Grain Boundary Motion under High Temperature Low Cycle Deformation

- Study of Mechanism, Kinetics and Driving force

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Abstract

The understanding of the mobility of grain boundaries in polycrystals as well as bicrystals constitutes an important aspect in materials design for high temperature services. Investigations of the motion of grain boundaries in single and two phase systems have merited significant attention because of their relevance to many technological processes. The extent of such movement controls the grain size, texture and redistribution of impurities and these changes, in turn, affect the mechanical behaviour as well as the electronic and magnetic properties of materials. The present study was performed to investigate the mechanism and kinetics of grain boundary motion during high temperature low cycle deformation (HT-LCF) of high purity aluminium bicrystals under two distinct conditions of cyclic deformation. The study has also been supplemented by microstructure and microtexture evolution.

The first part of the study emphasises the high temperature cyclic stress response of $<112>$ tilt grain boundaries where motion was caused by a plastic stress of cyclic deformation, which generates a difference in dislocation density of the two grains, these causes a driving force for boundary motion. For both low angle and high angle boundaries, a distinct evidence of irregular motion has been observed. In particular, the differences in dislocation density on both sides of the grain boundary has been found to be responsible for the motion of boundaries. For low angle grain boundaries, the movement of grain boundaries was not possible as long as the misorientation angle of the boundary was below a critical range of 13.8°. Grain boundaries with misorientation angle above 13.8° were found to be mobile under cyclic deformation conditions. The differences of slip density ($\Delta f_i$) and Schmid factors between the two grains of the deformed structures of bicrystal were observed most accountable for indexing of driving force of cyclic motion of grain boundaries. The effect of cyclic stress was relatively more clear and comparable. Larger stress reduces the chance for grain boundary motion due to homogeneous deformation structure in the two grains. The comparison of this study with grain boundary behaviour in polycrystals has also been discussed.
The second part of this work includes the mechanism and kinetic study of grain boundary motion under completely elastic amplitude of stress at elevated temperatures, which permits an estimation of the activation parameters for such cyclic motion. For this purpose, bicrystals with symmetrical <112> and <100> tilt and <100> twist grain boundary were grown with a range of misorientation angles 7° to 42°. These grown crystals were cut with different geometry (inclined at angles 0°, 15°, 30°, 45°, 60°, 75° and 90° with respect to the stress axis) where, all the samples were cyclically deformed under an elastic amplitude of stress. The nature of <112> and <100> tilt grain boundary displacement was found different for different geometries. The displacement marks of grain boundary during deformation were clearly visible on the surface of the bicrystals for perpendicular geometry, whereas, for other inclined boundaries, markings were not found so prominently. At the same time, grain boundary motion of <100> twist boundaries were completely different in nature as compared to the tilt boundaries. The overall boundary displacement was very much scattered along the grain boundary with some kind of bulging and snaky motion.

Activation energies were calculated from the slope of the plot of displacement per cycle against different temperatures. Irrespective of geometry, two sharp regions of misorientation angle dependence of activation energies were estimated during the motion <112> and <100> tilt boundaries. In case of <112> tilt boundary, the transition angle between low angle and high angle grain boundary misorientations were reasonably similar to the values obtained for shear stress driven boundary motion. For instance, in the present study it was in the range of 13.6° to 15.5°, as compared to 13.6° in the previous experiments [30-32]. Similarly, in case of <100> tilt boundary, the transition angle between low angle and high angle grain boundary misorientation was estimated in the range of 7.8° to 11.7°, whereas, a misorientation angle 8.6 ± 0.15° was estimated for the pure shear stress driven grain boundary motion experiments [33]. However, transition angle between low angle and high angle grain boundary was difficult to establish for twist boundary motion, especially with regard to the measured activation energy due to a limited number of samples. Nevertheless, the average measured activation energy for the motion of twist boundary was found comparable to the activation enthalpies of grain boundary self diffusion.

Driving forces for motion of <112> and <100> tilt boundaries were calculated by using dislocation dynamics approach, assuming that the grain boundary motion is basically controlled by the movement of structural edge dislocations. Formation of trace-marks as observed on the sample surfaces during cyclic motion was found strongly dependent on the direction of force acting on dislocations rather than the mode of deformation. Similarly, the
mechanism of the <100> twist boundary motion has been discussed with in terms of the movement of structural screw dislocations by cross slip. Mechanism models with respect to driving force for the motion of grain boundaries have also been discussed. A relation between normal stress and shear stress were established in terms of the dislocation arrangement of the grain boundary. The contribution of sliding part and movement part during the entire boundary motion can approximately be predicted by the force acting on dislocation during deformation.
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b - Burgers vector; as index, designates that the quantity relates to grain boundary

d - Distance between dislocations in a low angle grain boundary; as index,
designates that the quantity relates to dislocations

v - Velocity; usually, the normal velocity of grain boundary or a surface

Γ - adsorption; Γ_i – adsorption of i-th component

P - Driving force

G - Gibbs potential: \( G = E – TS + pV = F + pV \)

V - Volume of the system

E - Internal energy; \( E_i \) – elastic modulus

p - Pressure

T - Temperature; \( T_m \) – melting temperature

F - Helmholtz free energy; \( F = E – TS \); also force

S - Entropy

Ω - Gibbs grand potential; \( \Omega_a \) – atomic volume

ν_D - Debye-frequency

m_i - mobility of i-th subject; \( m_b \) – grain boundary mobilt

μ - Chemical potential; also shear modulus

ρ - Density of a material or a phase

σ - Normal stress; \( \sigma^S \) – surface tension of free surface

τ - Shear stress

R - Radius; also gas constant

α - As a index of phase or a fraction of a system

H - Enthalpy; \( H_m \) – enthalpy of activation of grain boundary migration

δ - An infinitely small quantity; also grain boundary width

A(θ) - Constant factor

N - Number of cycle
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$Q_{gbm}$ - Activation energy of grain boundary motion

$\theta$ - Misorientation angle of grain boundary

$\Delta f_i$ - Difference of slip line density

$\Delta m$ - Difference of Schmids factor

$\Theta$ - Frequency of loading

$\Sigma$ - Inverse density of coincidence sites

$\varphi$ - Misorientation rotation angle
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Chapter 1

Introduction

The process of solid formation from liquid is eventually the nucleation and growth of small crystallites randomly orientated throughout the solidifying system, and therefore, each of these solid crystallites continues to grow until it meet with a neighbouring crystal. In general, the individual crystals will not be either of the same orientation or aligned, and this mismatched or mis-fitted area or surface between these individual crystals is called grain boundary, in broader sense it is also called interface, which is a longest but least known crystal defect in material research history.

The motion of the grain boundary in polycrystalline materials has been considered as an important phenomenon since this changes the physical, chemical, and mechanical properties of the material. The understanding of the mobility of grain boundaries in polycrystals as well as bicrystals constitutes an important aspect in the design of materials for high temperature services. Investigations of the movement of grain boundaries in single and two phase systems have merited significant attention because of their relevance to many technological processes. The extent of such movement controls the grain size, texture and redistribution of impurities and these changes, in turn, affect the mechanical behaviour of the materials.

Traditionally, a large attention has been confined to investigate grain boundary motion mostly, under simple stress driven or curvature driven motion. For instance, the stress-induced movement of planar low angle boundaries in zinc crystal was first observed by Washburn, Parker et al. [39] in 1952. The migration of curved (capillary force driven) grain boundaries, as a function of driving force, misorientation angle and temperature in NaCl and Cu-bicrystals was investigated by Sun and Bauer et. al. in 1970 [13-14] and Viswanathan and Bauer in 1973 [42] respectively. The temperature and pressure dependencies of $<100>$, $<110>$, and $<111>$ tilt grain boundaries in bicrystals of pure aluminium were also measured, and the corresponding activation enthalpies and activation volumes were determined [21-24]. A major amount of investigations on grain boundary migration were extensively focussed on aluminium and Zinc (Fig. 2.10, 2.11, 2.12, 2.13) [25, 34] considering a very specific boundary. Recently, an experimental observation of grain boundary motion under an applied shear stress has been reported [29-33] to prove that both low and high angle boundaries could be moved by an external mechanical stress.
Much less work has been published on investigation of cyclic stress driven grain boundary motion. Nevertheless, the possibility of a correspondence between migration and cyclic loading was first noted by Ritter and Grant [57] in low cycle axial fatigue experiments conducted on high purity aluminium. They observed an approximate one-to-one correspondence between migration steps and fatigue cycles, up to at least 36 cycles with a certain range of grain size of the materials. Subsequently, same kind of correspondence was also reported on the experiments on cyclic loading of copper [59] and Lead [46-47] crystals. Furthermore, a model for cyclic sliding and migration and its experimental consistency were presented by Langdon and Gifkins [48-49] in reverse bending and torsion fatigue at low cycle frequencies. Simultaneously, the motion of grain boundaries during cyclic deformation of high purity Al bicrystals [65-66] and the flow stress behaviour and microstructural development of nickel polycrystals during high temperature low cycle fatigue (HT-LCF) [61-63] were also reported.

However, most of these investigations were made to obtain a sharp knowledge of technological importance of the cyclic deformation and fatigue behaviour of polycrystalline materials rather than to confine on the fundamental aspects of grain boundary motion, microtexture and microstructure evolution during cyclic deformation. In contrast, study of grain boundary motion in polycrystalline materials has some conceptual limitations, for instance, it contains curved as well as planar grain boundaries, whose mechanism and kinetics of individual boundary motion are quite different, secondly, it contains triple junctions, which affect the motion of individual boundaries. The former experiments on boundary motion under cyclic deformation were performed without considering these limitations, which can be overcome by conducting the experiments on bicrystalline materials.

The present study basically emphasises the behaviour of grain boundary motion under two distinct cyclic deformation conditions:

1) High temperature response of tilt grain boundary under plastic amplitude of stress.
2) Mechanism and kinetics of grain boundary motion under a completely elastic stress and effect of inclination angle of grain boundaries with stress axis during cyclic motion.

The first part of the study focuses on the high temperature cyclic stress response of high purity aluminium bicrystals which were investigated by means of quasi in-situ measurements of the grain boundary as well as the grain body (i.e. inspecting the same area of the bicrystal samples before and after the deformation) at different combination of cyclic deformation parameters. Particular attention has been focussed towards the effect of cyclic stress at elevated temperatures on grain orientation during deformation and misorientation angle
variation due to grain boundary motion. At the same time it was clarified that the dislocation density difference was the prime source of driving force for the grain boundary motion.

The second part of the work mainly aims at investigating the mechanism and kinetics of cyclic motion of elastically deformed bicrystals at elevated temperatures. Experiments were carried out at different temperatures to find out the activation energies for different misorientation angles of grain boundaries to show the misorientation dependence of grain boundary migration under cyclic deformation. The driving forces for cyclic motion were calculated as well as discussed to propose a model mechanism during cyclic motion of geometrically different grain boundaries. Simultaneously, microstructure and microtexture resulting from the grain boundary motion were also described with respect to its deformation conditions. The activation parameters have been compared with the standard previously documented results.

For this purpose, bicrystals with symmetrical <112> and <100> tilt and <100> twist grain boundary were grown with a range of misorientation angles 7° to 42°. These grown crystals were cut with different geometry (0° to 90° inclination angle with stress axis). All the samples were cyclically deformed in plastic as well as elastic amplitudes of stress.

The deformation experiments were carried out using a set-up of cyclic deformation machine, which was completely fabricated in our institute workshop. The grain boundary displacement and deformed structure of each grain were monitored by using standard optical microscope. The grain orientations were measured by Electron Back Scatter Diffraction (EBSD) technique using Scanning Electron Microscope (SEM) before and after the deformation.
Chapter 2

Theory and Literature review

2.1 Structure of grain boundaries

2.1.1 Definition of grain boundary

The solid materials are essentially made up of atoms, ions or molecules, and in the crystalline state the vast majority of these are arranged in regular or periodic type arrays. When a solid form from the liquid it does so by the nucleation and growth of small crystallites randomly orientated throughout the solidifying system, and these grow as the liquid solidifies. The crystals continue to grow until they meet with the neighbouring crystals and, in general, the individual crystals will not be either of the same orientation or aligned. These two neighbouring crystals in general, called grains. The boundary where these two crystals meet becomes a surface and the mismatch in the orientation accommodated by distortion and gaps in the atomic arrangement is termed as grain boundary. In other words, grain boundary separates two regions of same crystals but of different orientations. A general definition of interface, given by Sutton and Balluffi [1] was that an interface, the free energy of which is at or near a local maximum with respect to one or more macroscopic geometrical degrees of freedom, which was contrasted with singular and vicinal interfaces. Because there are five macroscopic degrees of freedom it is possible for an interface to be ‘general’ with respect to some and ‘singular’ or ‘vicinal’ with respect to other macroscopic degrees of freedom. Therefore, according to the general definition of interface, a grain boundary can defined as a homophase interface involving a misorientation between the adjoining crystal lattices. Correspondingly, Gottstein and Shvindlerman [2] stated that these grain boundaries are the longest known but least understood lattice defects in crystalline materials which have been a major role on the structure and properties of the materials. In very coarse grained materials it can be discerned by the naked eye if the surface is properly prepared.

The major problem regarding fundamentals of grain boundaries is mainly due to their complex structure, which requires an extensive mathematical description for its macro and microscopic characterisation. In reality, the orientation relationship between two crystal lattices is a transformation, which has to be applied to one of the crystals to make both crystal lattices coincide. If a common origin is assumed, this transformation is a pure rotation, since
the relative positions of the crystal axes in both crystals are the same. Usually, the three Euler angles are used to define a grain boundary, but it is easiest to visualise a grain boundary when the rotation is represented in terms of an axis and angle of rotation. In many instances, it is very important to know the dependence of a property on the rotation angle for a given rotation axis. In this case it would be desirable to keep the crystallographic orientation of the grain boundary plane constant in order to obtain the dependence on the rotation angle only. When the grain boundary plane is perpendicular to the rotation axis, the boundary is referred to as

Fig. 2.1. Relative orientation of grain boundaries and rotation axes for different types of grain boundaries a) Twist boundary; b) Asymmetrical tilt boundary; c) Symmetrical tilt boundary. [2].
the twist boundary (Fig. 2.1a). Similarly, if the rotation axis is parallel to the grain boundary plane, then it is called tilt boundaries. Since there is several numbers of possible planes parallel to a given direction, there is an infinite number of tilt boundaries for a given rotation. If the adjacent crystals are mirror images of each other, then the grain boundary is referred to as a symmetric tilt boundary (Fig. 2.1c) and all other tilt boundaries are called asymmetric tilt boundaries (Fig. 2.1b). In contrast, the normal to grain boundary plane must be perpendicular to the rotation axis for tilt boundaries. Therefore, to investigate boundary structure – property relationships it is sensible to first confine to symmetric tilt boundaries and then treat asymmetric tilt boundaries as a deviation from the symmetric position. As an example the dependence of grain boundary energy on the angle of rotation for a <110> rotation axis in aluminium is given in Fig. 2.2. for symmetrical tilt boundaries.

![Figure 2.2](image)

Fig. 2.2. Dependence of the energy of symmetrical <110> tilt boundaries in Aluminium on misorientation angle [60].

### 2.1.2 Classification of boundaries with respect to atomic structures

**a) Low angle grain boundaries**

If the misorientation between adjacent grains is small (< 15°), then the entire boundary is comprised of a crystal dislocation arrangement, which is called low angle grain boundary (LAGB). This definition was obviously proved from the dislocation model as well, has been
confirmed by high resolution electron microscopy. In contrast, this is the misorientation range where mutual interactions between cores of primary dislocations are sufficiently small that the core structures are not topologically distinct from those of an isolated crystal lattice dislocation of the same Burgers vector and line direction. On the other hand, low angle boundary may be regarded as a semicoherent interface consisting of coherent patches separated by dislocations which destroy the continuity of the reference lattice or crystal lattice. An intensive discussion about the structure of low angle boundary can be find from [1]. More detail investigation for the transition between low angle and high angle grain boundaries were already discussed by different authors [2] with respect to activation enthalpy during motion of these boundaries. Symmetric tilt boundaries generally consist of a single set of dislocations (Fig. 2.3a), where the dislocation spacing $d$ decreases with increasing rotation angle $\phi$

$$\frac{b}{d} = 2 \sin \frac{\phi}{2} \approx \phi$$

(2.1)

For asymmetric low angle tilt boundaries at least two sets of edge dislocations are required, the Burgers vectors of which perpendicular to each other (Fig. 2.3b).

(a) [Diagram of grain 1 and grain 2 with dislocations]

(b) [Diagram of boundaries with dislocations]
Fig. 2.3. Dislocation configuration of a) a symmetrical b) an asymmetrical <100> low angle tilt boundary with tilt angle \( \varphi \) and inclination angle \( \Phi \) in a simple cubic crystal [2].

The fraction of dislocations of the second set increases with increasing deviation from the symmetric tilt boundary that is only comprised of the first set of dislocations. If the grain boundary is composed only of the second set of dislocations, another symmetrical tilt boundary is obtained which is normal to the symmetric tilt boundary that is comprised only of the first set of dislocations. Low angle twist boundaries require at least two sets of screw dislocations. Mixed boundaries are composed of dislocation networks of three burgers vectors.

b) High angle grain boundaries
Gottstein and Shvindlerman [2] indicated that for angles of rotation in excess of 15°, grain boundary energy decreases, however, measurements of grain boundary energy reveal no further change with increasing rotation angle, in contrast to the dislocation model which would predict an energy decrease for high angles of rotation. The rotation angle larger than 15° the dislocation model fails, because the dislocation cores tend to overlap. Therefore, grain boundaries with rotation angles in excess of 15° are distinguished from the low angle grain boundaries and are termed as high angle grain boundaries (HAGB). With increasing misorientation of the cores of primary dislocations interact increasingly as their separation decreases. The interactions lead to distortions of the structure of the cores compared with the core structure of an isolated crystal lattice dislocation of the same line direction and Burgers vector. In the high angle regime the distortions are sufficiently large that the core structures of intrinsic dislocations are topologically distinct from those of isolated dislocations [1]. At first glance a high angle grain boundary appears like a zone with random atomic arrangement (Fig. 2.4). In fact early models of grain boundary structure assumed grain boundaries to represent an under cooled liquid.

The recent understanding of the structure of HAGB is derived from geometrical concepts, based on dislocation models of LAGBs [3]. A fundamental reason for the failure of the lattice dislocation concept for larger angles of rotation is the requirement of a strictly periodic dislocation arrangement to minimize the grain boundary energy. For larger rotations the orientation difference between two consecutive periodic dislocation arrangements becomes substantial.
2. Structure of grain boundaries

Fig. 2.4. High-angle and Low-angle tilt boundary in soap bubble model [2].

c) CSL- boundaries

A boundary having a reference structure in which the two adjoining lattices in their unstressed states almost form a perfect coincide site lattice. The reference bicrystal structure constructed by deforming homogeneously either or both adjoining crystal lattices so that they form a coincide site lattice. Several model has been established to defined the coincide site lattice boundary, indeed, a more detail and convenient definition was found in [1, 2]. For example, from the book of Gottstein and Shvindlerman [2] the definition of CSL-boundary was firmly established as follows.

In perfect crystal atoms have a defined position, which is determined by the minimum free energy. Any deviation from this position necessarily increases the free energy. Therefore, it can be assumed that the crystal will try to keep the atoms as much as possible in their ideal position, also in the grain boundary. There are orientation relationships, where crystallographic planes continue through the grain boundary from one crystal to the other, i.e. there are atomic positions in the grain boundary which coincide with ideal positions of both adjacent lattices. Such lattice points are called coincidence sites. Since the orientation relationship between the adjacent crystals is described by a rotation, it can be investigated under what conditions coincidence will occur. As both crystal lattices are periodic, the coincidence sites also must be periodic; they defined by the term coincidence site lattice (CSL) [5]. As a measure for the density of coincidence sites or for the size of the elementary cell of the CSL, we define the quantity
Fig. 2.5a is only a very simple 2-dimensional case. In reality the coincidence lattice is a 3-dimensional lattice, the generation of which can be imagined as follows. We take a crystal lattice, each lattice point of which carries two atoms, for example one round and one triangular as shown in Fig. 2.5a. Now we rotate the triangular atoms while the round atoms remain unchanged and the origin for the axis of rotation is a lattice point. After this rotation there are again lattice points where triangular and round atoms coincide. These are the coincidence sites and because of periodicity of crystal lattices they generate a 3-dimensional lattice which is called the CSL. To apply this crystallographic construct to grain boundaries, we have to define the spatial orientation of the grain boundary plane. Grain boundaries between crystallites which have an orientation relationship corresponding to a high density of coincidence sites are called CSL boundaries or special boundaries. The smaller the $\Sigma$ (which is always an odd integer), the more ordered is the grain boundary. For example, low angle boundaries

\[
\Sigma = \frac{\text{Volume elementary cell of CSL}}{\text{Volume elementary cell of crystal}}
\]  

(2.2)

Fig. 2.5. a) Coincidence site lattice (CSL) and structure of a 36.9°<100> ($\Sigma=5$) grain boundary in a cubic crystal lattice. Right side of the figure: grain boundary plane parallel to the paper (twist boundary); left side of figure: grain boundary plane perpendicular to the paper (tilt boundary) b) Coincidence site lattice (CSL) and displacement shift complete (DSC) lattice at 36.9° <100> rotation in a cubic lattice [2].
2.2 Grain boundary motion

2.2.1 Introduction

Grain boundaries separate regions of homophase interface involving misorientated crystal structure with the neighbouring crystal lattices. The displacement of a grain boundary is entirely equivalent to the growth of one crystallite with the expense of shrinking of its neighbours. In this sense the grain boundary constitutes the contact area of the internal surfaces of adjacent grains. It is repeatedly mentioned for a long time that the displacement of the crystallite surfaces distinctly distinguishes grain boundary motion from a diffusive flux of atoms across the boundary, but later, it is proved that diffusion of atoms across boundary does not necessarily correspond to grain boundary migration. It is evident that grain boundary motion consists of the generation of lattice sites at the surface of growing crystal and consequently destruction of lattice sites at the surface of shrinking crystal, which essentially means, a non-zero net exchange of lattice sites across the boundary (Fig. 2.6a). More generally, the motion of low angle grain boundaries will involve the simultaneous glide and
climb of their structural dislocations. An applied shear stress (normal to the boundary plane) exerts a force on the boundary urging it to move by a process involving the motion of each primary anticoherency dislocation in a direction normal to the boundary plane by means of combined glide and climb. Successive dislocations in the array must then execute alternating positive and negative climb, and this can be achieved by establishing local self-diffusion currents so as to allowing the overall boundary motion to be conservative. The two basic underlying processes which transport atoms from the shrinking crystal to the growing crystal are the local shuffles which occur as the dislocations glide and the diffusional transport which occurs when they climb. Similarly, topologically, high angle boundaries can be able to move conservatively via the simultaneous glide and climb of their primary dislocations in general as the same way low angle boundaries. However, singular or vicinal high angle boundaries show resistant to motion via the simultaneous glide and climb process, since this process would inevitably cause significant perturbations in the regular arrangement of these closely spaced dislocations and, therefore, significant perturbations in the boundary energy. Therefore, these boundaries would be more easily movable as semicoherent interfaces via the lateral movement of secondary dislocations possessing step character by combined glide and climb. A detail discussion of these diffusion processes can find in the Book of Sutton and Balluffi [1].

