CHARACTERIZATION OF DISLOCATION BOUNDARY EVOLUTION WITH MONOCHROMATIC X-RAY DIFFRACTION

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ABSTRACT

Inhomogeneities of plastic deformation result in the formation of different types of dislocation boundaries. With respect to their origin and properties two main types are distinguished: incidental dislocation boundaries (IDBs) and geometrically necessary boundaries (GNBs). Both types of boundaries are connected with disorientations, which are lower across IDBs than across GNBs. Thick IDBs result in long-range internal stresses causing a broadening of monochromatic X-ray diffraction reflections both in radial direction of the diffraction vector and in orientation space. GNBs arise from differences in the activated slip systems on both sides of the boundary. Their diffracted intensity distribution becomes highly anisotropic in reciprocal space. The different functional behaviour of monochromatic intensity distributions for IDBs and GNBs can be used to track their evolution during plastic deformation.

1. INTRODUCTION

Deformation-induced boundaries have been classified according to morphological differences and their expected origin (Kuhlmann-Wilsdorf and Hansen 1991): cell walls are assumed to form by statistical mutual trapping of glide dislocations and have been termed Incidental Dislocation Boundaries. IDBs are usually thick and curved boundaries with a loose arrangement of the dislocations within the walls. Extended, straight, and parallel dislocation boundaries have been termed Geometrically Necessary Boundaries. GNBs have a dense dislocation population and are assumed to form by a different slip activity on each side of the boundary. The morphological differences indicate different mechanisms in the evolution of IDBs and GNBs. For example, the average values for the boundary spacings or the disorientation angles differ for IDBs and GNBs as well as their dependence on strain (e.g. Pantleon 2002).

2. DIFFRACTION BY CRYSTALS WITH DISLOCATION BOUNDARIES

The analysis of X-ray scattering by dislocation structures is based on the general kinematic treatment of scattering from defects (Krivoglaz 1996; Barabash and Krivoglaz 1982; Wilkens 1979). Results are summarized for different cases where the scattering intensity distribution is dominated by:

- dislocation cell walls (incidental dislocation boundaries)
  - with vanishing or non-vanishing disorientation angle across the boundary
  - with or without correlation between neighbouring boundaries or
- ideal low angle boundaries representing geometrically necessary boundaries.

In reciprocal space the exact positions of regular reflections hkl are related to the orientation and the spacing of the reciprocal lattice. The momentum transfer corresponding to an ideal Bragg or Laue reflection matches the reciprocal lattice vector $\mathbf{G}_{hkl} = \mathbf{k}_{hkl} - \mathbf{k}_0$ with an incident wave vector $\mathbf{k}_0$ and an ideal scattered wave vector $\mathbf{k}_{hkl}$ ($|\mathbf{k}_0| = |\mathbf{k}_{hkl}|$). The diffuse scattering intensity depends on the deviation $\mathbf{q} = \mathbf{Q} - \mathbf{G}_{\mathbf{hkl}} = \mathbf{k} - \mathbf{k}_{hkl}$ between the diffraction vector $\mathbf{Q} = \mathbf{k} - \mathbf{k}_0$ and $\mathbf{G}_{hkl}$. The intensity distribution

$$I(\mathbf{Q}) = \left| \sum_s f_s \exp(i\mathbf{Q} \cdot (\mathbf{R}_s^0 + \mathbf{u}_s)) \right|^2 \tag{1}$$

