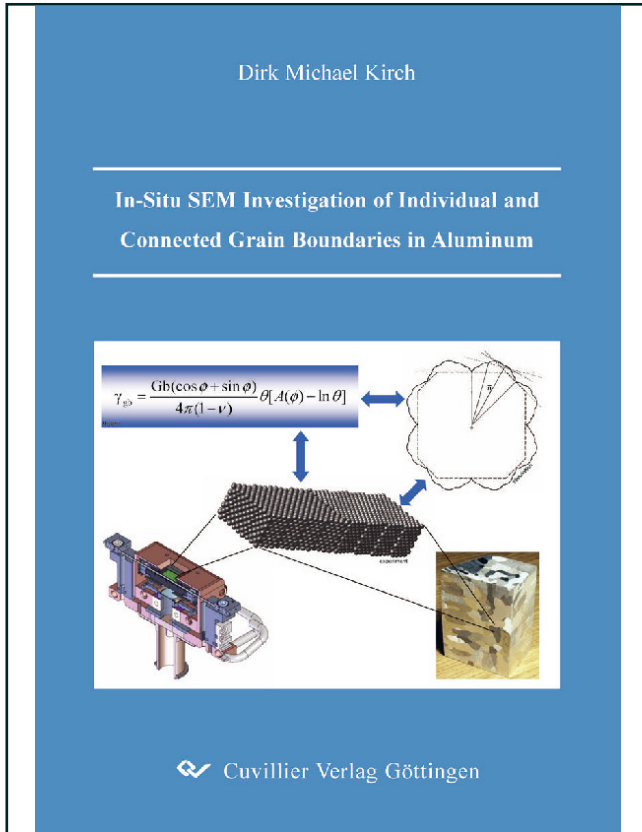




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**In-Situ SEM Investigation of Individual and
Connected Grain Boundaries in Aluminum**



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Chapter 1

Fundamentals of grain boundaries and triple junctions

1.1 Grain boundaries

The tremendous properties of crystalline metallic materials are based on their inherent defects. Without those crystal defects, this class of materials would not have their great success as functional and constructional materials. The broad spectrum of defects is mainly categorized by its dimension. One of the longest known and most important defects in metals is the grain boundary, a two-dimensional planar defect, which separates two crystal domains of the same crystallographic structure but of different orientation. Since any crystalline material, except for single crystals, is granular-structured in regions of different orientations, grain boundaries are the fundamental defect in polycrystalline materials and, therefore, tremendously influence their properties.

Although grain boundaries have already been known since the end of the 19th century [1], there is very little fundamental knowledge of grain boundaries due to their complex structure which requires advanced mathematical description tools.

1.1.1 Terminology

In a three-dimensional system, a grain boundary has eight degrees of freedom and therefore eight independent parameters are necessary to give

a mathematically exact definition of a grain boundary. Three parameters are required to define the orientation relationship between the two adjacent grains, which is commonly presented either by a Euler angle triplet $(\varphi_1, \Phi, \varphi_2)$, by Miller indices or by an angle-axis pair in the Rodrigues-Frank space [2, 3]. Since the last is most appropriate for this work, a short introduction will be given to the Frank-Rodrigues representation in one of the next sections.

Apart from the misorientation information; two parameters are required to determine the spatial orientation of the grain boundary plane, i.e. boundary inclination, by means of the normalized crystallographic normal vector of the plane of inclination $\mathbf{n} = (n_1, n_2, n_3)$ with respect to one of the adjacent grains. In addition to these five macroscopic parameters there are three independent microscopic values of the translational vector $\mathbf{t} = (t_1, t_2, t_3)$. Basically, all intrinsic properties of the grain boundary, in particular mobility and energy, are functions of these eight parameters. The five macroscopic parameters can be influenced externally, whereas the translational vector \mathbf{t} is determined by the crystal with respect to a minimum of the total energy.

To determine the dependency of the grain boundary properties, e.g. energy, on the five macroscopic parameters, it would be necessary to keep all but one degree of freedom constant and to systematically vary that free parameter. In realistic experimental setups only one set of parameters can be kept constant. The most common way of investigation is to change the orientation relationship in terms of a fixed axis of rotation and variation of the rotation angle under the constraints of a constant plane of inclination with respect to a reference point or vice versa.

