The Sphere Rotation Method: Determination of Low Energy Orientation Relationships by an X-Ray Texture Analysis

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Dedicated to the memory of Professor Günter Wassermann

Sphere rotation experiments are reviewed, with emphasis on the latest results. The method and its evaluation are described in detail. The sphere rotation method has been applied so far to study the correlation between structure and energy of homo- and heterophase boundaries, also to study the relation of temperature, pressure, mobility, corrosivity, and embrittlement to the energy of grain (=homophase) boundaries. The material specific properties, especially the electronic structure, influence the number of low energy orientations to a great extent. All current criteria developed for the explanation of low energy boundary orientations do not fit the observations in a unique way. The common characteristics of hetero- and homophase boundaries are represented.

KEY WORDS: Grain boundaries, phase boundaries, low energy boundaries, sphere rotation method, pole figure measurements, corrosion, embrittlement, electronic structure.

INTRODUCTION

In the past ten years the sphere rotation method has proven to be a suitable means of determining low energy orientation relationships of phase and grain boundaries as a function of many parameters (such as pressure, temperature, bonding type etc.). These investigations have led to a fundamental understanding of the behaviour of

such boundaries. In the present study, measurements of this kind are presented and discussed critically. In this contribution the latest results will be emphasized, especially those describing the influence of the electronic structure on the behaviour of boundaries

METHOD

The sphere rotation effect was first suggested by Shewmon (1966) for diffusion measurements. When two single crystalline spheres are sintered together, an interface between both spheres is formed. Comparable diffusion processes develop by sintering a sphere on a plate (Figure 1a). The interfacial energy depends on the orientation relationships between the two crystals (Figure 1b). By annealing the specimen, the two crystals rotate relative to one another into an orientation of low energy. In this position, the sphere has achieved a state of local equilibrium. In this form, the technique was not easy to apply because it required the measurement of many orientation relationships. Herrmann *et al.* (1976) opened the way for utilizing the sphere rotation effect by using a large number of spheres in contact with a flat single crystal plate (Figure 2). This kind of experiment is known as sphere-rotation method or sphere/plate method or rotating-sphere-on-a-plate method. When two spheres

Figure 1a Schematic diagram showing a boundary (AB) formed by sintering a single crystal ball (b) onto a mono crystal plate (p). The crystal lattices of the ball and the plate are misorientated with respect to one another by an angle θ^* . The atomic flux indicated by the arrows results in an anti-clockwise rotation of the sphere so that the misorientation angle changes from θ^* to θ_0 (Fig. 1b) (from Herrmann *et al.*, 1976).

Figure 1b Schematic curve of the grain boundary energy per unit area versus tilt angle. A boundary of low energy is obtained for the orientation relationship corresponding to the tilt angle θ_0 (from Herrmann *et al.*, 1976).

relative to one another are moved, all five degrees of freedom of the boundary are changed. In contrast to this, a fixed plate restricts the sphere rotation to three degrees of freedom. Despite this restriction, the advantages of measurement are prevailing.

At the beginning of the annealing time, a statistical distribution of orientations of the spheres on the plate can be observed. During subsequent annealing, the spheres rotate and, after the rotation has finished, the total spectrum of low energy orientation relationships can be measured (Figure 3). X-ray texture analysis was used to determine the distribution of the orientations of the spheres. To a first approximation, the intensity of the diffracted X-ray reflections

Figure 2a Scanning electron micrograph showing the lateral arrangement of the spheres on the plate (from Herrmann *et al.*, 1976).

Figure 2b Scanning electron micrograph of a sphere sintered onto a monocrystal plate (from Herrmann *et al.*, 1976).

is proportional to the number of spheres rotated into an orientation relationship of low energy and thus, proportional to the width (catchment area) of the corresponding energy cusp (Goodhew and Allen, 1977). For grain boundaries it can be assumed that the width of a cusp is nearly proportional to its depth (in a boundary energy vs. lattice misorientation plot) (Goodhew, 1980). This is correct if the shapes of all cusps are such that they have equal slopes. Since the slope is determined by the number of dislocations which have to be introduced into the boundary in order to create a given misorientation increment, the above mentioned proportionality between width and depth of a cusp seems to be justified according to a first rough approximation. The same assumption should also be valid for phase boundaries.