Fig. 2.6. a) Example of grain boundary motion which deletes and generates lattice sites on the surface of the shrinking and growing grain, respectively b) the free energy of a moving atom changes by the driving force Pb$^3$ when it crosses the boundary. G$_m$ is the free energy barrier for bulk diffusion [2].
2.2 Grain boundary motion

Several work has been done on these particular subject, one example can be discussed after a intensive literature survey in [2], it has been seen that there is no real theory on grain boundary migration rather all theoretical attempts were based on simple rate theory of atoms crossing the boundary with net energy gain. It is tacitly assumed that the atom detaching from a crystal to join the opposite surface destroys a lattice site rather than creating a vacancy and its attachment to the adjacent crystal surface generates a new lattice rather than eliminating a vacancy, which assumption reduced the concept of grain boundary motion to diffusive flux across the boundary, and therefore, it was considered a simplest case [8] to get the grain boundary velocity \( v \) like as

\[
v = b(\Gamma_+ - \Gamma_-)
\]  

(2.3)

Where, \( \Gamma_+ \) and \( \Gamma_- \) are the jump frequencies in the respective directions and \( b \) the diameter of an atom. If the Gibbs free energy of both crystals per unit volume is different by the driving force,

\[
P = -\frac{dG}{dV}
\]  

(2.4)

then each atom of volume \( \Omega_a \approx b^3 \) will gain the free energy \( Pb^3 \) when becoming attached to the growing grain but has to expend this free energy when moving in the opposite direction. The corresponding free energy variation across the boundary is sketched in Fig. 2.6b.

Therefore, considering the above case, boundary velocity will be like as

\[
v = b \left( \nu_+ e^{\frac{G_m^+}{kT}} - \nu_- e^{\frac{G_m+Pb^3}{kT}} \right)
\]  

(2.5)

If the attacking frequencies \( \nu_+ = \nu_- = \nu \approx \nu_D \) (\( \nu_D \) – Debye-frequency) and the migration free energy \( G_m \) is the same in both jump directions, then

\[
v = b\nu_D e^{\frac{G_m}{kT}} \left( 1 - e^{\frac{Pb^3}{kT}} \right)
\]  

(2.6)

In real cases, including recrystallization in a heavily cold worked crystals or metals, \( Pb^3 \ll kT \) at temperatures where boundaries are observed to move and therefore,

\[
\exp\left(-\frac{Pb^3}{kT}\right) \approx 1 - \frac{Pb^3}{kT}
\]  

(2.7)

which simplifies the equations (2.6) and (2.7) like as

\[
v = \frac{b^4\nu_D}{kT} e^{\frac{G_m}{kT}} P = mP
\]  

(2.8)
2. Grain boundary motion

where \( m \) is referred to as grain boundary mobility. Indeed this model was modified later [9], by assuming the detachments to occur in a sequence of steps or that thermal grain boundary vacancies have to assist diffusion [38], however, this changing will change only the pre-exponential term \( (m = m_0 e^{H/kT}) \) but proportionality term \( (v/P) \) will be unaffected. In principle, this simple model was basically emphasised that the activation free energy for the diffusion jump of atoms across the boundary was assumed to correspond to activation energy for volume diffusion.

2.2.2 Driving forces for grain boundary motion

The conservation motion of an interface will generally occur in response to a driving pressure which will exist whenever the conservative motion of the interface causes the total free energy of the system to decrease. This pressure is given by

\[
p = -\frac{\delta G}{\delta x}
\]

where, \( \delta G \) is the change in free energy of the system when unit area of interface moves a distance \( \delta x \) normal to itself. The driving pressure is also denoted by driving force which has a dimension of energy per unit volume, which is conceptually equivalent to a pressure, in other words, force acting per unit area on a grain boundary. Theoretically, the driving force or pressure for motion of interface may originated from a wide variety of sources. For instance, a gradient of any thermodynamic variable offers a source of such driving force, such as, a gradient of temperature, applied external pressure, density of defects and so on. However, these all theoretical source of driving force can not be practically possible. The relevant driving forces was tabulated to the following way in the book of Gottstein and Shvindlerman [2], although the list of potential driving forces is very long, indeed, a long list of sources of driving pressure can be find in [1].

1) An excess density of defects (e.g. dislocations) in one of the adjoining grains is a powerful source of driving force. There are several advantages of this kind of driving force, such as ease of fabrication, excellent reproducibility, variation within a large force (driving force \( P = \rho \mu b^2/2 \), where \( b \) is Burgers vector, \( \mu \) is shear modulus, and \( \rho \) is dislocation density). In reality, this kind of source of driving force is very relevance to describe the recrystallization phenomenon.

2) The energy of low angle grain boundaries like in the classical experiments of [10], proved to be one source of driving force for migration. The authors proved that the striation substructure provided the driving force for grain boundary migration, as it frees the
crystals from striations [8]. This kind of driving force can be considered as relatively reproducible and sufficiently stable. Major advantage of this driving force was the possibility to study the motion of a flat grain boundary.

3) The driving force in several methods of investigating grain boundary motion is exerted by the pressure difference $\Delta p$ on both sides of the grain boundary (e.g. $P = \Delta p$). This kind of driving force was exhibited in curved grain boundary motion. Usually, pressure difference $\Delta p$ stems from the difference of capillary forces on both sides of a curved grain boundary. A capillary pressure is equal to

$$\Delta p = \sigma_b \left( \frac{1}{R_1} + \frac{1}{R_2} \right)$$

where $\sigma_b$ is a surface tension, $R_1$ and $R_2$ are the main radii of curvature. This source of driving force offers a number of advantages, such as, possibility to control and to change, good reproducibility, and a good stability in a given temperature.

4) The anisotropy of any physical property, e.g. the elastic constants or the magnetic susceptibility or applying external mechanical stress, can be utilised as a source of driving force for grain boundary migration. The origin of the driving force for boundary migration in a magnetically anisotropic material was considered by Mullins [11].

5) The anisotropy of surface tensions of the free surfaces of a bicrystal represents a source of driving force for grain boundary migration. If the crystallographic planes that constitute the surfaces of the adjoining grains in bicrystals have a different surface tension $\Delta \sigma_s$, the boundary feels a driving force $P = 2\Delta \sigma_s / \lambda$, where $\lambda$ is the thickness of crystals.

## 2.2.3 Measurement of grain boundary mobility

### a) Polycrystal methods

In the previous sections we have already discussed about the capabilities and circumstances for the grain boundaries to move, and this ability of the boundaries are a common phenomenon for the technological processes as recrystallization and grain growth. Therefore, its a natural thrust to extract data on grain boundary motion, which means, grain boundary mobility, from the temporal evolution of grain size during recrystallization and grain growth in polycrystals, but these experimental data fails to solve the physical concept of grain boundary migration. As result, the relationship between grain boundary mobility and its crystallography, effect of temperature, pressure or external stress, impurity content and the mechanism of grain boundary migration cannot be studied by these recrystallization methods. On the other hand, recrystallization and grain growth are multi-component processes of
2.2 Grain boundary motion

microstructure evolution, which is strongly involving on polycrystal structures. Therefore, these methods only give a rough estimate rather a reliable or reproducible picture.

b) Bicrystal methods

As polycrystal methods having various problems, it can be mention that bicrystal methods were permitted to obtain more reliable and reproducible physical data for grain boundary migration. However, these bicrystal techniques require substantial experimental efforts to produce bicrystals with precise orientations and misorientation of grain boundaries. A basic advantage of all these techniques to utilise the capillary driving force, which is practically constant over a wide range of temperatures. The “wedge” bicrystal technique was frequently used [12] due to the simple relations between driving force \( P \) and the grain dimension, i.e. radius of curvature of grain boundaries (e.g. \( P = \sigma_b/a \), where, \( \sigma_b \) is the grain boundary surface tension).

The reversed-capillary technique, first proposed and developed by Sun and Bauer [14], was used many times to investigate grain boundary migration in crystals. The major advantages of this technique are the possibility to change the driving force by varying the tangent angle of grain boundary curvature. The shape of a moving grain boundary for this method was analysed by Mullins [11], Sun and Bauer [14], Furtkamp et.al [15]. The driving force for the reversed capillary method is given by

\[
P = \frac{\sigma_b}{R} = \frac{\sigma_b}{a} f(\alpha)
\]  

(2.11)

where \( R \) is the radius of boundary curvature and \( \alpha \) is the tangent angle of the boundary curvature. The unique disadvantage of the reversed-capillary technique is the lack of steady-state motion of a grain boundary. This disadvantage was avoided by taking the boundary curvature as half-loop or quarter-loop, as investigated in [13-15] and the average driving force \( (P) \) were given by

\[
P_{\text{half-loop}} = \frac{2\sigma_b}{a} \quad \text{and} \quad P_{\text{quarter-loop}} = \frac{\sigma_b}{a}
\]  

(2.12)

The above mentioned methods are commonly performed by monitoring the grain boundary positions by observed the groove on the surface of the bicrystal under a optical microscope in a defined time interval and measure the velocity of grain boundary migration, this technique is called discontinuous type.

The other method so called continuous method requires determination of the boundary position in any time with automation fashion. This continuos method is more precise and elegant with respect to discontinuous, and it was used to investigate boundary motion by several techniques such as, use of reflection of polarised light [16], photoemissions [17], X-
ray topography [18] and diffraction [19], or backscattered electron density [20] as found in literature.

Among all these continuous methods, a recently developed elegant technique was used to investigate crystal shape evolutions with an orientation contrast difference system, using a hot stage under SEM for in situ measurement of boundary motion. At the same time, a simple, precise and versatile technique was used to identify the position of boundary utilising X-ray diffraction. This technique is called X-ray Interface Continuous Tracking Device (XICTD), was developed [15-16, 19] to measure the continuous velocity of grain boundary without inferring the boundary migration.

### 2.2.4 Kinetic parameters of grain boundary mobility

As we have seen into the previous sections, that the driving force for grain boundary migration is always small than thermal energy, therefore, this migration is a drift motion followed by a trend to reduce the energy content of crystalline aggregates. A drift velocity, however, is always proportional to the driving force like as

\[ v = m_b P \]  

and the proportionality fraction \( m \) is the grain boundary mobility

\[ m_b = \frac{v}{P} \]  

with the proportionality between migration rate and driving force being unambiguously established, which can be used to determine the grain boundary mobility from grain boundary motion experiments and investigate the influence of temperature, applied pressure, grain boundary crystallography and chemical composition on grain boundary mobility. As evident from the previous experiments, proved that grain boundary migration is a thermally activated process. Thus, its kinetics follows Arrhenius type temperature dependence

\[ v = v_0 \exp\left(-\frac{H_m}{kT}\right) \]  

Since the driving force is independent of temperature, thus, temperature dependence of \( v \) is the temperature dependence of the grain boundary mobility like as

\[ m_b = \frac{v}{P} = m_0 \exp\left(-\frac{H_m}{kT}\right) \]  

where \( H_m \) is the activation enthalpy of grain boundary migration and \( m_0 \) the corresponding pre-exponential mobility factor. It is noted that

\[ H_m = E_m + pV^* \]  

(E_m – activation energy) depends on the pressure p, which can be used to determine the activation volume V'. However, for all practical purposes grain boundary migration proceeds under a constant pressure.

### 2.2.5 Mechanism models of grain boundary motion

It has been repeatedly mentioned that there is no discrete theory of grain boundary migration, except from some experimental results and computer simulations or high resolution TEM studies of boundary migration. Nevertheless, recently, lot of efforts has already been done on stress driven as well as curvature driven grain boundary migration to investigate the exact mechanism of grain boundary migration by [21-34]. Before these, we should review and critically assess few of the well known proposals made into the previously done experiments.

Early models [35-36] of grain boundary structure assumed that grain boundary comprised a certain volume of thickness, δ>b, (b- Burgers vector), i.e. much broader than the spacing of atoms in the adjacent lattices and the boundary motion is a steady-state motion like as diffusion flux of atoms across the boundary. This concept was first proposed by Gleiter [9], who treated grain boundary migration like an evaporation/condensation process on the internal surfaces. But unfortunately, this model lacks predictive power since most involved crystallographic parameters are unknown.

Mott [36] assumed in this theory of boundary migration, which also called Island model, that the boundary contains small patches of perfect crystal structure which become detached and attached to the crystal surfaces by partial melting and solidification processes. Although, this model was also based on unrealistic assumptions of the boundary structure and does not incorporate boundary crystallography, while this is the first attempt to describe boundary motion as a group motion of atoms.

Recently one model was proposed by Babcock and Balluffi [80]; they conducted in-situ experiments on grain boundary migration and sliding in thin film Au bicrystals in a hot stage of a TEM, where they confirmed the concept of dislocation model of combined grain boundary sliding and migration by correlating successfully the amount of sliding and migration with the number of moving SGBD and their Burger vectors. However, this well-defined and correlated motion constituted only a negligible part of the entire grain boundary migration.

More recent molecular dynamics computer simulations on flat <100> twist boundaries [75-76] have confirmed such cooperative processes, although the actual trajectory of atoms during
boundary migration is very complex owing to a high level of thermal activation in and close to the boundary causing concomitant rapid diffusive motion and high defect concentration. One possible mechanism as proposed by [17] in analogy to crystal growth. The centres of the spirals are grain boundary screw dislocations which are assumed to be decomposition products of crystal dislocations, absorbed in the grain boundary during grain boundary motion. Accordingly, the spiral density is proportional to the dislocation density, i.e. proportional to driving force like as $v = m(P)P \sim P^2$. Indeed, spiralling grain boundary screw dislocations were observed experimentally too.

### 2.2.6 Experimental observations for planar and curved grain boundary motion

As a thermodynamic point of view the grain boundary migration in the polycrystalline or bicrystalline metallic material were found when the boundary displacement leads to the reduction of a total energy of the system. There are several ways by which this can be accomplished, in specific, the most important concept was that to use free energy of the GB itself or utilises the free energy difference of the adjacent grains. The most frequently used experimental method was known to be curvature driven displacement of grain boundaries, investigated by [19, 25, 26-28, 21-24]. Second type of concept was to force to move the planar boundary by implementing an external driving force on the grain boundary [29-34]. Usually, bicrystal with grains that have some orientation dependent property like elastic constants or magnetic susceptibility was utilised for the investigation of planar grain boundary migration, indeed, the driving force does not depend on boundary properties, such kind of driving force can be obtained by the action of an external mechanical stress or magnetic field on the bicrystal. These features were investigated in different way by several scientists, like as the following authors.

#### a) Washburn and Parker [39-40]

The stress-induced movement of planar low angle boundaries in zinc crystal (temperature range of 25° to 400°C) was first observed by Washburn, Parker et al. [39] in 1952, with polarised light in an optical microscope. It was noted that the dynamic behaviour of the boundaries seemed to require that they consist of an array of edge dislocations of like sign distributed more or less uniformly over the plane of the boundary. The authors also determined the activation energy for the movement of the boundaries in the temperature range of 300 to 400°C was about 21,500 calories per mole. Attention was drawn to the close agreement between this value and those of the activation energies for self-diffusion, creep,
and recovery in zinc crystals. A mechanism was suggested for the formation of a substructure in crystals plastically deformed at low temperatures which depended only on the stress-induced movement of small angle boundaries. This mechanism was based on observations that the rate of boundary motion decreased with increasing boundary angle. Small angle boundaries overtook and united with those of larger angle, forming slower moving, or even stationary substructure boundaries.

b) Sun and Bauer [13-14]
Migrations of [100] tilt boundaries in NaCl and Cu-bicrystals were investigated by Sun and Bauer et al. in 1970 [13] and Viswanathan and Bauer in 1973 [42] respectively, as a function of driving force, misorientation angle and temperature, (Fig. 2.7) where they utilised a novel method wherein hyperbola-like shapes were generated by capillary forces acting on a boundary originally subtending an acute angle with a free surface. Initially, intrinsic (I) boundary motion, characterised by an activation energy $Q_I$ which increases from 1.5 to 2.2 eV as decreases from 30 to 5°, was sustained by the driving force. For large, $Q_I$ denotes the activation energy for self diffusion across the boundary. For small however, $Q_I$ nearly

![Fig. 2.7. Misorientation angle dependence of activation enthalpy during grain boundary migration in NaCl bicrystals [13] and for Cu-bicrystals [42].](image)
coincides with the activation energy for bulk self diffusion of the slower ionic species (Cl), indicating the occurrence of dislocation climb processes involving self diffusion through the bulk crystal. They also investigated the motion of curved grain boundary with unknown grain boundary energy.

c) Fukutomi and Horiuchi [43]

In 1981, Fukutomi and Horiuchi, investigated migration of tilt grain boundaries in Al-bicrystals with taking different type of tilt axis and misorientation angle, under mechanical stress. The activation energy of migration was determined for different type of grain boundaries with a various temperature range, although, they used very high applied stress on the bicrystals (nearly 1 MPa), which considered to be plastic stress for the samples at higher temperatures. The grain boundary mobility (v) were measured by equation like as

$$v = A(\theta).\sigma^n.\exp\left(-\frac{\Delta H}{kT}\right)$$

(2.18)

where $A(\theta)$ is the constant factor, $\Delta H$ is the activation enthalpy, $\sigma$ is the applied stress with exponent $n = 1 \pm 0.2$, $k$ Boltzman constant, and $T$ is absolute temperature.

Fig. 2.8. Activation enthalpy for different misorientation angle during migration of boundaries in Al-bicrystals [43].
The authors were also reported a transition of low to high angle of misorientation of boundaries with respect to activation enthalpy during migration, for example, for higher angle (> 5°), the ΔH was measured 0.82 eV, whereas, smaller angle of misorientation (< 5°), it was 1.2eV-1.4eV with a same tilt axis (Fig. 2.8).

d) Lücke and Detert [44-45]
The experimental facts concerning the influence of small amounts of impurities upon recrystallization were briefly reviewed by Lücke et.al [44] and an atomistic theory of these phenomena was developed. In this theory it was assumed that interaction forces exist between the foreign atoms in solid solution and the boundary, increasing the concentration of foreign atoms in the boundary. At high concentrations (or low temperatures) the moving boundaries were held back by the foreign atoms and the speed of the boundary was controlled by the speed of the foreign atoms diffusing behind the boundary. There was a critical concentration of impurities for the moving grain boundaries in which, boundary moves faster and recrystallization temperature changes as a function of concentration. The quantitative observations from experimental results leads to established a absolute rate of boundary migration, as a function of temperature and activation energy of recrystallization. It was also noted that the rate of recrystallization process increases with increasing difference in radius of solute atoms and solvent atoms, and the interaction between boundary and solute atoms were shown to be elastic in nature.

e) Gottstein and Shvindlerman [21-24]
Majority of investigations on grain boundary migration was studied extensively for Lead (Fig. 2.9) [10], aluminium (Fig. 2.10, 2.11, 2.12, 2.13) [19] and Zinc [25] with considering a very specific boundaries. For instance, the temperature and pressure dependencies of <100>, <110>, and <111> tilt grain boundaries in bicrystals of pure aluminium were measured, and the corresponding activation enthalpies and activation volumes were determined [21-24]. While the activation enthalpy strongly depended on orientation, in particular close to low Σ coincidence orientation relationships, the activation volume was found independent of orientation except for <110> tilt boundaries. The absolute values of the activation volume for <100> and <111> tilt boundaries were about the same as for bulk self-diffusion, which agrees with previous measurements on polycrystals. The activation volume for migration of <110> tilt boundaries substantially exceeded the activation volume for bulk self-diffusion. It is concluded that at least <110> tilt boundaries migrate by a group mechanism rather than by diffusive jumps of single atoms.
The influence of impurities on grain boundary mobility in a Σ7 (38.2° <111>) and in an off-coincidence boundary (40.5° <111>) was investigated by [26-28] (Fig. 2.14). The grain boundary mobility was found to strongly depend on grain boundary crystallography and material purity. The measured concentration dependence of activation enthalpy and pre-exponential mobility factor did not comply with predictions of traditional impurity drag theory.

Fig. 2.9. Activation enthalpies ($H_m$) were measured for different misorientation angle ($\varphi$) of <100> tilt boundaries in zone refined lead [10].
Fig. 2.10. The dependence of the activation enthalpy of migration for ⟨100⟩ tilt grain boundaries in Al of different purity (□ for 99.99995 at%; σ for 99.9992 at%; and µ for 99.98 at%) [19].