However, in literature [4] it has been recently reported that grain boundary free energy distribution was measured as a function of all five macroscopic parameters, using a statistical approach, exploiting EBSD measurements [5].

Presentation of Misorientations

The orientation relationship between two crystal lattices, mostly referred to as misorientation, is a spatial transformation applied to one crystal in order to let both crystal lattices coincide. Assuming a common origin for both crystal lattices, this is a pure rotation of one crystal coordinate system with respect to the other and can be simply described by a rotation transformation matrix g^m :

$$\{C_i\} = g^m \{C_j\} \quad (1.1)$$

where $\{C_i\}$ and $\{C_j\}$ are the respective crystal coordinate systems.

The simplest way to describe such a rotation matrix is in terms of a rotation axis $\langle hkl \rangle$, which is common for both crystal coordinate systems and a rotation angle θ (Fig. 1.1), since it is in many instances of major interest to know the influence of the rotation angle on a grain boundary property for a fixed rotation axis. Therefore, it is desirable to keep the grain boundary inclination constant and consider only a rotation angle dependency.

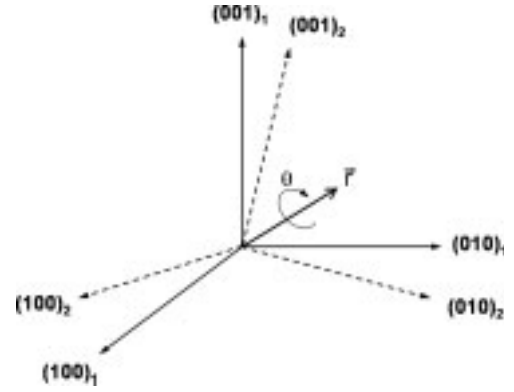


Figure 1.1: Rotation of θ about \vec{r} let crystal coordinate system 1 and crystal coordinate 2 coincide.

Based on this description, grain boundaries can be divided into three different classes. If the rotation axis is perpendicular to the grain boundary plane, the grain boundary is referred to as twist boundary (Fig. 1.2a). In this special case, the grain boundary plane is clearly defined and independent of the rotation angle. Figures 1.2b and 1.2c show the grain boundary class, which is called tilt boundary, and has its rotation axis within the grain boundary plane. For this class an infinite number of possible grain boundary planes for a given rotation angle exists. In the case of a mirror symmetry between the two adjacent grains the boundary is labeled as a symmetrical tilt grain boundary (Fig. 1.2b). All other configurations are referred to as asymmetrical tilt boundaries (Fig. 1.2c).

Grain boundaries which do not fulfill either of the criteria of the first two classes are termed mixed or random grain boundaries and consist of mixed twist and tilt components.

Rodrigues-Frank Space

When reducing the classification of grain boundaries to a pure orientation relationship it is convenient to use the Rodrigues-Frank notation, as already mentioned above [2, 3]. In that form of presentation the angle-axis designation of the rotation matrix g^m between two adjacent grains is expressed as a three-dimensional vector \vec{R} which combines the angle θ and axis \vec{r} of rotation into one mathematical entity:

$$\vec{R} = \tan(\theta/2)\vec{r} \quad (1.2)$$

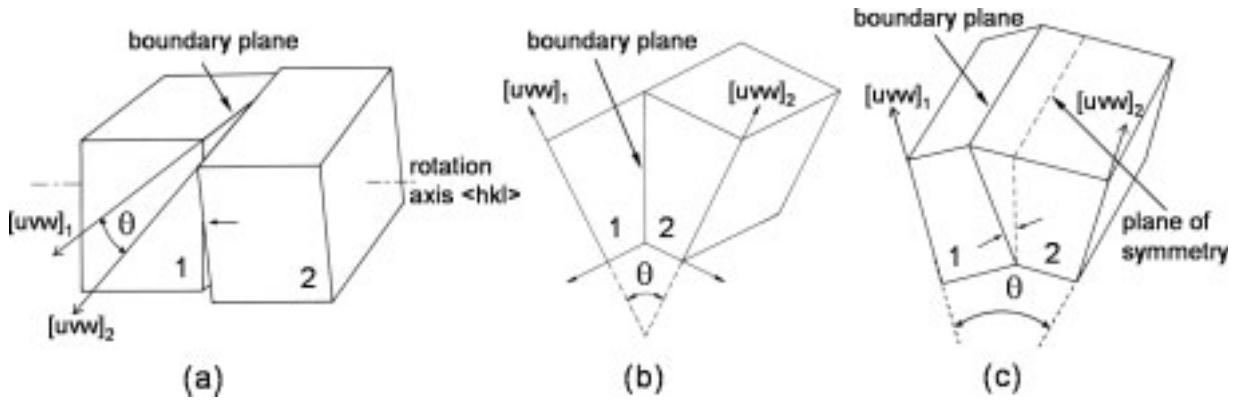


Figure 1.2: Various types of grain boundaries: (a) twist grain boundary, (b) symmetrical tilt grain boundary, (c) asymmetrical tilt grain boundary [6]

The great advantage of this parametrization is that \vec{R} defines a vector which lies in a Cartesian coordinate system whose axes correspond to the crystal axes. The three dimensional space in which such populations of R -vectors, corresponding to misorientations, are represented, is known as the Rodrigues-Frank (RF) space. Since \vec{R} is directly derived from the angle/axis pair, it can be described by a number of different crystallographic-related solutions, depending on the crystal symmetry. The length of \vec{R} is a function of θ and it is, thus, most convenient to choose that representative example of the crystallographic-related solutions which corresponds to the smallest misorientation and therefore lie closest to the origin. By using this smallest angle description the RF space can be reduced to a limited polyhedral zone around the origin containing all misorientations. This polyhedron is called the *fundamental zone of Rodrigues space* and any R -vector lying outside the fundamental zone can be re-expressed as an equivalent vector lying within the zone by re-choosing the crystallographic-related solution with the smallest angle.

The shape of the fundamental zone is fully governed by the crystal symmetry of the respective material. Fig. 1.3a shows the fundamental zone for cubic symmetry. For convenience, the axis of the zone is aligned with the crystal axis for the representation of misorientations. The fundamental zone itself can be further subdivided into 48 subvolumes, each containing only one representative for any crystallographic-related solution [8]. In the case of an orthorhombic sample geometry, which is used throughout this work, one eighth of the fundamental zone (Fig. 1.3b) already contains the full information of all possible misorientations.