If the number of reflections in the pole figure is small, those reflections belonging to each other and defining a special orientation relationship can be determined in a straight forward manner. This is no longer true if the reflections superpose and/or if more than one reflection possess the crystallographically required angles. Individual reflections can be combined in a way producing fictitious misorientations. Pole figures which can be indexed unambiguously are generally observed for low energy orientations of interphase boundaries, contrary to the pole figures of many grain boundaries.

Maurer (1987) used Cu spheres on a Cu plate to show that pole figures with these complications can be evaluated. It was taken into

Figure 3 Sections through the {111} pole figure of a Cu/Cu specimen as a function of the annealing time. (a) a (100) plate without spheres; (b) a specimen annealed at 1030°C for 15 min (random orientations); (c) for 6 h, (d) for 12 h, and (e) for 48 h (finished rotation).

consideration that some reflections are superimposed. The ambiguity of overlapping reflections was reduced by evaluating the intensity of the reflections. The orientation distribution was determined by analysing the measured X-ray $\{111\}$ pole figures (Cu- K_{α} -radiation). This analysis involved calculating the pole figure for various assumed preferred misorientations and comparing the results with the measured pole figure. This process was continued until reasonable, self-consistent results were obtained. By this technique, a number of boundaries were detected which were only weakly represented in the pole figure. Nevertheless, the method for the unequivocal evaluation of these complex pole figures remains to be improved. An approximate method of tackling this problem is to evaluate the highest intensities of the pole figures only (Lojkowski *et al.*, 1987). With this simplification, however, it is possible to disregard important low energy orientations.

Recently, studies have been in progress to apply modern method of texture analysis (ODF-method) to the problem (Bunge and Gleiter, 1987).

GRAIN BOUNDARIES

Herrmann *et al.* (1976) used this method for the first time to investigate the orientation relationships of grain boundaries of low energy in Ag and Cu, respectively. Both materials were found to exhibit different types of low energy boundaries. This result was interpreted in terms of different electronic structures. Ag and Cu possess the same crystallographic structure but a different band structure. The particular low energy orientation relationships, identical in both systems, were defined as electronic-insensitive grain boundaries. The other ones, being system specific, were called electronic-sensitive grain boundaries.

Sautter *et al.* (1977) extend this interpretation to the grain boundaries of Ag-Au and Ag-Bi alloys. A later study by Maurer and Gleiter (1985) deals with the degree of influence of the electronic structure on the number of low energy orientation relationships in Ni-Cu alloys. The results of these investigations will be discussed more extensively in a separate section.

The evaluation of the pole figures obtained by means of sphere-

Figure 4 $\{111\}$ Pole figure of the peak positions theoretically expected by the coincidence model for a (111) orientated plate (from Maurer and Gleiter, 1986).

rotation experiments suggests that a coincidence orientation relationship is not necessarily a prerequisite to low energy orientations (Lojkowski and Gleiter, 1985; Maurer and Gleiter, 1986).

Figure 4 is an example for a calculated pole figure assuming that the energy geometrically depends on the 3-dimensional density of coincidence sites (after data from Mykura, (1980)). Figure 5 shows an experimentally observed pole figure. A comparison reveals that the observed number of low energy orientations is smaller than expected. Based on the detailed evaluation of data of rotated Cu spheres on a (100) Cu plate (Maurer, 1987), Sutton and Balluffi (1987) pointed out that there exists no obvious correlation between measured orientation relationships of low energy boundaries and current geometric criteria which have been proposed in the litera-

Figure 5 $\{111\}$ Pole figure of the peak positions measured for a (111) Ag-plate with Ag spheres annealed at 950° for 450 h (from Maurer and Gleiter, 1986).

ture as coincidence site density, grain boundary periodicity, or 0-lattice site density.

The authors confirmed this observation in discussing geometric criteria and comparing them with available experimental results indicating boundaries of relatively low energy.