Fig. 2.11. The dependence of the activation enthalpy of migration for ⟨111⟩ tilt grain boundaries in Al of different purity (● for 0.4ppm, and ♦ for 1ppm) [22]

Fig. 2.12. Activation enthalpy vs misorientation angle for the motion of investigated planer and curved ⟨112⟩ grain boundaries with different purity. [31]

An extended impurity drag theory was presented that takes into account interaction of the adsorbed atoms in the boundary, which predicted a concentration dependence of the activation enthalpy. The compensation temperature was found to depend on composition.
2.2 Grain boundary motion

Fig. 2.13. Activation enthalpy vs misorientation angle for the motion of all investigated planar, symmetrical $<112>$- and $<111>$-tilt boundaries. [30]

The motion of planar, symmetrical $<112>$- and $<111>$-tilt boundaries under the influence of a mechanical shear stress was investigated and compared with experiments on curved, symmetrical $<112>$- and $<111>$-tilt boundaries with the same angles of misorientation under the influence of a constant capillary force [32]. It was found that the motion of boundaries can be induced by the imposed external stress and the activation of a migration mechanism depends on the misorientation angle and the driving force and that grain boundaries have the possibility to move by different migration mechanisms. Irrespective of whether grain boundaries are planar or curved there was a distinct transition between low angle and high angle grain boundaries at the same misorientation angle (Fig. 2.12 and 2.13).

Fig. 2.14. Dependence of a) activation enthalpy $H$ and b) preexponential factor $A_0$ of the reduced grain boundary mobility for $38.2^\circ$ (open symbols) and $40.5^\circ$ (filled symbols) $<111>$ tilt grain boundaries on impurity content in pure Al [26].
2.3 Grain boundary motion under cyclic deformation

2.3.1 Fundamentals

It has been recognised from a long time that metal or materials subjected to a repetitive or fluctuating stress will fail at a stress much lower than that required to cause fracture on a single on a single application of load. Failures occurring under conditions of dynamic loading are called fatigue failures. Fatigue has become progressively more prevalent as technology has developed a greater amount of equipment, such as automobiles, aircraft, compressors, pumps, turbines, etc, where, fatigue accounts for at least 90 percent of all service failures due to repeated loading and vibration. Three basic factors are necessary to cause fatigue failure. These are (1) a maximum tensile stress of sufficiently high value, (2) a large enough variation or fluctuation the applied stress and (3) a sufficiently large number of cycles of applied stress. In addition, there are a host of variables, such as stress concentration, corrosion, temperature, overload, metallurgical structure, residual stress, and combined stresses, which tend to alter the conditions for fatigue.

2.3.2 Key parameters

There are a number of parameters for cyclic deformation to be considered which are very important to the characteristics of cyclic deformations. These parameters include amplitude of stress/strain, number of cycle, type of cycle, frequency of loading, temperature, etc. According to type of stress/strain cycle, the cyclic deformation can be classified as 1) Reversed cycle of stress, in which maximum and minimum stresses are equal. Where, tensile stress is considered as positive and compressive stress is considered as negative as shown in Fig. 2.15a for sinusoidal form. 2) Repeated stress cycle, in which the maximum and minimum stresses are not equal. In this illustration they are both in tension, but a repeated stress cycle which could just as well contain maximum and minimum stresses of opposite signs or both in compression as shown in Fig. 2.15b for sinusoidal form. 3) complicated stress of cycle in which the maximum and minimum stresses are always fluctuating in nature like as in Fig. 2.15c.

Cyclic deformation can also be classified on the basis of number of cycle, such as 1) Low cycle fatigue (N < 10^4 cycles) and 2) High cycle fatigue (N > 10^5 cycles). Low cycle fatigue is associated with high strain fatigue where a fatigue cycle incorporates a component of cyclic plastic strain. Fatigue failure generally occurs in a relatively small number of fatigue cycles (N < 10^4 cycles), typically a few thousands of cycles, compared with hundreds or thousands
or millions of cycles in normal fatigue failure. The fatigue cycle may be load, extension, stress or strain controlled or a mixture of these modes. Frequently, dwell periods are incorporated as part of the fatigue cycle and these may be from some seconds to many hours duration. Cycle rates for low cycle fatigue are normally less than 1 Hz. Low cycle fatigue is generally conducted at elevated temperatures between 200°C to 1100°C. Sophisticated data capture techniques are required to minimise the quantity of data collected whilst retaining the integrity of the test. Critical cycles are analysed to determine stress and strain components.

Fig. 2.15. Typical fatigue stress cycles (a) Reversed stress; (b) repeated stress; (c) irregular or random stress cycle [4].

### 2.3.3 Kinetic parameters during cyclic motion

As we have seen in section 2.2.4 that grain boundary migration is a drift motion and the driving force is always smaller than thermal energy. In general, boundary velocity follows a proportionality relationship with driving force. As evident from the previous experiments on cyclic motion of grain boundaries [46-56], proved that grain boundary migration is a thermally activated process. Thus, its kinetics follow Arrhenius type temperature dependence like as

$$v = v_0 \exp\left(-\frac{H_m}{kT}\right)$$  \hspace{1cm} (2.19)

In order to determine the activation energy for grain boundary migration under cyclic deformation, the average migration displacement per cycle was plotted against testing temperature assuming a similar equation as above (Fig. 2.16) and the relationship follows as
\[
\bar{m} = A \exp\left(-\frac{Q_{gbm}}{RT}\right) \tag{2.20}
\]
where, \(\bar{m}\) is the migration displacement per cycle was considered as boundary velocity (v), \(Q_{gbm}\) is the activation energy for grain boundary migration, rather use activation enthalpy (\(H_m\)), \(R\) is the gas constant, \(T\) is the absolute test temperature and \(A\) is a constant.

![Graph showing the relationship between \(\bar{m} / \text{cycle (\mu m)}\) and \(1000/T \text{ (K^4)}\).]

Fig. 2.16. Average migration displacement per cycle vs the reciprocal of the testing temperature after 8 cycles with \(f = 1.3 \times 10^{-2} \text{ Hz and } \Delta \varepsilon = \pm 0.25 \%\). [54, 56]

### 2.3.4 Experimental observations of cyclic grain boundary motion

It was observed in the early fatigue tests that well-defined striations often form on the fracture surfaces of specimens in which crack propagation was occurring in stage II on non-crystallographic planes approximately normal to the tensile stress. These striations have the appearance of an assembly of essentially parallel lines, approximately equally spaced, which are oriented so that they lie perpendicular to the direction of crack advance. But after several experiments with repetitive loading on single crystals [57] it was firmly established that, each striations were produced by single cycle and the spacing between consecutive striations increases with increasing stress. These one-to-one correspondence between fracture striations and the conditions of cyclic loading appears to be a matched by an analogous, but essentially un-investigated and this phenomenon was investigated time to time by the following authors.
a) **Ritter and Grant** [57]

The possibility of a correspondence between migration and cyclic loading was first noted by Ritter and Grant [57] in low cycle axial fatigue experiments conducted on high purity aluminium at temperatures from 260° and 427°C. Specifically, they found an approximate one-to-one correspondence between migration steps and fatigue cycles, up to at least 36 cycles, when using specimens with grain sizes of ~ 50-100 µm, but they failed to observe this correspondence when grain size was increased to ~ 1000-1500 µm. Subsequently, Wigmore and Smith [59] reported a one-to-one correspondence of migration traces and fatigue cycles in their experiments on copper under low cycle fatigue tests at 500°C.

b) **Langdon and Gifkins** [46-48]

Experiments were conducted on high purity lead at room temperature using reverse bending and torsion fatigue at low cyclic frequencies (≤ 1.50 Hz) and observed that there was a one-to-one correspondence between the markings from grain boundary migration and the number and pattern of cyclic loading, and this correspondence is maintained up to >100 cycles. Grain boundary sliding occurs in each cycle in addition to the migration, and this leads to the development of broad triple point folds. Simultaneously, it was also (with constant strain amplitude) shown that the average distance migrated in each cycle increases as the imposed frequency is decreased and the distance migrated was often exceptionally large in the first cycle, and in addition, for large grain size (> 2000 µm), the migration markings may lead to a zig-zag pattern where the individual segments lie fairly close to 45° to the stress axis. Correspondingly, a model was also established to describe the one-to-one correspondence and which was consistent with a fine structure observed within the migration markings.

c) **Yavari and Langdon** [50-52]

Subsequently, the model of Langdon and Gifkins [48] for one-to-one correspondence of cyclic motion of grain boundaries were further improved by Yavari et.al [50] and the experiments were conducted on polycrystalline Al under conditions of low cycle fatigue at an elevated temperature of 573°K, showed that the grain boundaries migrate in a regular and cyclic manner (Fig. 2.17), and if a specimen experiences a total of \( N \) cycles of loading with or without an interruption in the test and if migration occurs only in the forward direction, it was demonstrated that each migrating boundary exhibits one faint boundary trace plus \((N + 1)\) sharp markings on the surface of the samples. Their observations were also included that migration occurs more extensively in the early stages of the cyclic deformation, which was consistent with experimental measurements under conditions of low cycle fatigue at high
Fig. 2.17. Cyclic grain boundary migration in Al after testing at 573 K with a strain amplitude of ± 0.25 % and a frequency of 1.6 x 10^{-3} Hz: (a) after 7 cycles and (b) after 11 cycles. The initial trace marked A and the final markings are labelled I and M after 7 and 11 cycles, respectively. [53]

Temperatures with a strain amplitudes less than ~ ± 0.4% and total of 11 loading cycles, the average amounts of migration (\(\bar{m}\)) followed an empirical relationship like as

\[
\bar{m} = Af^{-0.35}N^{0.5}\Delta\varepsilon^{0.66}\exp(-62.0 / RT)
\]

(2.20)

where \(f\) is the frequency, \(N\) is the number of loading cycles, \(\Delta\varepsilon\) is the strain amplitude, \(R\) is the gas constant in kJ mol^{-1} K^{-1}, \(T\) is the absolute temperature and \(A \approx 3.2 \times 10^7\) ms^{0.35}. This relationship was excellent agreement with published data up to ~ 10^4 cycles by Snowden et.al [58] but the measured activation energy for migration during fatigue of Al was significantly lower than the value anticipated for grain boundary diffusion, which was considered to be due to the various tests were not conducted under conditions of constant maximum stress.

d) **Raman and Langdon** [53-56]

In support of the above investigations, a batch of experiments were conducted on polycrystalline aluminium in reverse bending fatigue over a vast temperature range by Raman et. al. [54]. They have indicated that a majority of the grain boundaries migrated in a regular
and cyclic manner with a quantitative distribution of migration distances for different boundaries. The most remarkable part of the conclusions of their works were that the random boundaries generally migrate by large amounts whereas the non-migrating boundaries are of the coincidence type, which was characterised by using electron channelling pattern (ECP) analysis, and consistent proportion of the boundaries (~ 10% of the total boundary population) exhibited no measurable migration. In continuation, the similar experiments were conducted at other temperature range to reach a major conclusion that migration often accompanies sliding during high temperature deformations and observed that the distances migrated by the boundaries were generally many times larger than the displacements due to boundary sliding, however, there have been no definitive experimental consistency whether these two process (migration and sliding) are alternative or simultaneous. The activation energy for migration was estimated as 76.1 ± 5.0 kJ mol\(^{-1}\) (Fig. 2.16) and this is consistent with other measurements on high purity Al as published in literature [43, 57]. Grain boundary sliding occurs at both the migrating and immobile boundaries, but at the migrating boundaries the total sliding offset, measured by a projection of interference fringes across the migrated region, tends to be small. Careful inspection shows that migration usually precedes sliding at the migrating boundaries, and the migration of triple junctions leads to the formation of very diffuse triple point folds.

e) Chen and Gottstein [61-63]

Nevertheless, the flow stress behaviour and microstructural development of nickel and aluminium polycrystals were investigated during high temperature low cycle fatigue in [61]. Their works confirmed that the flow stress decreases at large number of cycles, and there was always a tendency toward grain coarsening due to extensive grain boundary migration which was also decrease as the number of cycles increases as because of the boundaries move to align 45° with respect to stress axis, but locally grain refinement owing to dynamic recrystallization was the major softening mechanism at large cumulative strains. Observations on Al, which is known not to be recrystallize dynamically due to its high staking fault energy, has shown that stress maximum has to be attributed totally to dynamic recovery and grain coarsening. After large cycle numbers the dislocations were arranged in a well recovered cell structure and the average cell size scaled with the reverse flow stress, which substantiate that dynamic recrystallization can be set off at drastically lower stress in cyclic deformation than in monotonic deformation. Finally, their results supported the hypothesis that the initiation of dynamic recrystallization was nucleation controlled rather than growth process.
Fig. 2.18. Migration steps of grain boundary after 25 deformation cycles: a is initial position and b is final position [64].

Fig. 2.19. Σ7-grain boundary after 400 deformation cycles. Deformation bands due to primary and less favoured slip system marked by white lines (DD-direction of deformation) [64].

f) Weiss and Gottstein [65-67]
In the continuation of the work on cyclic grain boundary migration, most of the efforts were concentrated for a variety of polycrystalline metallic materials at elevated temperatures rather than for bicrystal materials. With compared to the previous experiments, this study, addressed
by Weiss et. al. [64] were the first attempt to investigate the details, about the motion of grain boundaries during cyclic deformation of bicrystals of very high purity aluminium at elevated temperatures and also discussed the relevance of their results for grain boundary behaviour in polycrystals. The character of deformation structure, in particular, the crystals comprising the bicrystals were not deformed uniformly, and regimes of differently high dislocation density developed, both in neighbouring bands and both sides of the grain boundary, and this developed locally large difference in dislocation density on both sides of the grain boundary was considered to be driving force for the displacement of the boundaries. At the same time, the authors proposed a model to explain the grain boundary motion and the grain boundary alignment during cyclic deformation of aluminium bicrystals.
Chapter 3

Experimentals and Calculations

3.1 Fabrication of bicrystals

The bicrystals were produced in two simple steps. The first step was to grow single crystal with specified orientation (plane and directions) and cut them to make preferred orientation of seed crystals to generate a grain boundary in bicrystal. The second step was to join the seed crystal to polycrystalline metal melt and put them into a vertical furnace to grow bicrystal with specific grain boundary. In general, Bridgman technique was used to grow seed crystals as well as bicrystals in a vertical furnace. The Bridgman crystal growth method is basically a controlled freezing process taking place under liquid - solid equilibrium conditions. The growth also takes place under a temperature gradient, and the mechanism leads to formation of a single nucleus from which a single crystal will propagate and grow. This is achieved by allowing the solid - liquid interface to move slowly until the whole molten charge is solidified.

3.1.1 Growth of seed crystal

In the present technique, a cylindrical shaped graphite crucible was used to produce single crystals. A schematic sketch of this crucible is shown in the Fig. 3.1. The crucible consists of two parts, because of easy removal of the crystal, and both parts were made up of pure graphite due to its compatible thermal conductivity to the molten aluminium. To produce a specific orientation of single crystal, a seed crystal with a desired orientation was connected to a polycrystalline metallic billet and put them into the graphite crucible. The crucible was placed vertically and fixed it in top and bottom side in such a way that the crucible with the polycrystalline metallic charge can move through the hot zone of a vertical furnace with a constant velocity (Fig. 3.2). As the seed crystal was placed at the bottom of the vertical crucible and the melting zone was started from bottom of crucible, the partially melted seed crystal was easily welded to the metallic charge without formation of any oxide layer on the solid–liquid interface, because of the larger weight of metallic charge. In the continuation of the process, as the crucible was vertically moved away from the hot zone of the furnace, polycrystalline molten charge slowly started to crystallise with the same orientation as that of
the orientation of seed crystal until the whole polycrystalline metallic charge became single crystal. The contact area between the seed crystal and polycrystalline metallic charge was taken possibly smaller to reduce chances of formation of differently oriented grains and the furnace environment was kept inert to avoid the oxidation of graphite at high temperature.

Fig. 3.1. Sketch of graphite crucible for seed crystal and bicrystal production [29].

Fig. 3.2. Movable vertical furnace for crystal production.
3.1.2 Production of bicrystal

Single crystals of very pure aluminium (total impurity content 7.7 ppm) were grown from a polycrystal melt as discussed in section 3.1.1. To produce bicrystals with symmetrical grain boundary with specific misorientation angle $\theta^{\circ}<uvw>$, the grown seed crystal of corresponding orientations and directions of plane were cut at an angle $\theta/2$ in two parts.

![Fig. 3.3. Sketch of polycrystal billet after joining to the seed crystals.](image)

One of these two seeds was rotated on $180^{\circ}$ as $\theta/2$ along the direction of crystal growth $<hkl>$ (Fig. 3.4). After cutting and proper rotation, the seed crystals were placed into the bottom of the vertical crucible connected with a polycrystalline billet (Fig. 3.3) to decrease the possibilities of appearance of differently oriented grains, and put them into the graphite crucible. Thereafter, the same procedure was followed to produce bicrystal as in the seed crystal production (see section 3.1.1).

3.1.3 Shape and size of bicrystal samples

The grown bicrystals were cut with special shape (i.e. I-shaped) and size with two specific dimensions to be fit for the cyclic deformation machine. One type samples were cut with a dimension of about 18 mm in total length and 12 mm in gauge length, 5 mm in width and 4.5
3.2 Deformation methods and parameters

mm in thickness and other dimensions of the samples were 14 mm in total length and 8 mm in
gauge length, 5 mm in width and 4.5 mm in thickness (Fig. 3.5) and simultaneously, with

![Diagram of samples with different grain boundary geometry](image)

Fig. 3.5. Shape and size of samples with different grain boundary geometry with respect to
stress axis.

geometrically different grain boundaries with respect to the stress axis, for example, all grain
boundaries have a total range of inclination angle 0° to 90° (with 15° interval) during all
experiments. One of the most important advantages of these I-shaped specimens was easy to
polish and microscopically investigate the whole flat surface. The flat part of the gauge
section of each of these bicrystals was polished to create an area for microstructural and
microtextural investigations.

3.2 Deformation methods and parameters

After characterisation of the initial state of microstructure and texture of the bicrystals within
the polished area, all samples were deformed under low cyclic fatigue (LCF) condition at
elevated temperatures. However, the deformation plans were in two sections with respect to
the applied amplitude of stress at higher temperatures, for example, the first one was in
completely plastic deformation range and the second part was in completely elastic range.

3.2.1 Cyclic deformation machine (DZM)

Before going to detail about the deformation methods, plans and parameters, we should also
know something about Cyclic deformation machine (Druck und Zug Maschine (DZM)),
which was fabricated and build up in our institutional workshop. The working principle of
the machine was elaborated in the ‘Ph.D thesis of Sabine Weiss’ [64]. This machine was
specially upgraded for the current experiments with regard to its grips (to be compatible for
3.2 Deformation methods and parameters

3.2.2 Elastic plus plastic deformation parameters

In this group of investigations, all samples were deformed by cyclic deformation conditions at several combinations of temperatures (200°C, 300°C and 400°C) in air with total (elastic plus plastic) stress amplitudes of 2, 4, and 6 MPa at a constant cyclic frequency of $1.7 \times 10^{-2}$ Hz with sinus waveform as shown in table 3.2. The bicrystals were deformed up to a maximum of 80 cycles depending on their geometry to guarantee a uniaxial deformation. The surfaces of all samples were polished before each stage of deformation for microstructural and microtextural analysis.

Fig. 3.6. (a) Experimental set up for cyclic deformation of Al bicrystals. (b) Pneumatic grips of the cyclic deformation machine (DZM)

the new shape of the samples), heating system (to get an accurate temperature into sample), strain gauge (to get better strain measurements) to achieve a truly elastic stress of deformation (Fig. 3.6a and 3.6b). The technical specification of the machine is briefed in table 3.1.
Table 3.1. Technical Specification of cyclic deformation machine

<table>
<thead>
<tr>
<th>Component</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heated rods (four cartridge heaters per rod)</td>
<td>$T_{\text{max}} = 600^\circ\text{C}$</td>
</tr>
<tr>
<td>Pneumatic grips</td>
<td>$P_{\text{max}} = 0.1-2.5$ bar</td>
</tr>
<tr>
<td>Vacuum chamber</td>
<td>$p \leq 10^{-6}$ mbar</td>
</tr>
<tr>
<td>Load cell</td>
<td>$F_{\text{max}} = \pm 100$ N</td>
</tr>
<tr>
<td>Strain transducer</td>
<td>$\Delta l = \pm 10$ mm</td>
</tr>
<tr>
<td>Maximum cross head velocity</td>
<td>$V_{\text{max}} = 21.83 \times 10^{-3}$ mm/s</td>
</tr>
<tr>
<td>Maximum No of cycles</td>
<td>$N_{\text{max}} = 10^5$</td>
</tr>
</tbody>
</table>

Table 3.2. List of bicrystal samples and the deformation parameters.

<table>
<thead>
<tr>
<th>Sample groups</th>
<th>Initial $\theta$ [°]</th>
<th>Deformation Temperature [°C]</th>
<th>Amplitude of stress [MPa]</th>
<th>Number of cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group1</td>
<td>11.7 ± 0.9</td>
<td>200</td>
<td>4</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>14.4 ± 0.8</td>
<td>300</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>15.4 ± 0.5</td>
<td>400</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Group2</td>
<td>11.9 ± 0.4</td>
<td>2</td>
<td>4</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>13.8 ± 0.5</td>
<td>4</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>15.9 ± 0.5</td>
<td>6</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>30.3 ± 0.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Group3</td>
<td>13.2 ± 0.3</td>
<td>300</td>
<td>4</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td>14.7 ± 0.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>17.1 ± 0.6</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>23.6 ± 0.5</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.2.3 Completely elastic deformation parameters

In these experiments, bicrystals containing several type of grain boundaries with a range of misorientation angles from 7° to 42.5° with $<112>$ tilt, $<100>$ tilt and $<100>$ twist rotational axis (table 3.3) were investigated. All samples were deformed by cyclic deformation machine at several combinations of temperatures (370°C to 460°C) in air with total (elastic) stress amplitudes of $\pm 0.5$ MPa at a constant cyclic frequency of $1.7 \times 10^{-2}$ Hz with sinus waveform. The bicrystal samples were deformed up to a maximum of 20 cycles depending on their geometry to guarantee a uniaxial deformation.

