced from rolled single crystal, while hydroextruded and annealed specimen had the frequency of low-angle boundaries of 11% which is close to the value (16%) for the polycrystal specimen produced by annealing rolled polycrystal. It is clear that the GBCD of thermo-mechanically produced polycrystals depends on the mode of deformation, the amount of prestrain and the initial state of the matrix.

Watanabe et al. (40) studied the effect of annealing temperature on GBCD in rolled and annealed Fe-3mass% silicon alloy. As seen in Fig.8 the frequency of low-angle boundaries and of coincidence boundaries decreases with increasing annealing temperature. In counterbalancing the frequency of random boundaries increases. There seems to be a general trend that the frequency of random boundaries increases as grain size increases, as indicated by Fig.9. Such trend can be expected from the recent discussion on change in boundary energy during grain growth made by Watanabe (41) on the basis of a regular polygonal grain growth model.

Quite recently Watanabe et al. (42) have determined the GBCD for rapidly solidified and annealed Fe-6.5mass% Si alloy ribbons with high ductility and excellent magnetic properties developed by Arai et al. (43). We expected that the origin of high ductility of the material might be associated with the presence of a high frequency of low-energy boundaries resistant to fracture. As expected we observed a high frequency of low-energy boundaries of about 45% (low-angle boundaries: 24.8% and coincidence boundaries: 20.2%). So nearly a half of existing boundaries were found to be of low-energy. It should be mentioned that the GBCD for slightly annealed specimen (1363K, 10min) was almost the same as that theoretically calculated for random grain orientation distribution. This may suggest that the microstructure had not been much different from that of the as-solidified state. The frequency of low-angle boundaries should be 2.3%, however it increased drastically after full annealing (1363K for 1h). The result obtained is shown in Fig.10 together with GBCD data obtained for other materials.

In fact it was found that the frequency of coincidence boundaries can be well described by an inverse cube root law of Σ for iron and its alloys as seen from the figure. For the full annealed Fe-6.5mass%Si alloy ribbon the frequency of Σ1 (low angle boundary is regarded as Σ1 coincidence boundary), Σ5, Σ15, Σ25 coincidence boundaries which occurred more frequently, fell on a straight line. The inverse cube root law holds for the iron and its alloy polycrystals used, the slope of the lines is different among them. Moreover the GBCD data obtained from fcc nickel (43)
and 304 stainless steel (26) cannot be described by the inverse cube root law. This may be due to the presence of extremely high frequency of Σ3 twin boundaries (more than 40%) and Σ3-related coincidence boundaries probably produced by multiple-twinning. This suggests that the mechanisms of the formation of grain boundaries are different between the studied bcc materials and fcc materials.

Fig.7 Grain boundary character distributions observed on aluminium polycrystals produced by annealing compressed single crystals with different initial orientations (38).

Fig.8 Effect of annealing temperature on GBCD in alpha iron-3mass% silicon alloy (40).

Fig.9 The frequency of coincidence boundaries with Σ3-29 as a function of grain size in iron-3% silicon (40).

Fig.10 Σ dependence of the frequency of coincidence boundaries in some bcc and fcc polycrystals.
The success of quantitative description of the incidence of coincidence boundaries by the inverse cube root law implies the possibility of prediction of the incidence of the coincidence boundaries with a given $\Sigma$. In the case of the Fe-6.5mass% Si alloy ribbon with (100) texture, we can expect high incidence of $\Sigma 1, \Sigma 5, \Sigma 13, \Sigma 17$ for <100> rotation. Actually we observed high frequency of the coincidence boundaries with the $\Sigma$ values mentioned above, except $\Sigma 17$. Lower $\Sigma$ coincidence boundaries occurred more frequently in ascending order of $\Sigma$ value, probably associated with their boundary energy.

Finally it should be mentioned that in Fig.10 the straight lines for iron and its bcc alloys and the random grain orientation distribution merge at a point around $\Sigma^{-1/3}=0.3$ which corresponds to $\Sigma 37$. This may imply that it would be possible to increase the frequency only for the coincidence boundaries with $\Sigma$ values smaller than 37. In other words, the coincidence boundaries with $\Sigma 1 - \Sigma 37$ may be regarded as low-energy.

In the present author’s opinion, this may be a useful criterion for the upper bound of $\Sigma$ value which is physically meaningful. We can expect that such quantitative description of GBCD particularly for low-energy boundaries will enable us to predict GBCD in relation to the method and condition of material fabrication and material itself.