The low energy boundaries identified by Lojkowski *et al.* (1987) in Ag and Cu can be interpreted on the basis that they form facets. The facets contain densely packed structural units. However, little correlation was found between these structural units and simple existing geometric criteria for low energy. These structural units are composed of symmetrical tilt boundaries of short periodicity or are created by joining together a $\{111\}$ surface with a stepped $\{100\}$ surface in such a way that the ledges on the surface of one grain are

locked in the surface of the other grain. The effect of the crystallographic orientation of the grain boundary plane on the grain boundary energy is as important as the effect of the misorientation angle. As only the highest intensities are taken into account, no difference was observed as far as the crystallographic parameters of low energy boundaries in copper, silver and a Cu-0.1% Bi alloy at temperatures above $0.95T_m$ are concerned.

As expected from Gibbs' equation, $G = H + pV - TS$, the free energy of a boundary depends on temperature and pressure. Hence, the low energy orientation relationships should be pressure- and temperature-sensitive. In fact, investigations of low energy boundaries in Ag by Meiser and Gleiter (1980) indicate that some special orientations existing at a high pressure were not observed at ambient pressure and vice versa (Figure 6). Also, using the sphere rotation method, Erb and Gleiter (1979) proved that the number of low energy orientations decreases as the boundary entropy is enhanced due to an increase in temperature (Figure 7).

In a study by Lojkowski *et al.* (1987), low energy orientation

Figure 6 Orientations of low energy in silver at different pressures. The orientation relationships that are dependent on pressure are marked by arrows (from Meiser and Gleiter, 1980).

Figure 7 Orientations of the low energy boundaries in copper as a function of the temperature. Some of the special orientation relationships are independent of the temperature. Four of those are marked by arrows in the section at 1050°C. The peak marked in the recording at 750°C is not present at 1050°C (from Erb and Gleiter, 1979).

relationships were analysed in detail for Cu and Ag. At temperatures close to the melting point ($0.8-0.996T_m$), $[110]$ tilt boundaries were identified as being associated with those energy minima which exhibited the largest catchment areas for rotating spheres independent of the plate orientation. Within the resolving power, up to $0.8T_m$, no preferred tilt angles of the $[110]$ tilt boundaries can be detected. At higher temperatures, a few special tilt angles are favoured.

If the sphere-rotation method is slightly modified, other properties such as the boundary mobility, corrosivity and boundary

embrittlement can be investigated and correlated with the boundary structure. For example, Wirth and Gleiter (1981) studied the dependence of boundary mobility on boundary energy in the system Cu-5 at.% Ag.

The migration of boundaries was caused by the discontinuous precipitation reaction of the supersaturated solid solution. With slight undercooling, discontinuous precipitation is not initiated at low energy boundaries. With increasing undercooling, a growing fraction of low energy boundaries is able to become a discontinuous reaction front. During the discontinuous precipitation reaction, the diffusion coefficient in the interfaces is enhanced by a factor of about 10^3 (consistent with data reported in the literature).

Therefore, it is assumed that a static grain boundary and a discontinuous reaction front have different atomic structures created by a structural transformation.

A further field of research of the sphere rotation method comprises investigations concerning the dependence of the corrosion sensitivity of the interfaces on the boundary misorientations (Erb *et al.*, 1982). These studies were carried out by putting the specimen into a solution which preferably corroded the grain

Figure 8 (a) Schematic diagram of the specimens used for the corrosion experiments and (b) the process of removing individual spheres by boundary corrosion (from Erb *et al.*, 1982).

Figure 9 Comparison between identical sections of the $\{111\}$ texture diagrams of two specimens. The specimen of Figure 9a was annealed for 50 h at 1050°C (without any corrosion) to rotate all the spheres into the orientation relationships of minimum energy. The specimen of Fig. 9b contained randomly oriented spheres and was corroded for 40 h (from Erb *et al.*, 1982).

boundaries (Figure 8). If the entire boundary region between a sphere and the plate is dissolved, the corresponding sphere falls off. Those spheres whose boundaries have the highest (lowest) corrosion sensitivity are the first (last) to fall off. The orientation relationships of those spheres with the highest and the lowest corrosion sensitivity were determined by texture analysis. The pole figures obtained were compared with pole figures corresponding to boundaries of low energy (sphere rotation experiments) (Figure 9). In the case of grain boundaries in Cu, the comparison of the two pole figures led to the conclusion that boundaries of low energy are least sensitive to corrosion. In the texture diagrams, the reflections of the spheres after corrosion (without rotation) are sharper than those after rotation (without corrosion). This difference is due to the fact that it is not the energy of the grain boundaries *per se* which defines the corrosion sensitivity in the boundaries but the dislocation structure in the boundary. This was confirmed by microscopic investigations.