In aluminium of purity 99.999 at% the yield stress, using the same machine as that used for cyclic deformation, were found in the range of 12 MPa to 2.5 MPa at room temperature
3.2 Deformation methods and parameters

Table 3.3: List of samples and deformation parameters.

<table>
<thead>
<tr>
<th>Sample groups</th>
<th>Initial $\theta$ [°] &lt;112&gt; tilt axis</th>
<th>Temperature (T) [°C]</th>
<th>Stress ($\pm \sigma$) [MPa]</th>
<th>Number of cycles (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;112&gt; tilt axis</td>
<td>GB $\perp$ to stress axis</td>
<td>GB $\parallel$ to stress axis</td>
<td>GB $\angle 45^\circ$ to stress axis</td>
<td>GB $\angle 15^\circ$-75$^\circ$ to stress axis</td>
</tr>
<tr>
<td>6.3</td>
<td>7.6</td>
<td>7.7</td>
<td>5.3</td>
<td>370</td>
</tr>
<tr>
<td>7.4</td>
<td>12.5</td>
<td>12.7</td>
<td>7.8</td>
<td>385</td>
</tr>
<tr>
<td>7.9</td>
<td>13.6</td>
<td>14.6</td>
<td>8.5</td>
<td>400</td>
</tr>
<tr>
<td>8.5</td>
<td>14.6</td>
<td>16.5</td>
<td>29.2</td>
<td>425</td>
</tr>
<tr>
<td>13.7</td>
<td>16.6</td>
<td>16.2</td>
<td>31.7</td>
<td>450</td>
</tr>
<tr>
<td>14.2</td>
<td>15.8</td>
<td>29.3</td>
<td></td>
<td>460</td>
</tr>
<tr>
<td>15.5</td>
<td>28.6</td>
<td>30.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>16.8</td>
<td>30.2</td>
<td>41.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>30.9</td>
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<td>30.5</td>
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<td>30.7</td>
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<tr>
<td>42.1</td>
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<td></td>
</tr>
<tr>
<td>3.7</td>
<td>3.7</td>
<td></td>
<td>370</td>
<td>0.5</td>
</tr>
<tr>
<td>6.8</td>
<td>6.6</td>
<td></td>
<td>385</td>
<td></td>
</tr>
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<td>9.2</td>
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<td></td>
</tr>
<tr>
<td>12.7</td>
<td>34.5</td>
<td></td>
<td>450</td>
<td></td>
</tr>
<tr>
<td>13.5</td>
<td>42.5</td>
<td></td>
<td>460</td>
<td>0.5</td>
</tr>
<tr>
<td>16.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>17.5</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>22.2</td>
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</tr>
<tr>
<td>30.1</td>
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<tr>
<td>42.5</td>
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<tr>
<td>5.9</td>
<td>9.3</td>
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<tr>
<td>7.9</td>
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<tr>
<td>9.2</td>
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<tr>
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</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>460</td>
</tr>
</tbody>
</table>

Fig. 3.7. (a) Microstructure of elastically (450°C, 0.5MPa. 20 noc) deformed bicrystal, where, no sign of slip or deformation band into structure (b) Misorientation profile of elastically (450°C, 0.5MPa. 20 noc) deformed bicrystal, where, no misorientation gradient developed.
and at elevated temperatures, respectively. Moreover, to be sure of elastic range of deformation, the samples were examined under optical microscope (Fig. 3.7a), where is no sign of slip band and deformation band formation, and misorientation profile across the grain boundary were drawn by EBSD mapping using scanning electron microscope (Fig. 3.7b), which are not showing any misorientation gradient.

### 3.3 Measurement of GB displacement and electron microscopy

After deformation and cooling to room temperature, the specimens were subjected to microstructural and microtextural characterisation. The orientations in the vicinity of the grain boundary were measured for each processing stage by EBSD technique using a scanning electron microscope. For the moving grain boundaries, each deformation cycle was accompanied by traces on the surface of the sample so that the displacement of the boundary could be monitored by image analysing technique using standard optical microscope.

#### 3.3.1 Measuring parameters during plastic deformation

In this part of experiments, some special parameters were calculated to index the driving force during grain boundary motion under plastic amplitude of stress. Therefore, from these EBSD orientations data, the average point-to-point misorientation θ₁ in one grain and θ₂ in the other grain, near the grain boundary were calculated to show the misorientation difference (θ₁ - θ₂ = Δθ) between these grains (Fig. 3.8a). At the same time, misorientation profile and Schmid factors at different distance of these two grains were also calculated to show inhomogeneous deformation structures into the grains (Fig. 3.8b).

From optical micrographs, visible slip lines aligned nearly 45° to the grain boundary, which were formed during deformation into the grains, were counted to evaluate the slip density difference (Δfᵢ) of each case (Fig. 3.8b). This (Δfᵢ) factor can be defined as the visible slip line density difference per unit length along the grain boundary. For examples, from (Fig. 3.8b),

\[
Δfᵢ = f_{g₁} - f_{g₂}
\]

(3.1)

where, \(f_{g₁}\) and \(f_{g₂}\) are the number of slip lines in grain1 and grain2 per unit length along the grain boundary, respectively. It can be correlated with the dislocation density difference between the two grains during deformation. Therefore, slip density difference is an important index to discuss about the driving force of grain boundary migration.
3.3.2 Measuring parameters during elastic deformation

3.3.2.1 Driving force without considering the shear stress component

If we consider that no shear stress component exists and the grain boundary motion was only occurs only due to normal stress, thus, the force acting on dislocation can be estimated by the following way. For a given stress tensor \( \sigma \), Burgers vector \( b \) and line element \( s \) of the dislocation, the force per unit length on the dislocation can be calculated by Peach-Koehler equation [41], like follows,

\[
F = \sigma \times b
\]  \hspace{1cm} (3.2)

As we assumed that there was no shear stress component (Fig. 3.9), the stress tensor can be composed as

\[
\sigma = \begin{pmatrix}
\sigma_{xx} & 0 & 0 \\
0 & 0 & 0 \\
0 & 0 & 0
\end{pmatrix}
\]  \hspace{1cm} (3.3)

With taking same co-ordinate system of Burgers vector \( b_1 \) and \( b_2 \), the dislocation line \( s \) as of the stress tensor (Fig. 3.9). Where,

\[
b_2 = \begin{pmatrix}
\frac{b \cos \theta}{2} \\
\frac{b \sin \theta}{2} \\
0
\end{pmatrix}, \quad b_1 = \begin{pmatrix}
\frac{b \cos \theta}{2} \\
\frac{b \sin \theta}{2} \\
0
\end{pmatrix} \quad \text{and} \quad s = \begin{pmatrix}
0 \\
0 \\
-1
\end{pmatrix}
\]  \hspace{1cm} (3.4)

From the equations (3.3) and (3.4) the force on the dislocations can be summarised as

\[
F_{1 \text{dis}} = \sigma_{xx} b \cos \theta / 2
\] \hspace{1cm} and \hspace{1cm}

\[
F_{2 \text{dis}} = \sigma_{xx} b \cos \theta / 2
\]  \hspace{1cm} (3.5)
Fig. 3.9. The calculation of driving force for cyclic motion of grain boundary without considering the shear stress component.

It can be seen from equation (3.5) that in this case there will be a motion of the dislocations only in y-direction, i.e. parallel to the grain boundary plane and not in x-direction, i.e. perpendicular to the GB plane, since the force component in x-direction is zero. However, our present experiments have convincingly showed that the GB moves along the direction normal to the GB plane, so there must be a force on dislocation perpendicularly acting along x-direction; this is only possible if there is a shear stress component acting during deformation. The driving force with considering shear component can be elaborated in the following section.

3.3.2.2 Driving force with considering the shear stress component

In this part of experiments, the driving forces for cyclic motion were calculated in a classical way for the geometrically different grain boundary motions. A schematic sketch of the experimental set-up for applying cyclic stress to the grain boundary has been shown in Fig. 3.10. The ‘T’ shaped bicrystal samples were put into a temperature controlled heating stage of grips and fixed by an internal piston from both, upper and lower sides. Only the lower grip was movable and computer controlled, and the applied stress on the sample i.e. on the grain boundary was generated by moving the lower grip.
The observations of grain boundary motion were performed by taking a segment of grain boundary from a grown bicrystal casting, and locate the initial and final position of grain boundary after cyclic deformation under a optical microscope. The distance between the initial and final position of grain boundary was defined as the total displacement of grain boundary, and velocity of boundary motion \( (v) \) is defined as total displacement \( (m) \) divided by total time \( (t) \) to complete whole input cycles for deformation, i.e \( v = \frac{m}{t} \).

In general, every symmetrical tilt boundary can be described as an alignment of a group of edge dislocations and in turn, the motion of grain boundary can be considered as displacement of these edge dislocations due to the applied stress on the sample or more precisely, on grain boundary. Therefore, the force on a single dislocation would be the driving force for grain boundary motion. But, if we consider normal force acting on the single dislocation on grain boundary and try to calculate the driving force for cyclic motion, it results zero component on the dislocation. However grain boundary motion was convincingly detected in every case, which indicates that there is some force acting, that causes motion. We have examined the other testing machines and found that the two grips of the machine are never be aligned in a perfect straight line, rather, there was always some tiny misalignment between the two grips, that means, there was always a shear stress acting on the sample as well as on the grain boundaries (Fig. 3.10 and Fig. 3.11).

![Schematic sketch of cyclic deformation machine grips and direction of deformation.](image-url)
For a given stress tensor \( (\sigma) \), Burgers vector \((b)\) and line element \((s)\) of the dislocation, the force per unit length on the dislocation can be calculated by Peach-Koehler equation \([41]\), like as,
\[
F = (\sigma \cdot b) \times s
\]
(3.6)

**a) Driving force for the motion of perpendicular boundaries**

In these experiments, shear stress was acting parallel to grain boundary plane (Fig 3.11a), although, the grain boundary plane was always normal to the applied stress axis. The stress tensor can be composed as
\[
\sigma = \begin{pmatrix}
\sigma_{xx} & \tau_{xy} & \tau_{xz} \\
\tau_{xy} & 0 & 0 \\
\tau_{xz} & 0 & 0
\end{pmatrix} \quad (3.7)
\]
with taking same co-ordinate system of Burgers vector \(b_1\) and \(b_2\), the dislocation line \(s\) as of the stress tensor (Fig. 3.11a). Where,
\[
b_2 = \begin{pmatrix}
b \cos \theta / 2 \\
b \sin \theta / 2 \\
0
\end{pmatrix}, \quad b_1 = \begin{pmatrix}
b \cos \theta / 2 \\
b \sin \theta / 2 \\
0
\end{pmatrix} \quad \text{and} \quad s = \begin{pmatrix}
0 \\
0 \\
-1
\end{pmatrix} \quad (3.8)
\]

From the equations (3.7) and (3.8) the force on the dislocations can be summarised as
\[
F_{1}^{\text{dis}} = \begin{pmatrix}
-\tau_{xy} b \cos \theta / 2 \\
\sigma_{xx} b \cos \theta / 2 + \tau_{xy} b \sin \theta / 2 \\
0
\end{pmatrix} \quad \text{and}
\]
\[
F_{2}^{\text{dis}} = \begin{pmatrix}
-\tau_{xy} b \cos \theta / 2 \\
\sigma_{xx} b \cos \theta / 2 - \tau_{xy} b \sin \theta / 2 \\
0
\end{pmatrix} \quad (3.9)
\]

From the equation (3.9) it is clear that the force acting on dislocations is only operative in \(x\)-direction and \(y\)-directions, therefore, the motion of dislocations is only possible either parallel and/or perpendicular to the grain boundary plane. Since, in experiments on perpendicular geometry, the grain boundary motion was found along the \(x\)-direction, thus, the force normals to the boundary, \( (F_{x}^{\text{dis}})_{1} \) and \( (F_{x}^{\text{dis}})_{2} \) cause the dislocations to move and make the whole boundary mobile. With taking \(\tau_{xy} \equiv \tau\), the force on dislocations
\[
F_{\text{dis}} = -\tau b \cos \theta / 2 \quad (3.10)
\]

As we know that each tilt boundary can be formally described by an arrangement of edge dislocations \([68]\), therefore, the force per unit grain boundary area is given by the product of
the force on a single dislocation per unit length and the length of dislocation line per unit area in grain boundary $\rho_{\text{dis}}$,

$$ p = \rho_{\text{dis}} F_{\text{dis}} = -\rho_{\text{dis}} \frac{tb \cos \theta}{2} \tag{3.11} $$

Fig. 3.11. Schematic diagrams for the forces acting on the dislocations of grain boundaries in a) perpendicular b) parallel c) $45^\circ$ inclined to the stress axis during cyclic deformation.

For symmetrical tilt boundaries, the dislocation density can be determined by

$$ \rho_{\text{dis}} = \frac{1}{D} = \frac{2 \sin \theta / 2}{b} \tag{3.12} $$

Thus, from the equations (3.11) and (3.12), we can obtain the driving force on the grain boundary like as

$$ p = -\rho_{\text{dis}} \frac{tb \cos \theta / 2}{b} = -\frac{2 \sin \theta / 2}{b} \frac{tb \cos \theta / 2}{b} = -\tau \sin \theta \tag{3.13} $$

b) Driving force for the motion of parallel boundaries

As the grain boundary were parallel to the applied stress axis, the shear stress was acting just normal to the grain boundary plane (Fig. 3.11b). The stress tensor can be written as
with taking same co-ordinate system of Burgers vector \( b_1 \) and \( b_2 \), the dislocation line \( s \) as of the stress tensor. Where, \( b_1 = \begin{pmatrix} -b \sin \theta / 2 \\ b \cos \theta / 2 \\ 0 \end{pmatrix} \), \( b_2 = \begin{pmatrix} b \sin \theta / 2 \\ b \cos \theta / 2 \\ 0 \end{pmatrix} \) and \( s = \begin{pmatrix} 0 \\ 0 \\ -1 \end{pmatrix} \)

From the equations (3.14) and (3.15) the force on dislocations can be represented as

\[
F_{1 \text{\,dis}} = \begin{pmatrix} \tau_{xy} b \sin \theta / 2 \\ -\sigma_{xx} b \sin \theta / 2 + \tau_{xy} b \cos \theta / 2 \\ 0 \end{pmatrix}
\]
\[
F_{2 \text{\,dis}} = \begin{pmatrix} -\tau_{xy} b \sin \theta / 2 \\ \sigma_{xx} b \sin \theta / 2 + \tau_{xy} b \cos \theta / 2 \\ 0 \end{pmatrix}
\]

From the equation (3.16) it is clear that the force acting on dislocations is only operative in \( x \)-direction and \( y \)-directions. Therefore, dislocations can move either parallel and/or perpendicular to the grain boundary plane. Since, in the experiments on parallel geometry, the grain boundary motion is possible in \( y \)-direction, thus, the force normal to the boundary \( F_{y \text{\,dis}} \), and \( F_{y \text{\,dis}} \) should be in same direction to move the whole boundary. From the equation (3.16), it is possible only if, \( \tau_{xy} b \cos \theta / 2 \geq \sigma_{xx} b \sin \theta / 2 \). Therefore, taking \( \tau_{xy} = \tau \) and \( \sigma_{xx} = \sigma \), we get a motion in \( y \)-direction only if the force acting along \( y \)-direction is

\[
\tau \geq \sigma \tan \theta / 2
\]

As we know that each tilt boundary can be formally described by an arrangement of edge dislocations [68], therefore, the force per unit grain boundary area is given by the product of the force on a single dislocation per unit length and the dislocation line length per unit area in grain boundary \( \rho_{\text{dis}} \).

The driving force \( (p) \) can be obtain by the equation (3.18)

\[
p = \rho_{\text{dis}} F_{\text{dis}}
\]

and the dislocation density for symmetrical tilt boundaries

\[
\rho_{\text{dis}} = \frac{1}{D} = \frac{2 \sin \theta / 2}{b}
\]
Combining equations (3.17), (3.18) and (3.19), we can obtain the driving force on the grain boundary while the boundary is perpendicular to stress axis

\[ p \geq \rho_{\text{amb}} (\sqrt{b} \cos \theta / 2 + \sqrt{\sigma} \sin \theta / 2) \geq 2 \sigma \sin^2 \theta / 2 \]  

(3.20)

From the equation (3.17) we can obtain a relation between shear stress and normal stress acting on grain boundary and equation (3.20) is the preliminary condition for the motion of grain boundary with parallel geometry.

The generated shear stress \( \tau \) exerts a force on the grain boundary and causes the boundary to move along the y-direction with a velocity \( (v) \), from this measured velocity \( (v) \), the grain boundary mobility can be calculated using the equation (3.21)

\[ v = mp \]  

(3.21)

The grain boundary motion under external stress is a thermally activated process as seen from the Arrhenius equation, thus, grain boundary mobility can be calculated as

\[ m = \frac{v}{p} = m_0 \exp \left( -\frac{\Delta H}{kT} \right) \]  

(3.22)

and then taking Logarithm both sides

\[ \ln \frac{v}{p} = \ln m_0 - \frac{\Delta H}{kT} \]  

(3.23)

where, \( p \) is driving force, \( v \) is the velocity and \( m \) is the mobility of grain boundary, \( \Delta H \) is activation enthalpy for migration, \( T \) is absolute temperature, \( k \) is Boltzman constant and \( m_0 \) is pre-exponential factor.

Correspondingly, the velocity and applied force are time dependent factor for cyclic deformation and can be described by the following equations

\[ v(t) = m_0 \cdot \exp \left( -\frac{\Delta H}{kT} \right) \cdot p(t) \]  

(3.24)

\[ p(t) = p_0 \cdot \sin(2\pi \vartheta t) \]  

(3.25)

where, \( v(t) \) is the time dependent velocity of grain boundary, \( p(t) \) is the time dependent driving force and \( \vartheta \) is the frequency of loading.

These are the pretended driving forces for this type of cyclic motion, but the crucial point is to know how much of total applied force contributed to this shear stress. Therefore, we shall compare our present result with the previously obtained results on pure shear stress driven grain boundary motion [29-33] in order to make a clear picture of the cyclic motion mechanisms.

c) Driving force for the motion 45° inclined boundaries
As the grain boundaries were 45° inclined to the applied stress axis, a certain amount of shear stress was acting to the grain boundary plane (Fig. 3.11c).

In that case, stress tensor can be projected as

\[
\sigma = \begin{pmatrix}
\sigma_{xx} & \tau_{xy} & \tau_{xz} \\
\tau_{xy} & 0 & 0 \\
\tau_{xz} & 0 & 0
\end{pmatrix}
\] (3.26)

and the burger vectors \( b_1 \) and \( b_2 \) and line element of dislocation \( s \) will be

\[
b_1 = \begin{pmatrix}
b \cos(\pi/4 - \theta/2) \\
-b \sin(\pi/4 - \theta/2) \\
0
\end{pmatrix}, \quad b_2 = \begin{pmatrix}
b \cos(\pi/4 + \theta/2) \\
-b \sin(\pi/4 + \theta/2) \\
0
\end{pmatrix} \quad \text{and} \quad s = \begin{pmatrix} 0 \\ 0 \\ -1 \end{pmatrix}
\] (3.27)

From the equations (3.26) and (3.27) the force on the dislocations can be described as

\[
F_{1z}^{\text{dis}} = \begin{pmatrix}
-\tau_{xy} b \cos(\pi/4 - \theta/2) \\
\sigma_{xx} b \cos(\pi/4 - \theta/2) - \tau_{xy} b \sin(\pi/4 - \theta/2) \\
0
\end{pmatrix}
\] \quad \text{and}

\[
F_{2z}^{\text{dis}} = \begin{pmatrix}
-\tau_{xy} b \cos(\pi/4 + \theta/2) \\
\sigma_{xx} b \cos(\pi/4 + \theta/2) - \tau_{xy} b \sin(\pi/4 + \theta/2) \\
0
\end{pmatrix}
\] (3.28)

From the equation (3.28) it is clear that the force acting on dislocations is only operative in \( x \)-direction and \( y \)-directions. Therefore, the motion of dislocations is possible either parallel or/and perpendicular to the grain boundary plane. Since, in experiments on 45° inclined geometry, the grain boundary displacement is anticipated along the \( x \)-direction as well as \( y \)-direction, thus, the product of \( (F_{x}^{\text{dis}})_1 \) and \( (F_{x}^{\text{dis}})_2 \) as well as \( (F_{y}^{\text{dis}})_1 \) and \( (F_{y}^{\text{dis}})_2 \) should be positive, i.e., \( (F_{x}^{\text{dis}})_1 \cdot (F_{x}^{\text{dis}})_2 \geq 0 \) and \( (F_{y}^{\text{dis}})_1 \cdot (F_{y}^{\text{dis}})_2 \geq 0 \).