4.3 Comments on Effects of Grain Size and Texture - The present author would like to make comments on the effects of grain size and texture on mechanical properties of polycrystals from the view point of GBCD. The mechanical properties of polycrystals may be more affected by grain boundaries than presently expected. More recently Grabski and coworker have launched a new approach to grain size effect on the flow stress in polycrystals in the light of recent knowledge of GBCD (44,45). High strength and high ductility achieved by grain refinement are likely related to the presence of a high frequency of low-energy boundaries. The effect of texture should also receive some reconsideration into the relationship to the GBCD in textured polycrystals since high incidence of low-angle boundaries and coincidence boundaries often appears in strongly textured polycrystalline materials.

As to theoretical back ground of high frequency of low-energy boundaries in fine-grained polycrystals, as mentioned before, Watanabe (41) has shown that the boundary energy must increase during grain growth in recrystallization process because of the requirement of geometrical arrangement of grain boundaries bordering growing grain. It is expected that fine-grained polycrystal which normally experienced low temperature annealing or only the early stage of recrystallization, may contain more special low-energy boundaries. Conversely the presence of a high frequency of high energy random boundaries may explain low ductility associated with intergranular fracture normally observed in coarse-grained polycrystals and the premature intergranular fracture associated with abnormally grown grain in fine-grained polycrystals.

5. Grain Boundary Design for Desirable Mechanical Properties.

5.1 The Control of GBCD.

We have shown that GBCD is the most important microstructural parameter in discussing the relationship between microstructure and properties of polycrystalline materials. This section discusses feasible ways how to endow polycrystalline solids with desirable mechanical properties by grain boundary design (5,36,37).

Here we consider the possibility of improvement in ductility by suppression of intergranular fracture. Our first approach by grain boundary design is to increase the frequency of low-energy boundaries which have been evidenced to be resistant to fracture in any stress condition and environment. Figure 11 schematically shows possible fracture processes occurring in model polycrystals with hexagonal network of low-energy boundaries (denoted by $\Sigma$) and high-energy boundaries (denoted by $R$) with varying fraction (47). We assume that only high-energy random boundaries can break and not at all on low-energy boundaries.

Under normal stress exceeding the intrinsic intergranular fracture stress, cracks are formed at random boundaries. When the fraction of random boundaries exceeds 2/3, intergranular cracks can propagate to neighbouring random boundaries resulting in typical intergranular brittle fracture both at low temperature (Fig.11A) and high temperature (Fig.11B). However, when the fraction of random boundaries is reduced to 1/3, the premature cracks formed at random boundaries perpendicular to stress axis cannot propagate further to neighbouring boundaries (Fig.11C). Accordingly intergranular fracture may be controllable by lowering the frequency of random boundaries.
or conversely by increasing the frequency of low-energy boundaries. From presently available experimental observations, polycrystals which have the frequency of low-energy boundaries exceeding about 40% appear to show high ductility. However this needs to be confirmed by more quantitative experiments. Partial digeneration of periodic arrangement of low-energy boundaries and high-energy boundaries may allow premature cracks to propagate within the region of digeneration as shown in Fig.11D. In view of this, some regular arrangement of low-energy boundaries and high-energy boundaries should be considered for suppression of intergranular fracture by controlling GBCD.

5.2 The Control of Grain Boundary Inclination.

As far as mechanical properties is concerned, the control of boundary inclination is considered effective for controlling intergranular deformation and fracture at particular boundary geometry to the applied stress. Intergranular fracture can occur most feasibly at random boundary perpendicular to the stress axis under the maximum normal stress. High temperature deformation and fracture associated with sliding will occur most preferably at random boundary lying at 45° to the stress axis. Thus, the inclination of grain boundary is one of factors controlling mechanical properties of polycrystals. Let us consider a special case where grain boundaries are lying along specific direction(s), for instance along rolling direction as often seen in textured polycrystals. In such case we can expect a strong anisotropy of intergranular deformation or fracture, again depending on GBCD in the materials.

As is shown by Fig.11E, when random boundaries border long sides of elongated grains and are lying more perpendicular to the stress axis, intergranular fracture would take place more dominantly at the fraction of random boundaries of 2/3. However by choosing the stress direction along the axis of elongated grain, intergranular fracture may be suppressed (Fig.11F). Thus it seems possible to suppress intergranular fracture by controlling the boundary inclination and GBCD. Combined effects of GBCD and the boundary inclination may enhance the anisotropy of intergranular deformation and fracture. The anisotropy of mechanical properties should be effectively utilized in the design for desirable mechanical properties of polycrystalline materials.

6. Conclusion- Recent knowledge of structure-dependent mechanical properties of grain boundaries can be utilized for the design and prediction of desirable bulk mechanical properties of polycrystalline solids by introducing a new microstructural parameter, i.e. grain boundary character distribution GBCD. The GBCD is considered the most important microstructural parameter controlling the bulk mechanical properties of polycrystalline solids.

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