Maurer *et al.* (1984) observed no enhanced corrosion in the crystal lattice in the vicinity of grain boundaries under conditions where the corrosive medium was not in contact with the actual grain boundary. For this experiment, a sphere rotation specimen was used (Figure 10): Plate 1 with the spheres 2 was coated from the

Figure 10 Schematic cross section through the experimental arrangement used in the work reported by Maurer *et al.* (1984): 1, single-crystal plate; 2, single-crystal sphere; *p*, corrosion protective layer; *s*, corrosive aqueous solution; *A*, corroded area; *t*, thickness of crystal 1.

“boundary side” with a corrosion protective layer, *p*, and was subsequently immersed into a suitable aqueous solution which was known to result in intergranular corrosion. No selective corrosion in the area, *A*, at the “other side” of a grain boundary was observed irrespective of the alloy composition, the boundary structure, the cooling rate, and the distance, *t*, of the boundary beneath the free surface exposed to the corrosion medium. The minimum spacing, *t'*, applied was less than $1\ \mu\text{m}$. The experimentally observed width, *d*, of the boundary region which is dissolved selectively during intergranular corrosion is of the order of $20\text{--}45\ \mu\text{m}$ and hence much larger than the minimum spacing, *t'*. Therefore, it was concluded that the width of the intergranular corrosion region is not due to an enhancement of the free energy of the lattice region in the vicinity of a grain boundary. The direct contact between the corrosive agent and the core of a boundary seems to be required to initiate intergranular corrosion.

In an experiment similar to that of Erb and Gleiter (1982), Roy *et al.* (1982) investigated the embrittlement of the grain boundaries by solute segregation to the boundaries. Those spheres breaking in a brittle manner along the grain boundaries fall off when the plate with spheres is vibrated ultrasonically. Low energy boundaries were found to exhibit the lowest brittleness. These results were interpreted in terms of preferred solute segregation.

This interpretation was confirmed by measurements of the oxygen

segregation at grain boundaries in Ag (Schuh *et al.*, 1986). During annealing in a hydrogen atmosphere, bubbles of water steam were formed in the grain boundaries due to a reaction of hydrogen with the oxygen segregated at the boundaries. The total volume of the bubbles in low energy boundaries was smaller than that in high energy boundaries.

THE EFFECT OF THE ELECTRONIC STRUCTURE ON THE BEHAVIOUR OF GRAIN BOUNDARIES IN METALS

Most of the models dealing with the structure of grain boundaries are of a crystallographic nature and neglect electronic effects. However, several observations (Herrmann *et al.*, 1976; Ottawa and Ishida, 1977; Liebert and Thomas, 1984) suggest that in metals conduction electrons may be of significance for the behaviour of grain boundaries. The same conclusion has been reached theoretically by Seeger and Schottky (1959) who utilized a free electron gas model to calculate the energy of high angle boundaries. The fundamental idea of their calculation is that the packing density of the atoms in a grain boundary is lower than in the perfect lattice. This causes a larger average distance between the atoms in the boundary. Hence, a deficit of the charge density develops in the boundary region. The low positive charge density is screened by a local rearrangement of the conduction electrons. For the metals silver and copper, the calculated boundary energy agrees reasonably well with the experimental data. It was therefore concluded that the electronic contribution is responsible for the larger fraction of the grain boundary energy.

In order to assess the significance of the electronic structure on the energy of grain boundaries, systematic experiments on the low energy boundaries in pure Cu (99,99%), 67 at.% Cu 33 at.% Ni, 33 at.% Cu 67 at.% Ni, pure Ni (99,99%), and 40 at.% Ni 60 at.% Co were carried out by applying the sphere rotation method (Maurer and Gleiter, 1985). As Cu–Ni forms solid solutions at all concentrations and as the lattice structure of all Ni–Cu alloys is fcc with little variation (2.53%) of the lattice constant, Cu–Ni-alloys provide a model system in which the electronic structure can be continuously varied from a transition metal to a noble metal without changing the

Figure 11 Density of states $g(E)$ in the $3d$ and $4s$ band as a function of the electron energy E . The arrows indicate the Fermi energy for (a) Ni, (b) Ni-33 at.% Cu, (c) Ni-66 at.% Cu, (d) Cu (from Schulze, 1977).

crystallography. The $4s$ and the $3d$ bands of Cu and Ni have similar shapes. Both bands overlap (Figure 11). In alloys of Cu and Ni, the $4s$ electrons of Cu enter the empty energy levels of the $3d$ band of Ni. In other words, alloying Cu and Ni results primarily in a continuous shift of the Fermi energy from the level of pure Ni to the value of pure Cu.