In case of \( (F_{x}^{\text{dis}})_1 \cdot (F_{x}^{\text{dis}})_2 \geq 0 \), and considering \( \tau_{xy} \equiv \tau \), the force on dislocations

\[
(tb \cos(\pi/4 - \theta/2))(tb \cos(\pi/4 + \theta/2)) \geq 0
\] (3.29)

The solution of the equation (3.29) will be

\[
\frac{\tau^2 b^2}{2} \left( \cos \frac{\pi}{2} + \cos \theta \right) \geq 0 \quad \text{or} \quad \frac{\tau^2 b^2}{2} \cos \theta \geq 0
\] (3.30)

Therefore, the equation (3.30) is a preliminary condition for the motion of 45° inclined grain boundary along the \( x \)-direction.
As we know that $\sigma_{xx} \neq 0$, therefore, there is always a possibility that a force acting along the y-direction results in the boundary movement along y-direction. Thus, in case of $(F_{y}^{disy})_1$ $(F_{y}^{disy})_2 \geq 0$, with taking $\tau_{xy} \equiv \tau$, and $\sigma_{xx} = \sigma$ the force on dislocations

$$(\sigma b \cos(\pi / 4 - \theta / 2) - \tau b \sin(\pi / 4 - \theta / 2))(\sigma b \cos(\pi / 4 + \theta / 2) - \tau b \sin(\pi / 4 + \theta / 2)) \geq 0 \quad (3.31)$$

with taking the simplified form of the equation (3.31) we can get

$$(\sigma^2 + \tau^2 ) \cos \theta - 2 \sigma \tau \geq 0 \quad (3.32)$$

Similarly, the equation (3.32) is a preliminary condition for the motion of $45^\circ$ inclined grain boundary along the y-direction.
Chapter 4

Results

4.1 Cyclic motion of $<112>$ tilt boundary under plastic deformation

This section of experimental results focuses on the high temperature cyclic stress response of high purity aluminium bicrystals, which were investigated by means of quasi in-situ measurements of the $<112>$ tilt grain boundary as well as the grain interior (i.e. inspecting the same area of the bicrystal samples before and after the deformation) at different combination of cyclic deformation parameters. Particular attention has been focussed towards the effect of cyclic stress at elevated temperatures on grain orientation during deformation and misorientation angle variation due to grain boundary motion, and at the same time it was clarified that the dislocation density difference was the prime source of driving force for the grain boundary motion.

![Initial grain boundary (GB) position with deformation direction (DD) (T and B represent the two grains in the bicrystal).](image)

4.1.1 Grain boundaries deformed at different temperatures (T)

This group of samples contains low angle as well as high angle grain boundaries. Before cyclic deformation, the initial position and character of the grain boundary and the
orientations of the two grains in the vicinity of the grain boundary were determined. The nature of all boundaries were nearly planar and perpendicular to the applied stress axis (Fig. 4.1). All boundaries were subjected to deformation at 200°C, 300°C and 400°C with constant amplitude of stress of 4 MPa and with 20 cycles.

Fig. 4.2. {111} pole figure of grain orientations inside bicrystals with <112> tilt grain boundaries and misorientation angles of 11.7° (a) before deformation and after deformation at (b) 200°C (c) 300°C (d) 400°C (T and B represent the two grains in the bicrystal).
Fig. 4.3. \{111\} pole figure of grain orientations inside bicrystals with \(<112>\) tilt grain boundaries and misorientation angles of 32.3° (a) before deformation and after deformation at (b) 200°C (c) 300°C (d) 400°C (T and B represent the two grains in the bicrystal).

4.1.1.1 Grain boundary with small misorientation angle (11.7°)

The \{111\}-pole figure (Fig. 4.2) shows the grain orientations of the bicrystal sample before and after the deformation. The misorientation angle was changed during the deformation in the range of 10.9° and 12.8°. The optical micrographs (Fig. 4.4) of this grain boundary and its vicinity before and after deformation at 200°C, 300°C and 400°C show that there was no
4.1 Cyclic motion of $<112>$ tilt boundaries under plastic deformation

Grain boundary motion during deformation, although, the $\Delta f_i$ slightly increases at 300°C and 400°C as shown in Table 4.1. The surface revealed an increase in slip lines with increasing temperature of deformation.

Fig. 4.4. Microstructure of LAGB (11.7°$<112>$ tilt axis) (a) before deformation (b) after deformation at 200°C (c) 300°C and (d) 400°C.

This deformation structure was accompanied by a misorientation angle development along the grain interior. The differences in Schmid factors in both the grains were less and increases from 0.006 during the deformation at 200°C to 0.015 during the deformation at 400°C (Table 4.3).
4.1 Cyclic motion of \(<112>\) tilt boundaries under plastic deformation

**Fig. 4.5.** Microstructure of HAGB (32.3°\(<112>\) Tilt axis) (a) before deformation (b) after deformation at 200°C (c) 300°C and (d) 400°C.

**Table 4.1:** List of parameters during cyclic grain boundary migrations at different deformation temperatures.

<table>
<thead>
<tr>
<th>Deformation No</th>
<th>Average GB displacement (\delta (\mu m))</th>
<th>(\Delta f_i) (diff. slip nos/(\mu m))</th>
<th>Deformation conditions</th>
<th>Misori. after deformation ((\theta^o)) (&lt;112&gt;) tilt axis</th>
<th>Initial Misori. ((\theta^o)) (&lt;112&gt;) tilt axis</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st</td>
<td>No motion</td>
<td>No slip band found</td>
<td>200°C, 4 MPa 20 Noc</td>
<td>10.9 ± 0.7</td>
<td>11.7 ± 0.9</td>
</tr>
<tr>
<td>2nd</td>
<td>No motion</td>
<td>0.001</td>
<td>300°C, 4 MPa 20 Noc</td>
<td>11.9 ± 0.7</td>
<td>14.4 ± 0.8</td>
</tr>
<tr>
<td>3rd</td>
<td>No motion</td>
<td>0.001</td>
<td>400°C, 4 MPa 20 Noc</td>
<td>12.8 ± 0.9</td>
<td>14.4 ± 0.5</td>
</tr>
<tr>
<td>1st</td>
<td>No motion</td>
<td>No slip band found</td>
<td>200°C, 4 MPa 20 Noc</td>
<td>14.4 ± 0.6</td>
<td></td>
</tr>
<tr>
<td>2nd</td>
<td>27.3</td>
<td>0.003</td>
<td>300°C, 4 MPa 20 Noc</td>
<td>12.9 ± 0.5</td>
<td>14.4 ± 0.8</td>
</tr>
<tr>
<td>3rd</td>
<td>29.3</td>
<td>0.004</td>
<td>400°C, 4 MPa 20 Noc</td>
<td>13.5 ± 0.4</td>
<td>15.4 ± 0.5</td>
</tr>
<tr>
<td>1st</td>
<td>No motion</td>
<td>0.0006</td>
<td>200°C, 4 MPa 20 Noc</td>
<td>14.5 ± 0.8</td>
<td></td>
</tr>
<tr>
<td>2nd</td>
<td>65.5</td>
<td>0.004</td>
<td>300°C, 4 MPa 20 Noc</td>
<td>14 ± 1.2</td>
<td>32.3 ± 0.5</td>
</tr>
<tr>
<td>3rd</td>
<td>35.1</td>
<td>0.003</td>
<td>400°C, 4 MPa 20 Noc</td>
<td>14.4 ± 0.9</td>
<td></td>
</tr>
<tr>
<td>1st</td>
<td>No motion</td>
<td>No slip band found</td>
<td>200°C, 4 MPa 20 Noc</td>
<td>31.4 ± 0.5</td>
<td></td>
</tr>
<tr>
<td>2nd</td>
<td>28.8</td>
<td>0.003</td>
<td>300°C, 4 MPa 20 Noc</td>
<td>32.1 ± 0.5</td>
<td></td>
</tr>
<tr>
<td>3rd</td>
<td>121.6</td>
<td>0.006</td>
<td>400°C, 4 MPa 20 Noc</td>
<td>28.8 ± 0.9</td>
<td></td>
</tr>
</tbody>
</table>

### 4.1.1.2 Grain boundary with larger misorientation angle (32.3°)

The results for bicrystals containing a high angle grain boundary (HAGB) of 32.3°<112> misorientation are presented here. The bicrystals were deformed under different temperatures as done in the previous experiments. The grain orientations of the bicrystal were changed during the deformation (Fig. 4.3). The misorientation angle was changed in the range of 28.8°
and 32.3°. The micrographs (Fig. 4.5c and d) show that grain boundary motion and slip line bands were formed for deformation at 300°C and 400°C. In this case, the maximum grain boundary displacement ($\delta \sim 122 \ \mu m$) has been observed at the deformation temperature 400°C and simultaneously, the maximum $\Delta f_i$ value has been noticed at 400°C (Table 4.1). The grain boundary displacement increases with increasing value of the difference of slip density ($\Delta f_i$) and Schmid factors.

Fig. 4.6. $\{111\}$ pole figure of grain orientations inside bicrystals with $\langle112\rangle$ tilt grain boundaries and misorientation angles of 11.9° (a) before deformation and, after deformation at (b) 2MPa (c) 4MPa (d) 6 MPa (T and B represent the two grains in the bicrystal).
4.1 Cyclic motion of $<112>$ tilt boundaries under plastic deformation

Fig. 4.7. $\{111\}$ pole figure of grain orientations inside bicrystals with $<112>$ tilt grain boundaries and misorientation angles of 30.3° (a) before deformation and, after deformation at (b) 2MPa (c) 4MPa (d) 6 MPa (T and B represent the two grains in the bicrystal).

4.1.2 Grain boundaries deformed at different amplitude of stress ($\sigma_a$)

This group of samples contains low angle as well as high angle grain boundaries. The initial orientations and status of the grain boundary were monitored by optical as well as scanning electron microscope. All bicrystals were deformed under three various amplitudes of stress (i.e. 2, 4 and 6 MPa) at same temperature (300°C) and constant number of cycles (20) at a constant frequency of loading ($1.7 \times 10^{-2}$ Hz) with sinus waveform. After each step of cyclic
deformation, the samples were examined by optical microscopy as well as by EBSD using
scanning electron microscope.

4.1.2.1 Grain boundary with smaller misorientation angle (11.9°)

This bicrystal contains a grain boundary with a misorientation angle of 11.9° and <112> tilt axis (Fig. 4.6a), which was deformed under different amplitudes of stress with constant
temperature and numbers of cycle. There was measurable change in structure of the two
grains after the deformation (Fig. 4.6b-d). The misorientation angle was changed from 11.9°
to 15.8°.

Table 4.2. List of parameters during cyclic grain boundary migrations at different
deformation amplitude of stress.

<table>
<thead>
<tr>
<th>Deformation No.</th>
<th>Average GB displacement δ (µm)</th>
<th>∆f_i (diff. slip nos/µm)</th>
<th>Deformation conditions</th>
<th>Misori. after deformation (θ°) &lt;112&gt; tilt axis</th>
<th>Initial Misori. (θ°) &lt;112&gt; tilt axis</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st</td>
<td>20.2</td>
<td>0.0035</td>
<td>2 MPa, 300°C, 20 noc</td>
<td>15.2 ± 0.5</td>
<td>11.9 ± 0.4</td>
</tr>
<tr>
<td>2nd</td>
<td>52.4</td>
<td>0.004</td>
<td>4 MPa, 300°C, 20 noc</td>
<td>15.8 ± 0.2</td>
<td></td>
</tr>
<tr>
<td>3rd</td>
<td>No motion</td>
<td>0.002</td>
<td>6 MPa, 300°C, 20 noc</td>
<td>14.6 ± 0.6</td>
<td></td>
</tr>
<tr>
<td>1st</td>
<td>No motion</td>
<td>No slip band found</td>
<td>2 MPa, 300°C, 20 noc</td>
<td>13.4 ± 0.2</td>
<td>13.8 ± 0.5</td>
</tr>
<tr>
<td>2nd</td>
<td>No motion</td>
<td>0.003</td>
<td>4 MPa, 300°C, 20 noc</td>
<td>10.6 ± 0.5</td>
<td></td>
</tr>
<tr>
<td>3rd</td>
<td>No motion</td>
<td>0.004</td>
<td>6 MPa, 300°C, 20 noc</td>
<td>12.6 ± 0.5</td>
<td></td>
</tr>
<tr>
<td>1st</td>
<td>No motion</td>
<td>No slip band found</td>
<td>2 MPa, 300°C, 20 noc</td>
<td>15.6 ± 0.8</td>
<td>15.9 ± 0.5</td>
</tr>
<tr>
<td>2nd</td>
<td>206.7</td>
<td>0.009</td>
<td>4 MPa, 300°C, 20 noc</td>
<td>15.8 ± 0.5</td>
<td></td>
</tr>
<tr>
<td>3rd</td>
<td>70.9</td>
<td>0.004</td>
<td>6 MPa, 300°C, 20 noc</td>
<td>14.9 ± 0.3</td>
<td></td>
</tr>
<tr>
<td>1st</td>
<td>26.5</td>
<td>0.0042</td>
<td>2 MPa, 300°C, 20 noc</td>
<td>29.8 ± 0.7</td>
<td>30.3 ± 0.5</td>
</tr>
<tr>
<td>2nd</td>
<td>35.6</td>
<td>0.0038</td>
<td>4 MPa, 300°C, 20 noc</td>
<td>28.8 ± 0.9</td>
<td></td>
</tr>
<tr>
<td>3rd</td>
<td>20.6</td>
<td>0.003</td>
<td>6 MPa, 300°C, 20 noc</td>
<td>31.6 ± 0.5</td>
<td></td>
</tr>
</tbody>
</table>

As this grain boundary is in the transition range of LAGB and HAGB (Fig. 4.6a), therefore,
the behaviour of this grain boundary was quite different from other low angle boundaries,
because, the grain boundary was moved over a large distance even in the case of low
amplitude of stress (Fig. 4.8) and the largest displacement was found at 4 MPa stress of
deformation with a very less variation of ∆f_i value (Table 4.2). The difference of Schmid
factors was very small at 6 MPa stress of deformation (~0.009) as shown in Table 4.4.
4.1 Cyclic motion of $<112>$ tilt boundaries under plastic deformation

Fig. 4.8. Microstructure of a LAGB (11.9°$<112>$ tilt axis) (a) before deformation (b) after deformation with 2 MPa (c) 4 MPa and (d) 6 MPa stress amplitude
4.1 Cyclic motion of \(<112>\) tilt boundaries under plastic deformation

Fig. 4.9. Microstructure of HAGB (30.3°<112> tilt axis) (a) before deformation (b) after deformation with 2 MPa(c) 4MPa and (d) 6 MPa stress amplitude.

Table 4.3. List of Schmid-factors for all grains into the bicrystal samples, deformed at different temperatures.

<table>
<thead>
<tr>
<th>Initial misori. (θ°) &lt;112&gt; tilt axis</th>
<th>Deformation no</th>
<th>Schmid-factor (m)</th>
<th>Difference of Schmid-factors</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Grain I</td>
<td>Grain II</td>
</tr>
<tr>
<td>11.7 ± 0.9</td>
<td>1</td>
<td>0.418</td>
<td>0.412</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.428</td>
<td>0.414</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.405</td>
<td>0.390</td>
</tr>
<tr>
<td>14.4 ± 0.8</td>
<td>1</td>
<td>0.321</td>
<td>0.314</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.417</td>
<td>0.391</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.353</td>
<td>0.318</td>
</tr>
<tr>
<td>15.4 ± 0.5</td>
<td>1</td>
<td>0.336</td>
<td>0.328</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.410</td>
<td>0.385</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.374</td>
<td>0.328</td>
</tr>
<tr>
<td>32.3 ± 0.5</td>
<td>1</td>
<td>0.422</td>
<td>0.432</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.414</td>
<td>0.437</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.397</td>
<td>0.444</td>
</tr>
</tbody>
</table>

Table 4.4. List of Schmid-factors for all grains into the bicrystal samples, deformed at different amplitude of stress.

<table>
<thead>
<tr>
<th>Initial misori. (θ°) &lt;112&gt; tilt axis</th>
<th>Deformation no</th>
<th>Schmid-factor (m)</th>
<th>Difference of Schmid-factors</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Grain I</td>
<td>Grain II</td>
</tr>
<tr>
<td>11.9 ± 0.4</td>
<td>1</td>
<td>0.449</td>
<td>0.441</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.440</td>
<td>0.410</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.379</td>
<td>0.333</td>
</tr>
<tr>
<td>13.8 ± 0.5</td>
<td>1</td>
<td>0.435</td>
<td>0.443</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.329</td>
<td>0.349</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.390</td>
<td>0.408</td>
</tr>
<tr>
<td>15.9 ± 0.5</td>
<td>1</td>
<td>0.451</td>
<td>0.438</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.460</td>
<td>0.448</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.404</td>
<td>0.324</td>
</tr>
<tr>
<td>30.3 ± 0.5</td>
<td>1</td>
<td>0.472</td>
<td>0.458</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.475</td>
<td>0.444</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.467</td>
<td>0.404</td>
</tr>
</tbody>
</table>

4.1.2.2 Grain boundary with larger misorientation angle (30.3°)

This bicrystal sample consisted of a grain boundary with 30.3° misorientation angle and <112> tilt axis, (Fig. 4.7a) which was also deformed at 2, 4, 6 MPa cyclic stress conditions with constant temperature of 300°C and 20 cycles (Fig. 4.7b-d). From the microstructure (Fig. 4.9) the slip density difference (Δf) was calculated for every stage of deformation. Grain boundary motion was found under all deformation conditions, as shown in Table 4.2. The maximum displacement was found at 4 MPa rather than at 6 MPa, which seems to be a common feature for all samples in this group. The difference of Schmid factors increases with respect to grain boundary displacement (Table 4.4).
4.1.3 Grain boundaries deformed at different number of cycles (N)

This group of samples contains one low angle and a high angle grain boundary. The initial orientations and status of the grain boundary were monitored by optical microscope and scanning electron microscope. Both bicrystal samples were deformed under three distinct number of cycles (e.g. N = 20, 40 and 60) at same temperature (300°C) and constant amplitude of stress (4 MPa) at a constant frequency of loading (1.7 x 10^-2 Hz) with sinus waveform.

Fig. 4.10. Microstructure of a LAGB (13.2°<112> tilt axis) (a) before deformation (b) after deformation with 20 (c) 40 and (d) 60 number of cycle.
4.2 Cyclic motion of $<112>$ tilt boundaries under an elastic stress of deformation

After each stage of cyclic deformation, the samples were examined by optical microscopy as well as EBSD by scanning electron microscope. The microstructures of both the bicrystals have shown (Fig. 4.10 and Fig. 4.11) that the displacement of grain boundary was possible only at higher number of cycle. The trend of grain boundary motion was quite similar to the former experiments.

**4.2 Cyclic motion of $<112>$ tilt boundaries under an elastic stress of deformation**

This section of work mainly emphasises the mechanism and kinetics of cyclic motion of elastically deformed grain boundaries at elevated temperatures. The grain boundaries contain various misorientation angles with $<112>$ rotation axis. Experiments were carried out at different temperatures to find out the activation energies for different misorientation angle in order to examine the misorientaion dependence of grain boundary motion under cyclic deformation. The driving force for cyclic motion was calculated and discussed to understand the mechanism and subsequently provide a model during cyclic motion of geometrically different grain boundaries. The comparison was also made for geometrically different grain boundary motion. Simultaneously, microstructure and microtexture of the grain boundary motions were also described with respect to the deformation conditions. The activation parameters are compared with the previously documented results.
4.2  Cyclic motion of $<112>$ tilt boundaries under an elastic stress of deformation

Fig. 4.12. Arrhenius plot of the GB-displacement for the first cycle during motion of LAGB and HAGB with $<112>$ rotational axis and grain boundary plane was perpendicular to the loading axis.

Fig. 4.13. Arrhenius plot of the GB-displacement for (N-1) no of cycle during motion of LAGB and HAGB with $<112>$ rotational axis and grain boundary plane was perpendicular to the loading axis.

4.2.1 Grain boundaries with perpendicular geometry

This group of samples contained a wide variety of misorientation angle of grain boundaries. The samples were deformed at different temperature (370°C-460°C) with very low amplitude
of stress (± 0.5 MPa) and with 20 numbers of cycles (Table 4.5). In all cases grain boundary plane and line were normal to the loading axis. Grain boundary motion was found in every case despite a sharp limit of temperature of deformation with respect to the mobility of boundaries was observed. As the deformation was purely in elastic regime, no sign of slip band formation and development of misorientation gradient across the grain interior was observed. However, the displacement markings during grain boundary motion were seen very prominent in microstructure (Fig. 4.16).

Table 4.5. Activation energies for planar symmetrical <112> tilt boundaries during cyclic motion. GB plane was perpendicular to the loading axis.

<table>
<thead>
<tr>
<th>Initial θ(°)</th>
<th>Deformation conditions (0.5 MPa, 20 noc) constant</th>
<th>Q_{CGBM} (D_1) [kJ/mole]</th>
<th>Q_{CGBM}(D_{N-1}) [kJ/mole]</th>
<th>Final θ(°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.5 ± 0.2</td>
<td>370°, 385°, 400°, 425°C</td>
<td>61.4 ± 2.8</td>
<td>100.2 ± 11.0</td>
<td>8.2 ± 0.6</td>
</tr>
<tr>
<td>13.7 ± 0.3</td>
<td>370°, 385°, 400°, 425°C</td>
<td>81.2 ± 14.2</td>
<td>113.1 ± 13.0</td>
<td>13.9 ± 0.5</td>
</tr>
<tr>
<td>14.2 ± 0.2</td>
<td>370°, 385°, 400°, 425°C</td>
<td>83.7 ± 8.3</td>
<td>123.7 ± 21.1</td>
<td>14.1 ± 0.5</td>
</tr>
<tr>
<td>15.5 ± 0.5</td>
<td>370°, 385°, 400°, 425°C</td>
<td>97.3 ± 21.7</td>
<td>121.3 ± 21.3</td>
<td>16.1 ± 0.5</td>
</tr>
<tr>
<td>16.5 ± 0.5</td>
<td>370°, 385°, 400°, 425°C</td>
<td>72.2 ± 7.9</td>
<td>79.4 ± 10.6</td>
<td>16.4 ± 0.7</td>
</tr>
<tr>
<td>30.9 ± 0.1</td>
<td>370°, 385°, 400°, 425°C</td>
<td>37.3 ± 2.4</td>
<td>48.7 ± 9.6</td>
<td>29.9 ± 0.5</td>
</tr>
</tbody>
</table>

The grain boundary displacement at different amplitudes of stress were measured at 425°C and 450°C at constant number of cycle as shown in Fig. 4.14 and Fig. 4.15. At 425°C, the diagram shows a linear relationship (Δν ∝ σ) between grain boundary displacement and cyclic stress, at the same time, at 450°C, the relationship is polynomial (Δν ∝ σ^2), which indicates the occurrence of some microplastic deformation at 450°C, although microstructure and microtexture data did not give any indication of plastic deformation. To avoid these conceptual difficulties, the activation parameters were calculated by taking only 4 points in the diagram (up to the temperature 425°C).