The most powerful aspect of the Rodrigues parametrization is its recti-

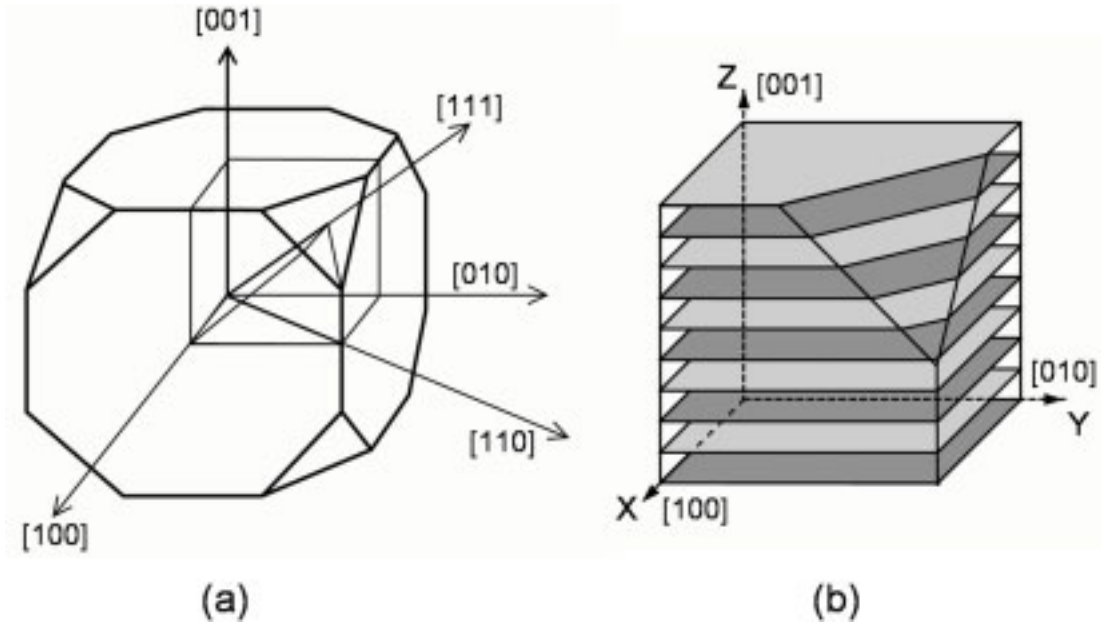


Figure 1.3: Fundamental zone of Rodrigues space for cubic symmetry (a); further reduction for orthorhombic sample geometry with sections through Rodrigues space perpendicular to the Z-axis (b) [7].

linear geometry, which simplifies its visualization and use:

- The axis of rotation manifests itself directly in the direction of \vec{R} . Hence, rotations about the same crystallographic axis lie on a straight line which intercepts the origin.
- The length of \vec{R} is given by the rotation angle. Thus, small angle grain boundaries cluster close to the origin.
- Orientations including common directions, e.g. fibre textures, lie on a straight line which does not necessarily intercept the origin. These *geodesic lines* remain straight even if the origin of the space is shifted [3, 9, 10].
- The edges of zones in RF space are straight lines, and the faces are planar.
- The RF space is *homochoric*, i.e. a random texture will be distributed uniformly throughout the space.

The features of the Rodrigues-Frank space above described are the essentials for the general understanding and use of it. A more detailed description and mathematical formalism can be found elsewhere [9, 10, 11, 12].

1.1.2 Atomic structure of grain boundaries

Apart from the classification with respect to the misorientation description of grain boundaries, it is most common to distinguish between small angle grain boundaries (SAGB) and large angle grain boundaries (LAGB) taking into account the difference in the internal microstructural configuration.

Small angle grain boundaries

If the angle of rotation between two adjacent grains is small enough (SAGB), the grain boundary is entirely composed of a periodic crystal dislocation arrangement. In the simple case of a symmetric $\langle 100 \rangle$ tilt boundary in a simple cubic crystal the configuration consists of a single set of edge dislocations with Burgers vector \vec{b} , where the dislocation spacing d is directly correlated to the rotation angle θ (Fig. 1.4a):

$$\frac{|\vec{b}|}{d} = 2 \sin \frac{\theta}{2} \quad (1.3)$$

With an increasing misorientation angle θ the spacing d decreases as shown in Fig. 1.4b.

In the more general case of an asymmetric tilt grain boundary (Fig. 1.2c) - rotation of the boundary plane out of its symmetric position by an inclination angle ϕ (Fig. 1.5b) - at least two sets of edge dislocations are required to describe the boundary structure. The Burgers vectors of these two dislocation sets must be perpendicular to each other (Fig. 1.5a) and with increasing deviation ϕ from the symmetrical position the fraction of the second set of dislocations (b_2) must increase,

$$\frac{1}{d_2} = \frac{|\vec{b}_2|}{\theta \sin \phi} \quad (1.4)$$

whereas the fraction of dislocations with Burgers vector b_1 decreases with ϕ

$$\frac{1}{d_1} = \frac{|\vec{b}_1|}{\theta \cos \phi} \quad (1.5)$$

Small angle twist boundaries require two sets of screw dislocations and in the most general case of mixed SAGBs the boundary structure is comprised of dislocation networks of three Burgers vectors [13].