The experiments show that the number of low energy orientation relationships constantly decreases with increasing Ni contents. For pure Ni (above and below the Curie temperature) only $\Sigma 3$ boundaries were observed as low energy orientations. However, in pure Cu, about 150 low energy orientations were found (Figure 12).

Figure 12 Decrease of the number of low energy boundaries with increasing Ni contents (from Maurer and Gleiter, 1986).

No correlation between the Σ -value and the increasing number of special boundaries was noticed. As the lattice structure and the lattice constants of Cu–Ni-alloys remain unaffected when the chemical composition is varied, it is difficult to interpret the observed variation of the number of low energy boundaries in terms of a crystallographic model. The decreasing number of low energy orientations in Ni and Ni-alloys may, however, be accounted for by electronic effects. In fact, the observations seem to suggest that the electronic structure controls the energy of grain boundaries to a large extent. This conclusion agrees with the results obtained for a NiCo alloy. Ni and Co (although chemically different) have comparable band structures. Indeed, little change of the boundary behaviour was noticed.

Two conclusions may be drawn from these observations: (i) Crystallographic parameters, such as coincidence site density, grain boundary periodicity, O-lattice site density etc. are not fully adequate to characterize the behaviour of boundaries. As the crystal lattices of all alloys investigated are identical, any type of crystallographic criterion would be the same for all alloys and, therefore, the same boundary behaviour would be expected. (ii) Computer simulations, based on pair-wise interaction potentials, do not seem to be entirely adequate to model the experimental observations reported, in particular the disappearance of all but one type of special grain boundaries ($\Sigma = 3$) in Ni and Ni-60 at % Co. No matter which types of potentials are used to model the band structure change in Cu–Ni alloys, grain boundary energy computations always indicate the existence of several low energy boundaries in pure Ni, pure Cu, and Cu–Ni alloys. In order to account for this discrepancy between calculations and experimental observations, the equation for the boundary energy, E , in the computations should consist of, at least, two terms: one term, $F(n(\mathbf{r}))$, describing the electronic energy contribution of the system which only depends on the electron density and a structure dependent second term so that $E = F(n(\mathbf{r})) + 1/2 \sum_{ij} V_{ij}$. Yet, the computations of the structure of grain boundaries published so far are exclusively based on the second term and result in predictions that are not in agreement with the experiments. This conclusion seems to be confirmed by measurements of the elastic constants of grain boundaries in metals (Korn *et al.*, 1987). Young's modulus E and the shear modulus G of

ultra-fine grained materials (Pd, 6 nm and Mg, 12 nm) which consist of about 30 to 50 vol % of grain boundaries are comparable to those of conventional materials. The obtained results suggest that the interatomic potentials in grain boundaries differ from the lattice potentials.

PHASE BOUNDARIES

Erb *et al.* (1982) were the first to extend the sphere rotation method to the investigation of the boundary between two different materials in the example of a NaCl-plate with Ag spheres. In a later study (Shirokoff and Erb, 1986), another three orientation relationships were found besides the two orientation types already known, due to an improved method. By means of the sphere-rotation-method, the orientation relationships observed in experiments with epitaxially grown layers (Matthews, 1965) were confirmed to be low energy orientations. However, the sphere-rotation-method offers better statistics than the expitaxial methods and thus, further low energy orientation relationships can be found.