Activation parameters for grain boundary motion were calculated from the slope of Arrhenius plot of the grain boundary displacement (m) per cycle (i.e., Δν = m/number of cycle) against reciprocal of the testing temperatures (Fig. 4.12 and Fig. 4.13), assuming the usual relationship

\[ \nu = A \exp\left(-\frac{Q_{gbm}}{RT}\right) \]  

(4.1)

where, ν is the migration displacement per cycle, \( Q_{gbm} \) is the activation energy for grain boundary migration, R is the gas constant, T is the absolute test temperature and A is a pre-exponential factor.
4.2 Cyclic motion of $<112>$ tilt boundaries under an elastic stress of deformation

![Graph](image)

Fig. 4.14. Grain boundary displacement at different amplitude of stress, deformed at 425°C with other parameters constant.

![Graph](image)

Fig. 4.15. Grain boundary displacement at different amplitude of stress, deformed at 450°C with other cyclic parameters constant.
Fig. 4.16. Grain boundary steps during cyclic motion of (a) LAGB of 8.5°<112> tilt boundary, deformed at temperature of 425°C (b) HAGB of 30.5°<112> tilt boundary, deformed at temperature of 450°C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were perpendicular to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).

Fig. 4.17. Non-uniform Grain boundary displacement in every cycles.

As the GB displacement during cyclic deformation was not found uniform, the activation parameters were calculated separately for the first cycle and for the rest of the cycle. Because it has evidently seen that grain boundary displacements were always higher in the first cycle
than rest of the cycles (Fig. 4.17). There is a conspicuous difference in activation energy with respect to low angle and high angle grain boundary motion as shown in Table 4.5. Moreover the misorientation angle dependence of activation energy during cyclic motion exhibits two distinct regions. The activation energy for the motion of low angle grain boundary with misorientation angle 8.5° was calculated as 61.4 ± 2.8 kJ/mole and 100.2 ± 11.0 kJ/mole if the GB displacement in first cycle and in rest of the cyclic is considered separately, whereas, for the high angle grain boundary with misorientation angle 30.9°, the activation energy was calculated, 37.3 ± 2.4 kJ/mole and 48.7 ± 9.6 kJ/mole respectively (Table 4.5).

The microstructure of the grain boundary (those have misorientation angle near to the transition angle, i.e. 13.6° to 16.3°) has shown a noticeable increase of grain boundary displacement with respect to increasing of misorientation angle (Fig. 4.18).

### 4.2.2 Grain boundaries with parallel geometry

This group of samples also contains varieties of grain boundary with different misorientation angles as summarised into Table 4.6, but the grain boundary-plane was parallel to the stress axis during deformation. All the samples were deformed using same parameters of cyclic loading as used into previous experiments (see section 4.2.1). Activation parameters were calculated from the slope of the Arrhenius plot of total grain boundary-displacement per each cycle and the reciprocal of testing temperature (Fig. 4.19).

![Fig. 4.18. The displacement of (a) 13.9° and (b) 16.3° grain boundary with perpendicular geometry after cyclic deformation with an elastic amplitude of stress at 460°C.](image)
4.2 Cyclic motion of $<112>$ tilt boundaries under an elastic stress of deformation

Table 4.6. Activation energies for planar symmetrical $<112>$ tilt boundaries during cyclic motion. GB plane was parallel to the loading axis.

<table>
<thead>
<tr>
<th>Initial $\theta(\degree)$</th>
<th>Deformation conditions (0.5 MPa, 20 noc) constant</th>
<th>$Q_{\text{CGBM}}$ ($D_N$) [kJ/mole]</th>
<th>Final $\theta(\degree)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$7.6 \pm 0.2$</td>
<td>$370\degree, 385\degree, 400\degree, 425\degree$</td>
<td>$78.2 \pm 7.8$</td>
<td>$7.5 \pm 0.5$</td>
</tr>
<tr>
<td>$13.6 \pm 0.3$</td>
<td>$370\degree, 385\degree, 400\degree, 425\degree$</td>
<td>$133.6 \pm 8.3$</td>
<td>$13.8 \pm 0.7$</td>
</tr>
<tr>
<td>$16.6 \pm 0.5$</td>
<td>$370\degree, 385\degree, 400\degree, 425\degree$</td>
<td>$75.5 \pm 5.2$</td>
<td>$17.1 \pm 0.5$</td>
</tr>
<tr>
<td>$30.9 \pm 0.1$</td>
<td>$370\degree, 385\degree, 400\degree, 425\degree$</td>
<td>$37.3 \pm 4.2$</td>
<td>$31.2 \pm 0.6$</td>
</tr>
</tbody>
</table>

For remarks, in this case, the total displacement of grain boundary was considered for the estimation of activation parameters, because, the migration traces were not clearly visible (except initial and final position of the boundary) within the ability of optical microscope. The activation energy for the motion of grain boundary with parallel geometry with the loading axis were measured $78.2 \pm 7.8$ kJ/mole for low angle and $37.3 \pm 4.2$ kJ/mole for high angle grain boundary (Table 4.6). In contrast, the displacement of low angle grain boundaries were always smaller than the high angle grain boundaries, which can be easily separated out from the Fig. 4.20. A strong misorientation angle dependence of activation energy during the motion of parallel grain boundary was also observed with two distinct regions (Table 4.6).

Fig. 4.19. Arrhenius plot of the GB-displacement for (N) number of cycle during motion of LAGB and HAGB with $<112>$ rotation axis and grain boundary plane parallel to the loading axis.
4.2 Cyclic motion of $<11 \overline{2}>$ tilt boundaries under an elastic stress of deformation

Fig. 4.20. Grain boundary steps during cyclic motion of (a) LAGB of $7.6^\circ <11\overline{2}>$ tilt boundary, deformed at temperature of $425^\circ$C (b) HAGB of $30.9^\circ <11\overline{2}>$ tilt boundary, deformed at temperature of $460^\circ$C with 0.5MPa amplitude of stress and 20 cycle. GB-plane were parallel to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).

Fig. 4.21. The grain boundary displacement of (a) $13.6^\circ$ and (b) $16.6^\circ$ at $460^\circ$C under cyclic deformation with an elastic amplitude of stress with parallel geometry.

The grain boundary displacement at higher temperatures ($> 450^\circ$C) was found the largest for all types of boundaries. The motion of the grain boundaries, those have misorientation angle near ($13.6^\circ$ to $16.6^\circ$) to the transition range have shown a clear evidence of increasing displacement rate at same temperature of deformations (Fig. 4.21).
4.2.3 Grain boundaries with 45° inclined geometry

(a) Behaviour of 45° inclined non-CSL boundary

This group of samples includes a range of misorientation angle (7.7° to 30.3°) of grain boundaries. The boundaries were symmetrical with <112> tilt rotational axis. Every sample was deformed at different temperature (370°C to 460°C) with amplitude of stress (±0.5 MPa) and number of cycles (20). The samples were cut in such a way that the grain boundary plane and line would be 45° inclined to the stress axis. The displacement of grain boundary was always in the normal direction of the grain boundaries without any rotation. The motions of this type of grain boundaries were found the largest with respect to the other geometry.

The grain boundary displacement was measured from microstructure of each bicrystals using an optical microscope by taking the distance between initial and final position of the grain boundary, and microtexture was monitored by scanning electron microscope. The activation parameters were calculated from the Arrhenius plot of grain boundary displacement per cycle against reciprocal of absolute temperatures (Fig. 4.22). There is a distinct demarcation between low angle and high angle grain boundaries with respect to its activation energies for motion (Table 4.7).

<table>
<thead>
<tr>
<th>Initial θ(°)</th>
<th>Deformation conditions (0.5 MPa, 20 noc) constant</th>
<th>Q_{CGBM} (D_N) [kJ/mole]</th>
<th>Final θ(°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.7±0.2</td>
<td>370°, 385°, 400°, 425°C</td>
<td>129.1±21.2</td>
<td>7.6±0.4</td>
</tr>
<tr>
<td>14.1±0.3</td>
<td>370°, 385°, 400°, 425°C</td>
<td>161.89 ± 7.7</td>
<td>13.9±0.5</td>
</tr>
<tr>
<td>16.5±0.5</td>
<td>370°, 385°, 400°, 425°C</td>
<td>129.5±3.6</td>
<td>17.1±0.5</td>
</tr>
<tr>
<td>30.3±0.1</td>
<td>370°, 385°, 400°, 425°C</td>
<td>79.3 ± 12.5</td>
<td>31.4±0.3</td>
</tr>
<tr>
<td>41.9±0.2</td>
<td>400°, 425°, 450°, 460°C</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Microstructure of a low angle and a high angle grain boundary are shown in Fig. 4.23, which shows noticeable grain boundary displacement by maintaining parallel line with the initial positions of boundaries, i.e. migration was possible without rotation of the boundaries.

(b) Behaviour of 45° inclined CSL boundary

A few numbers of samples contains only high angle grain boundaries (42.9°) with <112> tilt rotational axis (~Σ21). All these samples were deformed at different temperatures of 400°C to 460°C with taking other parameters same as former experiments. Although all CSL boundaries were inclined to stress axis by 45° like as the non-CSL boundaries,
Fig. 4.22. Arrhenius plot of the GB-displacement for (N) number of cycle during motion of LAGB and HAGB with \(<112>\) rotational axis. Grain boundary plane was inclined at 45° to the stress axis.
Fig. 4.23. Microstructure of the 45° inclined a) low angle grain boundary (7.7° <112> tilt), deformed at 425°C b) high angle grain boundary (30.3° <112> tilt), deformed at 425°C c) GB of 16.5° <112>, deformed at temperatures 450°C d) GB of 14.1° <112> deformed at 450°C with taking other deformation parameters constant (DD: Deformation Direction).

nevertheless, the microstructure shows a 5°-10° rotation of grain boundaries with respect to its initial positions, not only at the edge of the bicrystals (Fig. 4.24b) but also at the centre (Fig. 4.24a), rather than motions of the whole boundary. In some cases, boundary became
segmented during rotation such as to aligned normal to stress axis. (Fig. 4.25). As the displacement of the grain boundaries were non uniform, it is very difficult to set an Arrhenius type of plot and estimate the activation energies. The nature of motion of these boundaries was similar to a non-CSL boundary with respect to formation of grain boundary traces on the surface of the bicrystals.

4.2.4 Grain boundaries with other geometry (15°, 30°, 60° and 75°)

In this case, the samples contain only a low angle and high angle grain boundaries with different geometry with respect to the loading axis. The samples were deformed at two definite temperatures (425°C and 450°C) with elastic amplitude of stress and other parameters taking constant. Grain boundary displacements were measured in each case in order to compare the effect of grain boundary geometry on the grain boundary mobility at different temperatures (Fig. 4.27). The microstructures show that the displacement was always normal to the initial position of grain boundary. In some case; formation of surface trace marks has also been seen (Fig. 4.26). A comparison of grain boundary displacement with different geometry has been presented in Table 4.8 which always maintains an optimum angle of geometry with respect to the applied stress axis, where, grain boundary motion was the largest.

Table 4.8. Comparison of geometrically different grain boundary displacement at higher temperatures.

<table>
<thead>
<tr>
<th>Type of GB</th>
<th>Geometry of GB with respect to stress axis</th>
<th>GB-displacement at 425°C [μm]</th>
<th>GB-displacement at 450°C [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>LAGB</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>15°</td>
<td></td>
<td>62</td>
<td>143</td>
</tr>
<tr>
<td>30°</td>
<td></td>
<td>17</td>
<td>67</td>
</tr>
<tr>
<td>45°</td>
<td></td>
<td>189</td>
<td>221</td>
</tr>
<tr>
<td>60°</td>
<td></td>
<td>9</td>
<td>82</td>
</tr>
<tr>
<td>75°</td>
<td></td>
<td>109</td>
<td>90</td>
</tr>
</tbody>
</table>

For instance, at 45° inclined geometry, the GB-displacement was always largest. In addition, both 30° and 60° inclined grain boundaries were ought to be less mobile as compare to the other geometrically different boundaries (Fig. 4.27).

4.2.5 Comparison of cyclic motion for all geometrically different <112> grain boundaries
A strong dependence of activation energy was estimated with respect to the misorientation angle and inclination angle of the grain boundaries (Fig. 5.5 in Chapter 5). The trend of misorientation dependence of activation energy in every case was similar, always maintain two distinct regions, namely, larger and smaller activation energy region. In both cases there is sharp difference in low angle and high angle grain boundary with concern to their motion. For example, activation energy for cyclic migration were always larger in the range of misorientation angle of 13.6° to 15.5°, and beyond this limit, there is sharp decrease in activation energy, which projects a transition angle between high angle and low angle with respect to their motion under cyclic stress.

### 4.2.6 Microstructure and microtexture evolution for all <112> boundaries

The microstructure of cyclically deformed low angle, high angle and other angle grain boundaries with different geometry (0° - 90° angle to stress axis), were shown in Fig. 4.16 to Fig. 4.26. If we take a close look, grain boundary displacement trace marks on the surface of samples were more prominent in case of perpendicular grain boundary motion than any other type of boundary motion with no evidence of plastic deformation.
Fig. 4.26. Microstructure of bicrystals (with LAGB) during boundary migration of a) 75° b) 60° c) 30° d) 15° angle of inclination with stress axis (DD: Deformation Direction).

The displacement was much larger during first cycle of deformation than rest of the cycles. As a whole, the displacement profile at each cycles were scattered, hence, the velocity of grain boundary motion was non-uniform through out the process (Fig. 4.17). When we estimate the misorientation gradient across the grain interior, no evidence of microtexture development in the vicinity of the grain boundary was observed (Fig. 3.7b).

Fig. 4.27. Comparison of geometrically different boundary displacement at cyclic deformation temperatures 425°C and 450°C
Some experiments were designed in such a manner, that we could confirm the direction of cyclic motion with regards to its deformation mode, for example, only tension-tension cycle, only compression-compression cycle and fully reversed (mixed of tension and compression) as well. The results show that larger grain boundary velocity with increasing deformation temperatures during motion occur in case of compression-compression mode (Fig. 4.28) rather than tension-tension or reversed mode of deformation, which is a good point to be sure that compression part of the fully reversed cyclic loading has major contribution for the motion of grain boundaries, herewith, these results a non uniform motion profile of grain boundary at different temperatures.

4.3 Cyclic motion of <100> tilt boundaries under an elastic stress of deformation

This section of work mainly emphasises the mechanism and kinetics of cyclic motion of elastically deformed <100> tilt grain boundary at elevated temperatures. The grain boundaries contain various misorientation angles with <100> tilt rotation axis. The motion of a planar symmetrical <100>-tilt grain boundaries in high-purity aluminium was investigated, and the results were compared with the grain boundary motion behaviour under constant shear stress field [29]. The driving force for cyclic motion was also calculated as well as discussed to
make a model mechanism during cyclic motion of geometrically different (perpendicular, parallel) <100> grain boundaries and compared with the <112> tilt grain boundary motion.

4.3.1 Motion of <100> tilt grain boundaries with perpendicular geometry

To investigate the motion of <100> tilt boundary with perpendicular geometry, the specimens of high-purity Al bicrystals were cut in such a manner that the grain boundary would be perpendicular to the stress axis. The bicrystals contain grain boundaries with a range of misorientation angle of 3.7° to 42.5° having <100> tilt rotation axis. All the bicrystals were cyclically stressed for 20 cycles at high temperatures from 370°C to 470°C. In general, grain boundary motion was not detected at lower temperature (350°C) but as the temperature is higher than 370°C, there was always some kind of measurable grain boundary displacement (within the resolution range of optical microscope) (Fig. 4.30), which provided a basis to select a critical temperature of about 370°C, below which such a grain boundary does not move. The direction displacements of grain boundary was normal to the boundary plane (but parallel to the deformation direction) which left some impression markings in every cycles on the surface of the samples as could be seen in microstructure (Fig. 4.31). However, the markings in every cycle were not as distinct as that for the motion of <112> tilt boundaries except the displacement markings in first cycle (Fig. 4.32). The displacement of grain boundary was measured from the optical microstructure by taking the distance between initial and final position of boundary after deformation.

Table 4.9. Activation energies for planar symmetrical <100> tilt boundaries during cyclic motion. GB plane was perpendicular to the loading axis.

<table>
<thead>
<tr>
<th>Initial θ(°)</th>
<th>Deformation conditions (0.5 MPa, 20 noc) constant</th>
<th>( Q_{\text{CGBM}} ) (D) [kJ/mole]</th>
<th>Final θ(°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.7 ± 0.2</td>
<td>370°, 400°, 450°C</td>
<td>73.23 ± 4.8</td>
<td>3.4 ± 0.5</td>
</tr>
<tr>
<td>6.8 ± 0.3</td>
<td>370°, 385°, 400°, 425°C</td>
<td>128.15 ± 14.4</td>
<td>7.1 ± 0.3</td>
</tr>
<tr>
<td>7.8 ± 0.2</td>
<td>370°, 385°, 400°, 425°C</td>
<td>146.66 ± 11.4</td>
<td>7.5 ± 0.5</td>
</tr>
<tr>
<td>9.2 ± 0.5</td>
<td>370°, 385°, 400°, 425°C</td>
<td>77.36 ± 17.0</td>
<td>9.1 ± 0.2</td>
</tr>
<tr>
<td>11.7 ± 0.5</td>
<td>370°, 385°, 400°, 425°C</td>
<td>53.78 ± 15.9</td>
<td>12.7 ± 0.3</td>
</tr>
<tr>
<td>17.5 ± 0.5</td>
<td>370°, 385°, 400°, 425°C</td>
<td>78.12 ± 4.8</td>
<td>17.1 ± 0.5</td>
</tr>
<tr>
<td>33.9 ± 0.1</td>
<td>370°, 385°, 400°, 425°C</td>
<td>81.43 ± 11.9</td>
<td>33.3 ± 0.6</td>
</tr>
<tr>
<td>42.5 ± 0.1</td>
<td>370°, 385°, 400°, 425°C</td>
<td>72.62 ± 4.9</td>
<td>42.5 ± 0.5</td>
</tr>
</tbody>
</table>

The grain boundary displacement at different amplitudes of stress was measured at 425°C and 450°C at constant number of cycles for the <112> tilt boundary motion (see section 4.2.1). The results (Fig. 4.14 and Fig 4.15) indicated that some kind of microplastic deformation may
exist at higher temperature (> 425°C) of deformation, although, microstructure does not show any signs of plastic deformation at any temperature. However, to avoid these conceptual difficulties, the activation parameters were calculated by taking only 4 points in the diagram (up to the temperature 425°C). The activation parameters were calculated by assuming an Arrhenius type of equation as

$$v = A \exp\left(-\frac{Q_{gbm}}{RT}\right)$$

(4.1)

where, $v$ is the migration displacement per cycle, $Q_{gbm}$ is the activation energy for grain boundary migration, $R$ is the gas constant, $T$ is the absolute temperature and $A$ is a pre-exponential factor.

Fig. 4.29. Arrhenius plot of the GB-displacement for (N) no of cycle during motion of LAGB and HAGB with <100> tilt axis and grain boundary plane was perpendicular to the stress axis.

The activation energy was determined from the slope of the plot of grain boundary displacement per cycle vs reciprocal of the absolute temperature (Fig. 4.29), although it has been mentioned in section 4.2 that the grain boundary displacement rate is not uniform in every cycle, therefore, the activation energy calculated in this experiment is a near-to-accurate estimation (Table 4.9). The displacement rates of grain boundary motion were determined by considering the total number of cycle, as it is difficult to separate out the displacement in first cycle and in the rest of the cycle from microstructural observation. There is a distinct difference in activation energy with respect to low angle and high angle grain boundary motion as shown in Table 4.9. Moreover, the misorientation angle dependence of activation
energy during cyclic motion of $<100>$ tilt boundaries exhibits two distinct regions (Fig. 5.10 in Chapter 5). The activation energy for the motion of the low angle grain boundary with misorientation angle 3.7° was estimated to be $73.23 \pm 4.8$ kJ/mole. However, for 6.8° and 7.8° grain boundaries, the activation energy were found $128.15 \pm 14.4$ kJ/mole and $146.66 \pm 11.3$ kJ/mole respectively, whereas, for high angle grain boundary with misorientation angle 17.5°, 33.9° and 42.5°, the activation energies were calculated to be $78.12 \pm 4.8$ kJ/mole, $81.43 \pm 11.9$ kJ/mole and $72.62 \pm 4.9$ kJ/mole respectively (Table 4.9).

The microstructural observation of the grain boundaries (with misorientation angle near to the transition angle, i.e. 9.2° to 13.5°) has been shown an impressively increase in grain boundary displacement with respect to the increase in misorientation angle (Fig. 4.31). At the same time, the motion of grain boundaries, those have the misorientation angle (34.5°) near to the CSL boundary, were largest, whereas for 42.5° $<100>$ boundary did not show remarkable motion (Fig. 4.32) evendough, it is a high angle grain boundary.

Fig. 4.30. Grain boundary displacement of (a) LAGB of 3.7°$<100>$ tilt boundary, deformed at temperature of 450°C (b) HAGB of 30.1°$<100>$ tilt boundary, deformed at 450°C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were perpendicular to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).
4.3 Cyclic motion of $\langle 100 \rangle$ tilt boundaries under an elastic stress of deformation

Fig. 4.31. Grain boundary displacement of (a) $9.2^\circ\langle 100 \rangle$ tilt boundary, deformed at 450°C (b) $13.5^\circ\langle 100 \rangle$ tilt boundary, deformed at 450°C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were perpendicular to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).