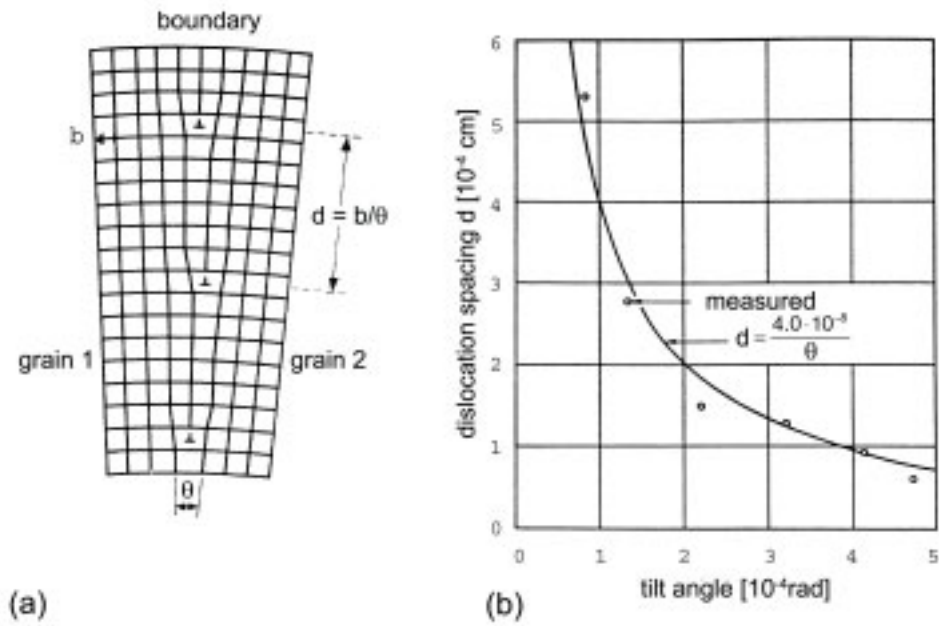


Figure 1.4: (a) Dislocation configuration of a symmetrical tilt $\langle 100 \rangle$ SAGB in a simple cubic crystal. (b) Measured and calculated dislocation spacing in a symmetrical SAGB in Germanium [6]

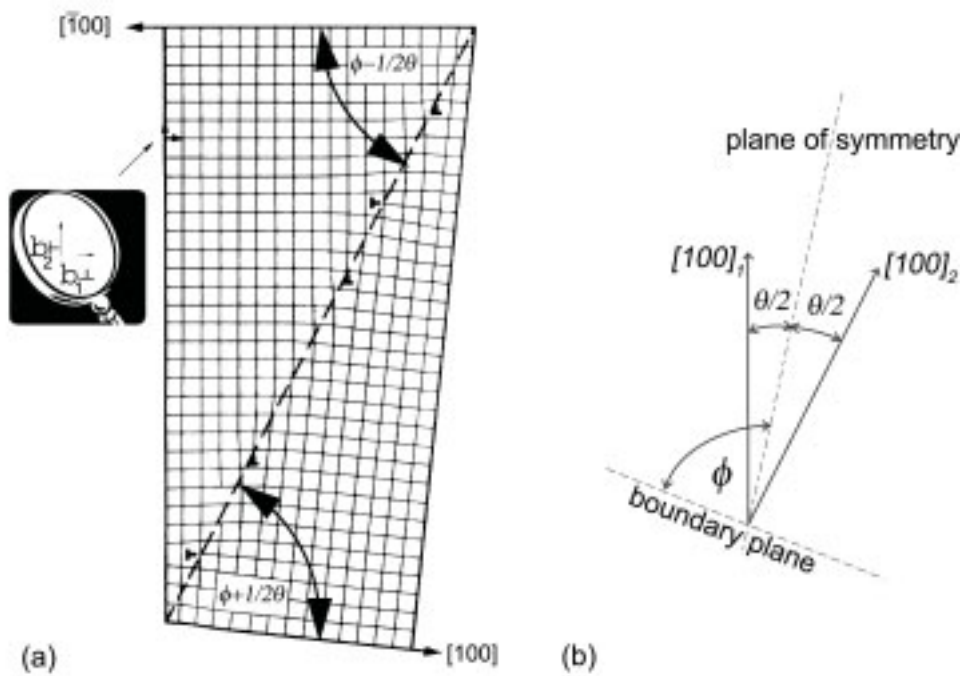


Figure 1.5: (a) Lattice dislocation arrangement [6] and (b) rotation angle θ and inclination ϕ of an asymmetric tilt SAGB [14]