By using the sphere-rotation method, Fecht and Gleiter (1985) investigated low energy interphase boundaries between different metal/ionic crystals. Their result confirmed Erb's finding that small

Figure 13 "Lock-in" between an Au- and a LiF-crystal when close packed $[110]$ -rows of Au atoms "lock" into every third valley of the " (001) -surface" of the LiF crystal (from Fecht and Gleiter, 1985).

lattice mismatch in the boundary is not the criterion controlling the energy of the boundaries as was expected according to the near CSL-model (Balluffi *et al.*, 1982). In order to explain the experimental data obtained for a variety of different types of interphase boundaries, the lock-in-model was proposed. According to this model, a low energy interphase boundary is achieved if densely packed rows of atoms at the "surface" of the metal lock into the valleys between densely packed atom rows of the "surface" of the ionic crystal (Figure 13). The higher the packing density of the rows of atoms in lock-in configuration, the lower the energy of an interphase boundary. The correlated lattice strain is not discussed. It is expected that, during the rotation of the spheres, parameters such as temperature and the type of chemical bonding between sphere and plate are the crucial parameters for the interfacial energy.

In a paper discussing the low energy boundaries found in the Ag/Ni system (Maurer and Fischmeister, 1987), it is shown that none of the existing geometrical models, such as the near CSL model or the lock-in-model, give an explanation of the observed orientation relationships. A comparison of these results with other sphere rotation studies reveals considerable similarity between all

Figure 14 Example for the assumed reduction: micro-twinning and facetting of the $(11\bar{1})_{\text{Au}} \parallel (111)_{\text{LiF}}$, $[01\bar{1}]_{\text{Au}} \parallel [01\bar{1}]_{\text{LiF}}$ boundary to form cube-on-cube boundaries (from Maurer and Fischmeister, 1987a).

Table 1 Crystallographic characterization of the basic set of boundary orientation types

Nr.	Parallel planes	Parallel directions*
1.	cube-on-cube	
2.	$(111)_s \parallel (100)_p$	$[01\bar{1}]_s \parallel [01\bar{1}]_p$
3.	$(211)_s \parallel (100)_p$	$[01\bar{1}]_s \parallel [01\bar{1}]_p$
4.	$(111)_s \parallel (011)_p$	$[01\bar{1}]_s \parallel [01\bar{1}]_p$
5.	$(211)_s \parallel (011)_p$	$[01\bar{1}]_s \parallel [01\bar{1}]_p$
6.	$(011)_s \parallel (100)_p$	$[01\bar{1}]_s \parallel [01\bar{1}]_p$
7.	$(100)_s \parallel (100)_p$	$[01\bar{1}]_s \parallel [01\bar{1}]_p$
8.	$(111)_s \parallel (100)_p$	$[01\bar{1}]_s \parallel [01\bar{1}]_p$
9.	$(211)_s \parallel (100)_p$	$[01\bar{1}]_s \parallel [01\bar{1}]_p$
10.	$(111)_s \parallel (011)_p$	$[01\bar{1}]_s \parallel [100]_p$

* The subscripts *s* and *p* refer to planes and directions in the lattices of the spheres and plates.

systems studied so far, including metals on oxides and on alkali halides. Assuming that the boundaries across the necks can both facet and form occluded $\Sigma 3$ twins (Figure 14), it is possible to reduce all 33 orientation types to 10 “elementary” boundary types (Table 1). In almost all systems, cube-on-cube is the most dominant orientation.

All remaining observed boundary types can then be described as boundaries in which a twinning plane of one material is parallel to a low index plane of the other material. An analysis of the preferred “elementary” boundaries shows that they could be understood as coincidence boundaries with a considerably extended tolerance radius. It is suggested that such “extended coincidence” might not be unreasonable for pairs of materials with weak interphase bonding.

Latest studies show that the orientation distribution of small silver spheres annealed on amorphous silica substrates reveals two cusps in the interfacial energy vs. misorientation curve (Shirokoff and Erb, 1987). These cusps correspond to orientations for which close-packed and second close-packed planes lie parallel to the silica surface. They are the same preferred orientations as previously reported for silver films as grown from the vapour phase on glass substrates. By investigating the Ag/SiO_x/Si interfacial system, three orientation relationships of low energy were observed (Shirokoff *et al.*, 1987). Two of these are identical to the cusps found for the

Ag/bulk silica system. The third orientation relationship is the Ag/Si epitaxy orientation which is present even when an oxide layer of about 180 Å in thickness separates the two phases. It appears that long range ordering effects in this system exist over distances of at least 180 Å.

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