Fig. 4.32. Grain boundary displacement of (a) $33.9^\circ\langle 100 \rangle$ tilt boundary, deformed at 450°C (b) $42.5^\circ\langle 100 \rangle$ tilt boundary, deformed at 450°C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were perpendicular to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).
4.3.2 Motion of $<100>$ tilt grain boundaries with parallel geometry

The bicrystal samples of this group of experiments were fabricated in such way that the grain boundary would be always parallel to the stress axis. The grain boundary length was comparatively larger than that for the perpendicular geometry. All the bicrystals contained grain boundary with misorientation angle $3.7^\circ$ to $42.5^\circ$, as similar to the previous section. The samples were deformed using same parameters of cyclic loading as used into the previous experiments (see section 4.3.1). Only two samples ($6.6^\circ$ and $34.5^\circ$) were deformed at different temperatures to estimate the activation parameters. In general, the direction of grain boundary displacement was normal to the boundary plane and deformation direction (Fig. 4.34). The displacements of the grain boundary were measured by taking the distance between initial and final position of the boundary after deformation.

Table 4.10. Activation energies for planar symmetrical $<100>$ tilt boundaries during cyclic motion. GB plane was parallel to the loading axis.

<table>
<thead>
<tr>
<th>Initial $\theta$ (°)</th>
<th>Deformation conditions (0.5 MPa, 20 noc) constant</th>
<th>$Q_{CGBM}$ (D) [kJ/mole]</th>
<th>Final $\theta$ (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.7 ± 0.2</td>
<td>400°C</td>
<td>129.48 ± 7.4</td>
<td>3.4 ± 0.5</td>
</tr>
<tr>
<td>6.6 ± 0.2</td>
<td>385°, 400°, 425°, 450°C</td>
<td>9.1 ± 0.2</td>
<td>6.8 ± 0.3</td>
</tr>
<tr>
<td>9.2 ± 0.5</td>
<td>400°C</td>
<td>17.1 ± 0.5</td>
<td>9.1 ± 0.2</td>
</tr>
<tr>
<td>17.5 ± 0.5</td>
<td>400°C</td>
<td>72.96 ± 11.3</td>
<td>33.3 ± 0.6</td>
</tr>
<tr>
<td>34.5 ± 0.1</td>
<td>385°, 400°, 425°, 450°C</td>
<td>42.5 ± 0.5</td>
<td>42.5 ± 0.5</td>
</tr>
<tr>
<td>42.5 ± 0.1</td>
<td>400°, 450°C</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

![Graph showing activation energies and reciprocal temperatures for different tilt angles.](image-url)
4.3  Cyclic motion of $<100>$ tilt boundaries under an elastic stress of deformation

Fig. 4.33. Arrhenius plot of the GB-displacement for (N) no of cycle during motion of LAGB and HAGB with $<100>$ tilt rotational axis. Grain boundary plane was parallel to the stress axis.

Fig. 4.34. Grain boundary displacement of (a) 3.7° $<100>$ tilt boundary, deformed at 400°C (b) 17.5° $<100>$ tilt boundary, deformed at 400°C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were parallel to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).

Fig. 4.35. Grain boundary displacement of 9.2° $<100>$ tilt boundary, deformed at 400°C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were parallel to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).
4.3 Cyclic motion of $<100>$ tilt boundaries under an elastic stress of deformation

Activation parameters were calculated from the slope of the Arrhenius plot of total grain boundary-displacement per each cycle and the reciprocal of testing temperature (Fig. 4.33). For remarks, in this case, the total displacement was composed for the estimation of activation parameters, because, the migration traces were not clearly visible (except initial and final position of the boundary) under the ability of optical microscope. The activation energy for the motion of grain boundary with parallel geometry with the loading axis were measured to be $129.48 \pm 7.4$ kJ/mole for the low angle ($6.6^\circ <100>$ tilt) and $72.96 \pm 11.3$ kJ/mole for the high angle ($34.5^\circ <100>$ tilt) grain boundary (Table 4.10).

The displacements of low angle grain boundaries were always smaller than the high angle grain boundaries, which can be easily separate out from the figures 4.34 and 4.36. The misorientation angle dependence of activation energy during grain boundary motion with parallel geometry was found to decrease with an increase in misorientation angle (Fig. 5.10). The grain boundary displacement at higher temperatures (> 450°C) was always the largest for all type of boundaries. The motion of grain boundaries were also found an increasing trend after certain misorientation angle (>9.2°) deformed at same temperature (Fig. 4.35).

Fig. 4.36. Grain boundary displacement of (a) 34.5°<$100>$ tilt boundary, deformed at 425°C (b) 42.5°<$100>$ tilt boundary, deformed at 450°C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were parallel to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).
4.4 Cyclic motion of <100> twist boundaries under an elastic stress of deformation

The mechanism and kinetics of cyclic motion of elastically deformed <100> twist grain boundaries at elevated temperatures were also studied. The grain boundaries contain various misorientation angles (5.9° to 11.2°) with <100> twist rotation axis. The cyclic motion of a planar symmetrical <100>-twist grain boundaries in high-purity aluminium was investigated, and the results were compared with the grain boundary motion behaviour of the tilt grain boundaries. The driving force for cyclic motion were also calculated as well as discussed to formulate a model mechanism during cyclic motion of geometrically different (perpendicular, parallel) <100> twist grain boundaries and compared with the tilt grain boundary motion.

4.4.1 Motion of <100> twist grain boundaries with perpendicular geometry

The shapes of the samples were chosen in such a way that the grain boundary plane would be always perpendicular to stress axis during deformation of the bicrystals. The range of misorientation angle of grain boundaries were 5.9° to 11.2° with <100> twist rotation axis. All the bicrystal samples of this geometry were deformed under a cyclic deformation conditions (similar as the previous experiments) within the range of temperature, 385°C to 460°C at a constant amplitude of stress (±0.5 MPa) and number of cycle (20) (Table 4.11).

Table 4.11. Activation energies for planar symmetrical <100> twist boundaries during cyclic motion. GB plane was perpendicular to the loading axis.

<table>
<thead>
<tr>
<th>Initial θ(°)</th>
<th>Deformation conditions (0.5 MPa, 20 noc) constant</th>
<th>Q_{CGBM} (D) [kJ/mole]</th>
<th>Final θ(°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.9° ± 0.2</td>
<td>385°, 400°, 425°, 450°C</td>
<td>74.34 ± 0.4</td>
<td>5.4° ± 0.4</td>
</tr>
<tr>
<td>7.9° ± 0.5</td>
<td>385°, 400°, 425°, 450°C</td>
<td>61.55 ± 7.2</td>
<td>8.2° ± 0.2</td>
</tr>
<tr>
<td>9.2° ± 0.5</td>
<td>385°, 400°, 425°, 450°C</td>
<td>69.4 ± 10.3</td>
<td>9.2° ± 0.4</td>
</tr>
<tr>
<td>10.5° ± 0.1</td>
<td>385°, 400°, 425°, 450°C</td>
<td>60.8 ± 16.4</td>
<td>10.9° ± 0.4</td>
</tr>
<tr>
<td>11.2° ± 0.1</td>
<td>385°, 400°, 425°, 450°C</td>
<td>110.8 ± 10.3</td>
<td>11.2° ± 0.6</td>
</tr>
</tbody>
</table>

In general, the grain boundary although made some kind of trace markings during motion upon deformation, the motion was very much scattered and non-uniform as compared to the tilt boundaries (Fig. 4.37), therefore, it is very difficult to decide the direction of grain boundary motion. However, the displacements of the grain boundaries were measured from the optical microstructure by taking the distance between initial and final position of the grain boundary (Fig. 4.38).
4.4 Cyclic motion of $<100>$ twist boundaries under an elastic stress of deformation

Fig. 4.37. Grain boundary displacement of (a) $5.9^\circ<100>$ twist boundary, deformed at $400^\circ$C (b) $7.9^\circ<100>$ twist boundary, deformed at $460^\circ$C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were perpendicular to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).

Fig. 4.38. Grain boundary displacement of (a) $9.2^\circ<100>$ twist boundary, deformed at $450^\circ$C (b) $11.2^\circ<100>$ twist boundary, deformed at $425^\circ$C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were perpendicular to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).
4.4 Cyclic motion of $<100>$ twist boundaries under an elastic stress of deformation

Fig. 4.39. Microstructure of cyclically deformed bicrystals to show typical cyclic motion of twist grain boundaries (10.5° $<100>$ twist boundary with perpendicular geometry) (a) locally rotation of grain boundary during deformation (b) locally bending of same grain boundary (DD: Direction of Deformation, I: Initial position; F: Final position of GB).

From the displacement data, the activation parameters were calculated from the slope of the Arrhenius plot of total grain boundary-displacement per each cycle and the reciprocal of testing temperature (Fig. 4.41). In this case, the total displacement was considered for the estimation of grain boundary velocity, because, the boundary traces due to motion in every cycle were not clearly visible (except initial and final position) under the ability of optical microscope.

Fig. 4.40. Grain boundary bulging during cyclic deformation of twist boundaries (10.5° $<100>$ twist) (DD: Direction of Deformation, I: Initial position; F: Final position of GB).
The activation energy for the motion of grain boundary with perpendicular geometry with the loading axis were measured to be 74.34 ± 0.4 kJ/mole for low angle boundary (5.9° <100> twist) and 110.8 ± 10.3 kJ/mole for high angle (11.2° <100> twist) grain boundary (Table 4.11), which is distinctly different from the activation parameters of tilt boundaries during motion.

Fig. 4.41. Arrhenius plot of the GB-displacement for (N) no of cycle during motion of 5.9° and 11.2° with <100> twist rotational axis and grain boundary plane was perpendicular to the stress axis.

In addition, the common trend of motion of twist boundary has shown an increase in displacement with increasing temperature, although it is very common nature for all boundaries, irrespective of misorientation angle and rotation axis. As it is already known that twist grain boundaries are less mobile than tilt boundary, but the present results show larger mobility, although, the displacement of boundary was very much localised, in turn, grain boundary rotation (Fig. 4.39) in different direction as well as grain boundary bulging (Fig. 4.40) was observed in microstructures after deformation. This type of motion of grain boundary was only found in case of twist boundary.

4.4.2 Motion of <100> twist grain boundaries with parallel geometry
The grain boundary of parallel geometry was similarly fabricated by taking the boundary plane parallel to the stress axis. This group of experiment contains only two type of grain boundary with regard to the misorientation angle (e.g. 9.3° and 10.9°) with <100> twist rotation axis. The bicrystals were deformed at different temperatures (385°C to 460°C) under constant amplitude of stress (0.5 MPa) and number of cycles (20). The step marks of grain boundary during motion was also found as in the previous experiments, but the markings in each cycle was not so clear in the range of optical microscope (Fig. 4.43), hence, the displacement of grain boundary was measured from the distance between initial and final position of boundary after deformation. The direction of displacement of the boundary was normal to the direction of deformation as seen into the last experiments. The grain boundary rotation seems to be a common feature during the motion of twist grain boundaries, irrespective of the inclination angle of grain boundary with stress axis, because, grain boundary rotation was also observed in case of the motion of parallel grain boundary (Fig. 4.44).

The velocity of grain boundary was calculated by taking the total displacement divided by total number of cycle, and the activation parameters were calculated from the slope of the Arrhenius plot of total grain boundary-displacement per each cycle and the reciprocal of testing temperature (Fig. 4.42). The activation energy for the motion of grain boundary with perpendicular geometry with the loading axis were measured 78.43 ± 19.7 kJ/mole for low angle (9.3° <100> twist) and 93.8 ± 12.7 kJ/mole for high angle (11.2° <100> twist) grain boundary (Table 4.12), which is different from the activation parameters of tilt boundaries during motion.

Table 4.12. Activation energies for planar symmetrical <100> twist boundaries during cyclic motion. GB plane was parallel to the loading axis.

<table>
<thead>
<tr>
<th>Initial θ(°)</th>
<th>Deformation conditions (0.5 MPa, 20 noc) constant</th>
<th>Q_{CGBM} (D) [kJ/mole]</th>
<th>Final θ(°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.3° ± 0.5</td>
<td>385°, 400°, 425°, 450°C</td>
<td>78.4 ± 19.7</td>
<td>9.2° ± 0.4</td>
</tr>
<tr>
<td>10.9° ± 0.1</td>
<td>385°, 400°, 425°, 450°C</td>
<td>93.8 ± 12.7</td>
<td>10.3° ± 0.4</td>
</tr>
</tbody>
</table>
4.4 Cyclic motion of $<100>$ twist boundaries under an elastic stress of deformation

Fig. 4.42. Arrhenius plot of the GB-displacement for (N) no of cycle during motion of 9.3° and 10.9° with $<100>$ twist rotational axis and grain boundary plane was parallel to the stress axis.

Fig. 4.43. Grain boundary displacement of (a) 10.9°$<100>$ twist boundary, deformed at 425°C (b) 9.3°$<100>$ twist boundary, deformed at 425°C with 0.5MPa amplitude of stress and 20 cycle and GB-planes were parallel to the loading axis. (DD: Direction of Deformation, I: Initial position; F: Final position of GB).
Fig. 4.44. Microstructure of cyclically deformed bicrystals showing local rotation of twist grain boundaries (10.9° <100> twist boundary with parallel geometry).
Chapter 5

Discussion

5.1 Cyclic motion of <112> tilt boundary under plastic deformation

The displacement of the grain boundary of this group was found under plastic amplitude of stress deformation. The results have shown that there is a strong effect of the cyclic deformation parameters on grain boundary motion. The prime motivation of this part of the work is to index the driving force of this type of grain boundary motion. It was evidently found that the dislocation density was not equal in both grains and that the difference of dislocation density across the grain boundary is the basic driving force for cyclic motion. Different aspects of the grain boundary motion are discussed in the following subsections.

5.1.1 Indexing of driving force during cyclic grain boundary motion

The observed movement of grain boundaries in bicrystals was accompanied by substantially visible (up to the range of optical microscope) slip bands on microstructure. To determine an approximate value of dislocation density difference between the grains during deformation, the visible slip lines were counted from microstructures for individual grains to calculate the slip density difference, which was defined as $\Delta f_i$. The slip density difference ($\Delta f_i$) basically can be related to the dislocation density difference ($\Delta \rho$) between the two grains, which was considered as a major source of driving force for grain boundary motion [2]. There was a wide fluctuation in $\Delta f_i$ values in every case of cyclic deformation. In the case of low angle grain boundaries, where grain boundary motion was not detected within the resolution range of optical microscope, $\Delta f_i$ values were very less irrespective of deformation conditions. A critical range of $\Delta f_i$ vs misorientation angle was established (0.003), where, above this range (> 0.003) all boundaries were mobile and high angle boundaries, and below this range (< 0.003) most of boundaries were low angle and immobile (Fig. 5.2). Nevertheless, general trend of grain boundary displacement is showing that $\Delta f_i$ has a strong effect on grain boundary mobility, in other words, boundary displacement increases with increasing $\Delta f_i$ values.
Therefore, based on these observations it is proposed that during cyclic deformation the two grains of the bicrystal deforms inhomogeneously, which results in locally large differences in slip band formation as seen in the microstructures, i.e. there were locally large differences in dislocation density which exert a driving force on the grain boundary and force the grain boundary to move into the grain of higher dislocation density, similar argument were addressed by Weiss et al [65]. At higher degree of deformation (e.g higher deformation temperatures and stress amplitude), the displacement of the grain boundary was restricted due to homogeneous deformation structures within the grains, which implies a smaller difference of slip density and Schmid factors. Therefore, it can be concluded that the (Δf) value is a considerable index to emphasise the source of driving force during cyclic grain boundary motion.

The Schmid factors were calculated from local orientations to determine the active slip systems of the individual grains during cyclic deformation. The differences of Schmid factors within the grains and between the grains were very small (< 0.015) for immobile grain boundaries but larger differences (> 0.015) were found for mobile boundaries (Table 4.3 and Table 4.4). Similar values were reported in previous works [65]. This difference was developed due to deviations from the ideal symmetries of the two grains in the initial state and to fluctuations of local orientations into the grains during deformation.

As already shown in [65], a difference in the Schmid factors in the two grains causes a driving force for grain boundary motion. Obviously, there exists a critical value of the difference in Schmid factors, below this critical value (< 0.015) grain boundary motion can not be observed within the resolution of the optical microscope as well as of the scanning electron microscope. Above this value, grain boundaries can move under cyclic deformation conditions.

5.1.2 Misorientation angle distribution during cyclic motion

Experimental results have proven that for misorientation angles smaller than 13.8°, no cyclic motion occurred, whereas for angles larger than 13.8°, the grain boundaries were found to be mobile during cyclic deformation (see Table 4.1 and Table 4.2). So it is obvious that the observed distinction between mobile and immobile grain boundaries must be attributed to the structural transition between low angle and high angle grain boundaries. The corresponding transition occurs at the angle of 13.8° (Fig. 5.1). A similar argument, despite, the deformation conditions were different in both cases, was also noticed in the shear stress experiments [30]. It could be proposed that the initial angle of misorientation is not a major factor for cyclic boundary motion, in other words, if the misorientation angle moves up to the transition angle
between LAGBs and HAGBs during deformation, then the motion could found in any circumstances of cyclic deformation. For example, in case of 11.9°<112> grain boundary, the displacement was measurable despite it has much less initial misorientation angle than the transition angle. After the deformation the misorientation angle has changed to 15.2°, which means, it goes above the transition angle.

Fig. 5.1. Misorientation angle distribution of low and high angle grain boundaries with respect to their mobile and immobile nature (▲ for mobile HAGB, Δ for mobile LAGB and ▼ for immobile LAGB and ▼ for immobile HAGB)
5.1 Cyclic motion of $<112>$ tilt boundaries under plastic deformation

Fig. 5.2. The plot of misorientation angle of grain boundary vs slip density difference ($\Delta f$) during cyclic deformation ( ▲ - mobile and ▼ - immobile boundaries).

Fig. 5.3. Diagram showing the difference of Schmid factor vs misorientation angle of grain boundaries during motion under cyclic deformation ( ▼ - mobile and ▲ - immobile boundaries).

Fig. 5.4. Combined relationship of the difference of Schmid factors and slip density during cyclic motion of different grain boundaries ( ▲ - mobile and ▼ - immobile boundaries).
5.1.3 Role of cyclic stress ($\sigma_a$) on boundary motion

The results show that the largest grain boundary displacement was noticed in the 4 MPa cyclic stress conditions for all type of grain boundaries. Subsequently, cyclic motion was either absent or was very less, when the boundaries were deformed at 6 MPa, inspite, the differences of slip densities were comparable to the values under 4 MPa stress of deformation. In the case of lower stress, it was evidently found that the grains comprising the bicrystal did not deform uniformly, and regimes of differently high dislocation density developed into both sides of the grain boundary, which results in a large driving force for the motion of grain boundaries. Correspondingly, at large stresses, high dislocation density developed due to activation of locally different slip systems in the grains, but the dislocation distribution became more homogeneous compared to the dislocation distribution in the bicrystal which are deformed at lower stresses. Because at larger stress of deformation, the probability for the activation of secondary slip system are more feasible. This results in a lower difference in dislocation density across the grain boundary and therefore, a small driving force to move the boundary. Based on these experimental results it can be interpreted that small stresses of deformation can be favourable for larger displacement of the grain boundaries.

5.1.4 Combined effect of indexing parameters of driving force

It has been mentioned in the previous subsections that the driving force for grain boundary motion can be attributed to difference of dislocation densities between the two grains during deformation, and these driving force can be indexed by several parameters (section 4.1). A critical range of slip density difference ($\Delta f_i$) vs misorientation angle was established (0.003) during motion of grain boundaries, where, above this range (>0.003) all boundaries were mobile and high angle boundaries, and below this range (<0.003) most of boundaries were low angle and immobile. However, there was also some indication that the $\Delta f_i$ value of some of the low angle boundaries has been showing larger value than the critical range (0.003) and mobile and at the same time, some of the high angle boundaries showing lower value than the critical range and immobile (Fig. 5.2). Simultaneously, the differences of Schmid factors within the grains and between the grains are very small (less than 0.015) for immobile grain boundaries but the larger differences (> 0.015) are found for mobile boundaries with some exceptional cases (Fig. 5.3). Therefore, this is an open question why some of the grain boundaries have been shown this type of exceptional behaviour. The most justified reason would be that, irrespective of
misorientation angle and condition of deformations, motion of grain boundaries under a plastic cyclic deformation is only possible by the combined effect of these parameters (Fig. 5.4). Therefore, an optimised range of indexing parameters of driving force during cyclic motion can be established from the diagram of $\Delta f_i$ (slip density difference) vs $\Delta m$ (difference of Schmid factors) with considering the mobile and immobile nature of the boundaries, for instance, the optimised condition for mobile boundaries are, $\Delta f_i > 0.003$ and $\Delta m > 0.015$ during deformation (Fig. 5.4).

5.1.5 Summary of the section

1. The motion of planar, symmetrical $<112>$ tilt high angle grain boundaries in aluminium was possible under the influence of high temperature cyclic stress field (plastic range) with different combinations of deformation parameters.

2. For low angle grain boundaries, the movement of grain boundaries were not possible as long as the misorientation angle of the boundary was below a critical range of 13.8°. Grain boundaries with misorientation angle above 13.8° were found to be mobile under cyclic deformation conditions.

3. The deformed structure of bicrystal shows a heterogeneous deformation into the grains and generates a difference in dislocation density between the grains, which was the driving force for motion.

4. The differences of slip density ($\Delta f_i$) and Schmid factors between the two grains of the deformed structures of bicrystal were observed most accountable for indexing of driving force of cyclic motion of grain boundaries.

5. The effect of cyclic stress was relatively more clear and comparable. Larger stress reduces the chance for grain boundary motion due to homogeneous deformation structure in the two grains.