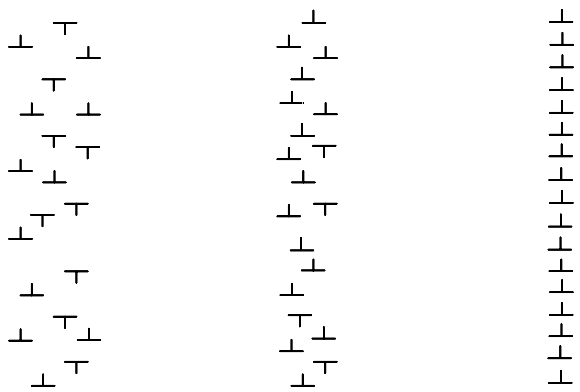
of the elastic scattering is a result of the contributions from all individual scattering cells s, where f_s is the scattering factor of an individual atom s at the relaxed position $\mathbf{R}_s = \mathbf{R}_s^0 + \mathbf{u}_s$ due to the presence of defects. The total displacement $\mathbf{u}_s = \Sigma_t c_t \mathbf{u}_{st}$ of the scattering unit cell s from its equilibrium position $\mathbf{R}_s^0$ is a superposition of the partial displacements $\mathbf{u}_{st}$ from all strain-inducing defects in the sample. The occupation factor c_t becomes 1, if there is a defect at position t, and 0 otherwise. Dislocations and dislocation boundaries practically do not change the scattering factors. Then the intensity of X-ray or neutron scattering can be re-written without any further assumptions or models for the defect arrangement as

$$I(\mathbf{Q}) = |f|^2 \sum_{s,s'} \exp\left(i\mathbf{Q} \cdot (\mathbf{R}_s^0 - \mathbf{R}_{s'}^0)\right) \exp\left(-T_{ss'}\right). \tag{2}$$

The first term $\exp(i\mathbf{Q} \cdot \mathbf{\Delta})$ describes the scattering by an ideal crystal. The influence of defects on scattering is represented by the second term

$$\exp\left(-T_{ss'}\right) = \prod_t \exp\left[i\mathbf{Q} \cdot \left(\mathbf{u}_{st} - \mathbf{u}_{s't}\right)\right]. \tag{3}$$

The function $T_{ss'}$ depends on the relative displacements $(\mathbf{u}_{st} - \mathbf{u}_{s't})$ between all possible atomic pairs s and s' contributing to the scattering. For crystals with dislocations and dislocation boundaries, T is determined by the details of the dislocation distribution and the morphology of the boundaries and becomes different for IDBs and GNBs. It also depends on the boundary spacing D, the wall thickness W, the arrangement of the dislocations in the wall, and their (mean) distance along the wall h.



(a) (b) (c)

Fig. 1: Scheme of investigated dislocation arrangements: (a) thick cell boundary (IDB) with almost vanishing disorientation, (b) IDB with significant disorientation and (c) low angle tilt boundary (GNB) without redundant dislocation content.

2.1 IDBs without (or rather small) disorientations. The formation of IDBs is a purely stochastic process (cf. Pantleon 2002): Glide dislocations trap each other statistically and form thick, loose cell walls with randomly distributed dislocations within the wall and almost equal numbers of dislocations of opposite sign ($+$ and $-$) of the Burgers vector within a cell wall. Passing dislocations are trapped with a capturing probability $P = D/\bar{\lambda}$ given by the mean free path $\bar{\lambda}$ of mobile dislocations.

The character of the stress and strain fields associated with the boundaries determines the diffracted intensity. The central part of the intensity distribution for IDBs with almost equal numbers of $+$ and $-$ dislocations around any reciprocal lattice point is described by a Gaussian function both in orientation space and in radial direction (parallel to the momentum transfer $\mathbf{G}_{hkl}$). If there is only one type of edge dislocations grouping into walls, the intensity distribution along the dislocation line vector is not altered compared to an undeformed crystal. The intensity distribution near a reciprocal lattice point has a disk shape in the plane perpendicular to the line vector of the dislocations. The full width at half maximum $\mathrm{FWHM}_{IDB} = (\mathbf{Q}\cdot\mathbf{b})\gamma\sqrt{nl/2}$ in this plane depends on the orientation of the Burgers vector relative to the diffraction vector and the total dislocation density n, an (implicitly defined) abbreviation $l \approx \ln\left(\sqrt{nl}L\right)$ and the effective size L of coherently (or semi-coherently) scattering regions. Compared with randomly distributed, individual dislocations the intensity distribution is contracted by a factor $\gamma = \sqrt{1-\ln(D/h)/2l}$ depending on the ratio $D/h = D^2 n$.

Owing to a possible spread in the positions of dislocations along a boundary, IDBs have a finite boundary width (cf. Fig. 1a). The width W of IDBs is larger then the average distance h between the dislocations within the wall. Such a relatively large width of boundaries causes an additional broadening at the tails of the intensity distribution

(Ungar, Mughrabi and Wilkens 1982). According to Wilkens (1979) contributions from the transition regions directly adjacent to the wall are significant only, if the distance between the boundaries D and the boundary width W are of the same order and $D \leq 5h$, but usually $D \gg 5h$ and the transition regions become unimportant.

2.2 IDBs with significant disorientations. The disorientation angle across dislocation boundaries increases with plastic strain due to statistical fluctuations and possible activation imbalances on both sides of a boundary (Pantleon 2002). A pure stochastic formation of dislocations as for IDBs leads to a square root dependence of the average (modulus of the) disorientation angle on plastic strain. With increasing strain, the numbers of positive and negative dislocations within an IDB become different, and the excess dislocation density nearly equals the total dislocation density. This results in a radical change in the intensity distribution, which might be used as an indication of the formation of disorientations. The intensity distribution has still a relatively narrow Gaussian shape along the momentum transfer (radial direction), but a much broader intensity distribution in the transversal direction (see next subsection).

Even in IDBs with certain disorientations, the dislocations are arranged in a statistical manner (cf. Fig 1b) giving rise to internal stresses. Even for an infinite dislocation wall with parallel identical dislocations (as a small angle tilt boundary) any small and uncorrelated disorder prevents cancellation of the periodic elastic field (Saada and Bouchaud 1993), thus allowing the long-range interaction to reappear as opposed to the case of a low angle tilt boundary with perfect equidistant dislocations.

2.3 IDBs with non-correlated disorientations. Diffraction by non-correlated IDBs results in a broad intensity distribution in the rocking direction (Barabash and Klimanek 1999). The transverse intensity distribution $I(q_\xi)$ of the rocking curve (related to the main axis ξ of the transverse plane perpendicular to the momentum transfer) has a shape close to a Gaussian function. The full width at half maximum of the rocking curve

$$\mathrm{FWHM}_{rock} = 2\sqrt{A_0}\sqrt{\frac{\pi L}{D}}\alpha Q \quad \text{with} \quad \frac{1}{\sqrt{A_0}} = \frac{1}{V}\int d\mathbf{R}_s^0 \sqrt{\frac{L}{A_{\xi\xi} R_s^0}} \tag{4}$$

becomes essentially proportional to the square root of the number N of equivalent boundaries within the coherently (or semi-coherently) scattering region of effective size L. The latter is determined by the beam size. The rocking contrast factor A_0 depends on the mutual orientation between the diffraction vector $\mathbf{Q}$, the rocking axis and the orientation of the boundary plane, and the disorientation (angle and axis) of the IDBs. If the disorientations through neighbouring IDBs and their positions are totally independent of each other, the mean disorientation angle $\langle|\theta_N|\rangle = \langle|\theta|\rangle\sqrt{N}$ across N boundaries increases proportionally to the square root of their number (cf. Fig. 2). The $\mathrm{FWHM}_{\mathrm{rock}}$ of the rocking curve increases with the square root of the beam size, following this increase with the number of boundaries taking part in the diffraction. If only one or two different types of IDBs are formed, the distribution in orientation space might be essentially anisotropic due to the contrast factor. It has a "long axis" direction and a "narrow axis" direction. In principle, this gives the possibility to determine the primary orientation of IDBs in the scattering volume.

2.4 IDBs with correlated disorientations. Disorientation angles in neighbouring IDBs are usually not independent (cf. Pantleon 2002). Dislocations of opposite Burgers vector corresponding to the same dislocation loop are not separated to infinity, but only to a finite distance, i.e. twice the mean free path of the mobile dislocations.

In the absence of macroscopic strain gradients in the crystal, disorientations cannot be cumulative over several boundaries and the disorientation across N boundaries levels off after an initial increase (cf. Fig. 2). The saturation value for the mean disorientation angle $\langle|\theta_\infty|\rangle$ depends on the average disorientation angle across a single boundary. For such a correlated arrangement of IDBs, the rotation field of a single IDB is screened to saturation level by surrounding IDBs on a length scale $2\bar{\lambda}$. The dependence of the rocking curve $\mathrm{FWHM}_{\mathrm{rock}}$ on the size of the beam will saturate at a value given by Eq. (4) with a correlation length r_{corr} replacing the effective size L of the coherently scattering region. This is in contrast to the case of non-correlated IDBs. The correlation length $r_{corr} \approx 2\bar{\lambda} = 2D/P$ for the disorientation angle across IDBs equals several cell diameters and is usually much less than the size of the beam. Such a saturation of the $\mathrm{FWHM}_{\mathrm{rock}}$ of rocking curves was observed experimentally for a deformed copper single crystal (Breuer, Klimanek and Pantleon 2000). Detailed analysis of such an experimental dependence gives the possibility to determine the correlation length for IDBs. However, due to the rocking contrast factor some boundaries may give zero (or very small) input into rocking curve and their broadening. Consequently, several reflections should be analysed to determine the contrast factor and the orientation of the IDBs.

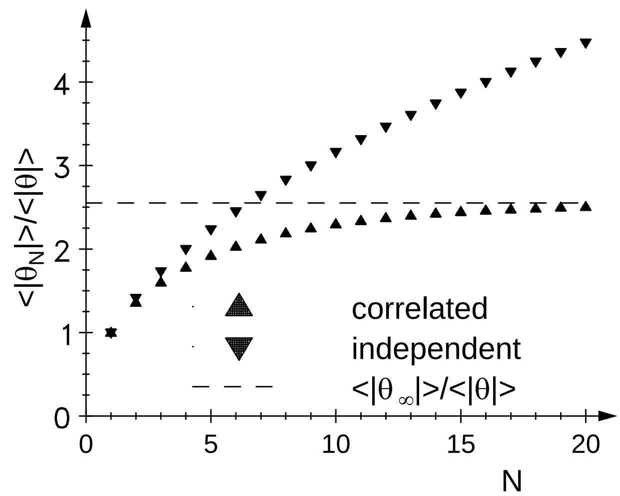


Fig.2: Modelled disorientation angles across N boundaries for independent and correlated disorientations with $P=1/3$ (Pantleon 2002).

2.5 GNBs. The elastic energy of dislocation boundaries is lowered when dislocations inside the boundary re-arrange, dislocations of opposite sign annihilate and the distance between the excess dislocations becomes constant along the boundary. Such ideal boundaries form very stable configurations. Planar geometrically necessary boundaries are much narrower than IDBs and good representations of ideal low angle boundaries. Pure tilt boundaries formed by equidistant edge dislocations (as in Fig. 1c) provide a rotation between the two adjacent regions, but do not contribute to any long-range strain. This leads to an important difference between IDBs and GNBs. The intensity distribution in radial direction due to thin and dense GNBs formed by equidistant edge dislocations is essentially different from the case of thick and loose IDBs with fluctuating distances between the dislocations in the wall. For crystals with GNBs the intensity distribution in the radial direction is close to a Lorentzian shape with a line width

$$2\delta\theta \approx \frac{\lambda\zeta}{2D}\sec\theta \tag{5}$$

and a geometrical factor ζ. Additionally, GNBs cause a broad intensity distribution in the plane transverse to the diffraction vector. The shape of the reflection in orientation space depends on the average orientation of the dislocation arrays and the diffraction vector. The intensity distribution in the rocking direction is close to a Gaussian shape and the $\mathrm{FWHM}_{\mathrm{rock}}$ given by Eq. (4) depends on the relative direction of rocking axis, diffraction vector and the dislocation arrangement within the boundary.

3. SUMMARY

The dislocation arrangement within boundaries influences the character of the internal strain fields and changes the condition for X-ray or neutron diffraction. Two main boundary types (IDBs and GNBs) are distinguished with respect to their origin, morphology and strain fields. The structural difference between IDBs and GNBs is reflected in the intensity profile both in the radial and rocking directions in reciprocal space. Correlations between disorientations across neighbouring boundaries result in a saturation of the dependence of the intensity distribution on the size of probed region.

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