5.2 Cyclic motion of $<112>$ tilt boundary under elastic stress

5.2.1 Mechanism for the motion of geometrically different grain boundaries with regard to kinetics

It has already been established that $<112>$ symmetrical planar grain boundaries can be driven by a mechanical stress field irrespective of the angle of misorientation [30, 31], similar kind
of argument can also be propose in the present study, although the type of stress was different from the previous one. The motion in the first cycle (in perpendicular geometry) is distinctly different with respect to its activation energy. But as a whole, in case of perpendicular and parallel geometry, for low angle grain boundary motion, activation energies were found to be larger than the grain boundary self diffusion enthalpies (0.6-0.9 eV),

Fig. 5.5. Misorientation angle dependence of activation energy of all <112> tilt grain boundaries of perpendicular geometry during cyclic motion.

Table 5.1 Comparison of activation energies with the activation enthalpies of diffusion during motion of planar symmetrical <112> tilt boundaries. GB plane was perpendicular to the stress axis.

<table>
<thead>
<tr>
<th>Initial θ(°)</th>
<th>Q_{CGBM} (D₁) [kJ/mole]</th>
<th>A.E. of diffusion (GB diffusion 0.6 – 0.9 eV Vol. diffusion 1.25 eV)</th>
<th>Q_{CGBM}(D_{N-1}) [kJ/mole]</th>
<th>A.E. of diffusion (GB diffusion 0.6 – Δ0.9 eV Vol. diffusion 1.25 eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.5 ± 0.2</td>
<td>61.4 ± 2.8</td>
<td>GB diffusion</td>
<td>100.2 ± 11.0</td>
<td>Vol. diffusion</td>
</tr>
<tr>
<td>13.7 ± 0.3</td>
<td>81.2 ± 14.2</td>
<td>GB diffusion</td>
<td>113.1 ± 13.0</td>
<td>Vol. diffusion</td>
</tr>
<tr>
<td>14.2 ± 0.2</td>
<td>83.7 ± 8.3</td>
<td>GB diffusion</td>
<td>123.7 ± 21.1</td>
<td>Vol. diffusion</td>
</tr>
<tr>
<td>15.5 ± 0.5</td>
<td>97.3 ± 21.7</td>
<td>GB diffusion</td>
<td>121.3 ± 21.3</td>
<td>Vol. diffusion</td>
</tr>
<tr>
<td>16.5 ± 0.5</td>
<td>72.2 ± 7.9</td>
<td>GB diffusion</td>
<td>79.4 ± 10.6</td>
<td>GB diffusion</td>
</tr>
<tr>
<td>30.9 ± 0.1</td>
<td>37.3 ± 2.4</td>
<td>Low value</td>
<td>48.7 ± 9.6</td>
<td>GB diffusion</td>
</tr>
</tbody>
</table>
whereas, for high angle boundary migration, activation energies were found to be smaller than the grain boundary diffusion enthalpies. But in case of 45° inclined geometry, for low angle grain boundary motion, activation energies (129.1 ± 21.159 kJ/mole) were close to volume diffusion enthalpies (1.25 eV), and for high angle grain boundary motion (79.3 ± 12.541 kJ/mole), it was close to grain boundary diffusion enthalpy (0.6-0.9 eV).

Fig. 5.6. Misorientation angle dependence of activation energy of <112> tilt grain boundaries of parallel and 45° inclined geometry during cyclic motion.

Although the observed activation enthalpies for different grain boundaries motion shows scattered results with compared to the pure shear stress driven experiments, but nevertheless, majority of these results have shown similar kind of activation enthalpies. Therefore, the mechanism for the motion of the grain boundaries was also similar. The activation energies of grain boundary motion does change within the misorientation angles 13.7° to 15.5° (Fig. 5.5 and Fig. 5.6), which reflects a transition regime between low angle grain boundary to high angle grain boundary. In general, the measured activation energies for the low angle regime of boundaries (< 13.7° to 15.5°) lie in the range of 100.2 ± 11.2 kJ/mole to 161 ± 7.7 kJ/mole, which is basically identical to the activation energies of volume diffusion. Similarly, for high angle grain boundary regime (> 15.5°) the activation energies for motion was measured in the range of 48.7 ± 9.6 kJ/mole to 79.3 ± 12.5 kJ/mole, which is essentially equivalent to the
activation energies of grain boundary self diffusion. These are independent for the geometrically different grain boundaries. These results have indicated that the grain boundary motion is assisted by dislocation climb. For instance, when the structural dislocations are isolated, as in low angle grain boundaries, volume diffusion is needed to provide the vacancies for dislocation climb, whereas, if the grain boundary forms a slab of dislocation like as high angle grain boundaries, vacancies can reach the structural dislocations via grain boundary diffusion. This argument has already been confirmed during <112> tilt grain boundary motion under pure shear stress conditions [30, 31], indeed, in the present case grain boundary geometry was different. However, it could be commented that motion mechanisms in cyclic loading are not exactly similar as compared to the pure shear stress driven boundary motion experiments. A comparison of activation energies for cyclic motion of grain boundary with the diffusion enthalpies has been presented in tables 5.1 and 5.2. It has been already reported that grain boundary motion under cyclic loading are a combination of sliding and migration [46-47]. Correspondingly, the grain boundary sliding and migration was explained on the basis of dislocation mechanism for sliding which is based on the movement of lattice dislocations along the grain boundary by a combination of dislocation climb and glide, however, it was reported in the case of cubic Zirconia bicrystals [69]. In general, symmetrical <112> tilt grain boundaries comprises of a group of edge dislocations with <110> Burgers vector, which are perfect lattice dislocations and glissile for a resolved shear stress perpendicular to the boundary plane, whose reaction to the applied stress ought to be purely mechanical and would not be thermally activated, however the presently observed grain boundary motion was undoubtedly thermally activated. One has to also realise that fabrication of perfect <112> symmetrical tilt boundary is basically impossible in reality and small deviations from <112> tilt axis are accepted. These dislocations, which makes deviations of ideal position of <112> tilt axis, have to be move by climb process as these are not generally glissile. Similarly, the bulk dislocation debris has to be swept up during motion of grain boundary.

However, in the present study, a reasonable amount of measured activation energies for grain boundary motion has been shown scattered with compared to the formerly reported results [29-32]. The most difficult point on this regard was that the cyclic stress was never constant which promotes a non uniform velocity of grain boundary motion and the applied stress was normal to the grain boundary plane, which essentially means a negligible force acting on the structural dislocations to move for motion of entire boundary. Secondly, the activation parameters were calculated by considering only four different temperatures because of the
restriction of plastic deformation at higher temperatures (see section 4.2). However, we have already estimated the acting stress components on dislocations to move as well the driving force for the motion of grain boundaries (section 4.2) due to development of shear stress field on both of the grains. Meanwhile, the real grain boundary plane won’t be perfectly oriented either normal or parallel to the deformation direction, which always contains some tiny angle to its loading axis, and these angles help to develop a shear stress on the structural dislocations on the grain boundary.

Table 5.2 Comparison of activation energies with the activation enthalpies of diffusion during motion of planar symmetrical <112> tilt boundaries. GB plane was parallel and 45° inclined to the stress axis.

<table>
<thead>
<tr>
<th>Initial θ(°)</th>
<th>Q_{CGBM} (D_N) [kJ/mole]</th>
<th>A.E. of diffusion (GB diffusion 0.6 – 0.9 eV Vol. diffusion 1.25 eV)</th>
<th>GB Geometry</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.6 ± 0.2</td>
<td>78.2 ± 7.8</td>
<td>GB diffusion</td>
<td>Parallel Geometric</td>
</tr>
<tr>
<td>13.6 ± 0.3</td>
<td>133.6 ± 8.3</td>
<td>Vol. diffusion</td>
<td></td>
</tr>
<tr>
<td>16.6 ± 0.5</td>
<td>75.5 ± 5.2</td>
<td>GB diffusion</td>
<td></td>
</tr>
<tr>
<td>30.9 ± 0.1</td>
<td>37.3 ± 4.2</td>
<td>Low value</td>
<td></td>
</tr>
<tr>
<td>7.7±0.2</td>
<td>129.1±21.2</td>
<td>Vol. diffusion</td>
<td>45° Inclined Geometry</td>
</tr>
<tr>
<td>14.1±0.3</td>
<td>161.9±7.7</td>
<td>Vol. diffusion</td>
<td></td>
</tr>
<tr>
<td>16.5±0.5</td>
<td>129.5±3.6</td>
<td>Vol. diffusion</td>
<td></td>
</tr>
<tr>
<td>30.3±0.1</td>
<td>79.3±12.5</td>
<td>GB diffusion</td>
<td></td>
</tr>
</tbody>
</table>

5.2.2 Mechanism for the motion of perpendicular boundaries with regard to dislocation dynamics

As we have already assumed that the motion under cyclic stress were essentially driven by shear stress acting on the boundary (Fig. 5.7). The detail mathematical calculation for the stress components acting on dislocations and the possible driving force has been discussed in section 3.3.2. The forces acting on the dislocation of grain boundaries are summarised in equations (3.9). As the motion of grain boundary for perpendicular geometry
Fig. 5.7. Schematic diagrams for the forces acting on the dislocations of grain boundaries in perpendicular to the stress axis during cyclic deformation.

was found along the x-direction thus, the force normal to the boundary \((F_x^{\text{dis}})_1\) and \((F_x^{\text{dis}})_2\), causes the whole boundary to move. As a consequence, \(\sigma_{xx}\) cannot be neglected \((\sigma_{xx} \neq 0)\) as the normal stress were applied with a reasonable amount, therefore, \((F_x^{\text{dis}})_1\) and \((F_x^{\text{dis}})_2\) will also be active parallel to the grain boundary, which may cause the boundary to slide along the y-direction. However, if we take a close look to the equation (3.9), the direction and magnitude of force components \((F_y^{\text{dis}})_1\) and \((F_y^{\text{dis}})_2\) of F1 and F2 dislocations are only compatible for sliding of boundary along the y-direction, until and unless, the force components \((F_y^{\text{dis}})_1\) and \((F_y^{\text{dis}})_2\) have the same sign, i.e, as \((F_y^{\text{dis}})_1\) is positive \((\text{e.g.} > 0)\), thus, \((F_y^{\text{dis}})_2 > 0\). Therefore, sliding will be possible only when,

\[
\sigma_{xx} b \cos \theta / 2 \geq \tau_{xy} b \sin \theta / 2
\]

with a consideration \(\tau_{xy} \equiv \tau\) and \(\sigma_{xx} \equiv \sigma\), equation (5.5) can be extended as

\[
\sigma \cos \theta / 2 \geq \tau \sin \theta / 2 \quad \text{or} \quad \frac{\sigma}{\tau} \geq \tan \theta / 2
\]

Thus, a preliminary condition for the sliding of grain boundary along y-direction (Fig. 5.7) can be established from the equation (5.6). In general, the ratio of normal stress and shear stress \((\sigma/\tau)\) for low angle grain boundary is lower than the ratio \((\sigma/\tau)\) for the high angle boundaries. In the present case, the normal stress was always constant but higher than the generated shear stress. Based on these evidences it can be proposed that sliding of boundary during the motion of perpendicular high angle grain boundaries are more prominent than low angle boundary. Therefore, the cyclic motion (in case of perpendicular geometry) was essentially the composition of sliding along the y-direction (parallel to the boundary) and migration along the x-direction (normal to the boundary). Probably, this is the reason that the boundary trace-marks were formed during cyclic motion of grain boundary.

Similar kind of stress distributions on dislocation were also estimated in the shear stress driven grain boundary motion [30], but at the same time, the nature of the motion was different in each case. Generally during cyclic motion, boundaries left trace-marks on the surface of the sample while, for shear stress experiments it was not the case. The main reason is that during the former experiments, the normal stress \((\sigma)\) was assumed to be zero, i.e the force parallel to the grain boundary is very small. Therefore, there was no possibility for the grain boundary sliding along the parallel directions. On the other hand, force normal to the
boundary \( (F^\text{dis}_{y}) \) causes the dislocation to move as well the entire grain boundary to move without any marking on surface except initial and final position of the boundary.

### 5.2.3 Mechanism for the motion of parallel boundaries with regard to dislocation dynamics

The activation energies for the motion of parallel boundaries were also calculated to characterise the behaviour of low angle and high angle grain boundary motion under cyclic loading conditions as presented in Table 4.6. Similar kind of motion was seen in these experiments, eventhough, the grain boundary displacement in first cycle and in rest of the cycles were not separately calculated, because in this case, the displacement-markings were not so clearly visible under the resolution range of optical microscope. The nature of parallel boundary motion were very much similar to the pure shear stress driven boundary motion, i.e. only the initial and final position of the grain boundary were seen into the microstructures. This argument would be more understandable if we look into the stress distribution and driving force components (see section 3.3.2) on the grain boundary during deformation. From the equation (3.16), the force normal to boundary \( (F^\text{dis}_{y})_1 \) and \( (F^\text{dis}_{y})_2 \) are not similar with respect to their direction and magnitude. Therefore, if these two components should hold same sign, the dislocation has to move along y-direction for the motion of entire boundary (Fig 5.8). Since, \( (F^\text{dis}_{y})_2 \) is a positive quantity, hence, \( (F^\text{dis}_{y})_1 > 0 \) (should also be a positive component for moving boundary), therefore from equation (3.16),

\[
\tau_{xy} b \cos \theta / 2 \geq \sigma_{xx} b \sin \theta / 2
\]

with a consideration \( \tau_{xy} = \tau \) and \( \sigma_{xx} = \sigma \), equation (5.7) can be extended like as

\[
\tau \cos \theta / 2 \geq \sigma \sin \theta / 2 \quad \text{or} \quad \frac{\tau}{\sigma} \geq \tan \theta / 2
\]

Thus, for the motion of grain boundary (parallel geometry) along y-direction (Fig. 5.8), a primitive condition can be established as a function of the ratio of shear stress and normal stress component acting on the dislocation from the equation (5.8). In general case, the shear stress component is always lower than the normal stress component, and at the same time, the ratio of shear stress and normal stress \( (\tau/\sigma) \) is lower in case of low angle boundary than that of high angle boundary. In the present case, normal stress is always larger than the generated shear stress component. Therefore, this can be a strong evidence that motion of high angle grain boundaries are more preferable than low angle boundaries, in particular, for parallel geometry.
5.2 Cyclic motion of <112> tilt boundaries under elastic stress

Fig. 5.8. Schematic diagrams for the forces acting on the dislocations of grain boundaries in parallel to the stress axis during cyclic deformation.

On the other hand, the normal stress component cannot be neglected ($\sigma_{xx} \neq 0$), therefore, some force will be acting parallel to the boundary (x-direction) to make the boundary sliding along that direction. But the direction of force components ($F_{x\text{dis}}$) of $F_1$ and $F_2$ dislocations are just in opposite sign (equation 3.16), which results in negligible amount of force acting along the x-direction for sliding the boundary. Therefore, grain boundary motion is more prominent than the sliding of boundary parallel geometry, in turn; boundary trace-marks were less visible on the surface of the samples during motion.

5.2.4 Mechanism for the motion of 45° inclined boundaries with regard to dislocation dynamics

The activation energies for 45° inclined grain boundary motion were calculated to characterise the behaviour of low angle and high angle grain boundary motion under cyclic loading conditions (Table 4.7), which has been shown more similar activation energies for motion of grain boundaries with respect to purely shear stress driven motion [30-32]. In this case, grain boundary displacement in first cycle and in rest of the cycles were not separately calculated, because in this case also the displacement-markings were not so clearly seen into the resolution range of optical microscope.

Therefore, from the equation (3.28) it is clear that the force acts on dislocations in both x and y-directions. Hence, the motion of dislocation is possible along x-direction as well as y-direction i.e. parallel and perpendicular to the grain boundary plane respectively (Fig. 5.9).
The conditions of the motion of 45° inclined boundary has also been described in equations (3.30) and (3.32).

Based on the equation (3.32), it can be proposed that, for a low angle grain boundary 10-15° ≥ θ ≥ 0, hence 0 ≤ cosθ ≤ 1, therefore, a ratio of normal and shear stress will be established like as

\[
\left(\frac{\sigma}{\tau}\right)_{LAGB} \geq 2
\]

with taking simplified form of equation (5.9), the ratio of normal stress and shear stress will be

\[
\left(\frac{\sigma}{\tau}\right)_{LAGB} \geq 1
\]  

(5.10)

similarly, for a high angle grain boundary 45° ≥ θ ≥ 15°, hence 0.7 ≥ cosθ ≥ 1

\[
\left(\frac{\sigma}{\tau}\right)_{HAGB} \geq 2\sqrt{2}
\]

(5.11)

Taking a simplified version of equation of (5.11), we can get a ratio between normal stress and shear stress as in equation (5.12)

\[
\left(\frac{\sigma}{\tau}\right)_{HAGB} \geq \pm 1 + \sqrt{2}
\]

(5.12)

Finally, the equations (5.10) and (5.12) emphasise that during motion, the ratio of normal stress and shear stress is higher for high angle grain boundary. From the equation (5.13)

\[
\left(\frac{\sigma}{\tau}\right)_{HAGB} \geq \left(\frac{\sigma}{\tau}\right)_{LAGB}
\]

(5.13)
However, in the present case, the stress applied normal to the boundary was same in amplitude, thus from equation (5.14)

\[
\left( \frac{1}{\tau} \right)_{HAGB} \geq \left( \frac{1}{\tau} \right)_{LAGB} \quad \text{or} \quad \tau_{HAGB} \leq \tau_{LAGB}
\] (5.14)

Therefore, from the equation (5.14) it can be propose that for the motion of low angle grain boundary the shear stress was larger than for the high angle boundary with equal applied normal force.

On the other hand, microstructures have shown a totally different kind of grain boundary motion of 45° inclined CSL boundaries under cyclic deformations (Fig. 4.24 and 4.25). There was a clear evidence that migration rate is much higher at the edge of the samples as compared to the entire boundary motion, which causes the whole boundary to rotate either at the center or at the edge of the samples rather than motion. The major difference between 45° inclined tilt non-CSL boundaries and 45° inclined CSL boundaries was that the motion of non-CSL boundaries were less scattered than CSL boundaries. Several possibilities can be proposed to describe this kind of difference in motion. Firstly, in general CSL boundaries are more mobile than non-CSL boundaries and secondly, if grain boundaries are curved at the edge of samples, these two combinations, causes a drastic motion of boundary at edge compared to other part of boundaries.

5.2.5 Mechanism for the motion of other geometry (15°, 30°, 60° and 75° inclined) of grain boundaries

Another four types of geometrically different (15°, 30°, 60° and 75° inclined to the stress axis) grain boundaries (in addition to perpendicular, parallel and 45° inclination) were investigated to exhibit a comparison of cyclic motion of low angle and high angle boundaries (Fig. 4.26). There is always a scattered displacement profile with respect to its geometry and type of grain boundaries. Largest displacement was found in case of 45° inclined boundary geometry (without considering the 45° inclined CSL-boundary motion), although, Chen et. al. [61-63] reported that there was always a tendency the boundaries to move in order to align 45° with respect to the stress axis with increasing number of cycles and results in reduced grain boundary motion. As a support to this, Weiss et. al. [65-66] have also shown a similar kind of rotation of grain boundary at about 45° aligned with the stress axis and reduced grain boundary motion (Fig. 2.19), which essentially means that the 45° inclined position of grain boundary is the stable positions with regard to migration aspect, indeed, the present results show an opposite trend. Based on the present results we propose a possibility that the grain
boundary originally 45° inclined to the stress axis, has always largest shear stress during cyclic loading and that shear stress exerts a large force on the dislocation on grain boundary to move in a particular direction.

5.2.6 Driving force – grain boundary geometry interaction

We have already elaborated the driving force for the motion of grain boundaries with three specific geometries (perpendicular, parallel and 45° inclined to stress axis). From the equations (3.13), the driving force for perpendicular grain boundary motion can be rewritten as

\[ p^\perp = \tau \sin \theta \quad \text{and} \]

\[ \sigma \geq \tau \sin \theta \quad \text{for sliding of perpendicular grain boundary} \] (5.15)

\[ \frac{\sigma}{\tau} \geq \frac{\tan \theta}{2} \]

Similarly, from equation (5.8), for parallel grain boundary motion, a ratio of normal stress and shear stress was established as

\[ \tau \geq \sigma \tan \theta / 2 \]

If we combine equations (5.15) and (5.17), the driving force for the motion of parallel grain boundary can be established as

\[ |p^\parallel| \geq (\sigma \tan \theta / 2) \sin \theta \geq \sigma (1 - \cos \theta) \]

but for parallel boundary, the forces were balanced, therefore sliding was negligible.

Correspondingly, for 45° inclined grain boundary motion, several conditions were found. When grain boundary moves along the x-direction (Fig. 5.9), then the preliminary condition will be,

\[ \frac{\tau^2 b^2}{2} \cos \theta \geq 0 \]

whereas, when the boundary moves along the y-direction (Fig. 5.9), then the condition will be (from equation 3.32),

\[ \cos \theta \geq \frac{2\sigma \tau}{\sigma^2 + \tau^2} \]

If we combine the equations (5.18) and (5.20), the driving force for motion of 45° inclined grain boundary along the y-direction will be

\[ |p^{45\parallel}| \geq \sigma (1 - \frac{2\sigma \tau}{\sigma^2 + \tau^2}) \geq \frac{\sigma (\sigma - \tau)^2}{\sigma^2 + \tau^2} \]

(5.21)
Based on above all equations of driving force for the motion of different boundaries, it can be proposed that geometry has a strong effect on the mechanism of grain boundary motion under cyclic loading. At the same time, if we compare the possibility of sliding for all geometrically different boundaries, it was found that sliding was only prominent during the motion of perpendicular grain boundaries.

5.2.7 Summary of the section

1. The motion of <112> symmetrical tilt grain boundaries with different geometry’s was investigated under the influence of cyclic stress at elevated temperatures.

2. The nature of grain boundary displacement was different for different geometry. The displacement marks of grain boundary during deformation were seen distinctly on the surface of the bicrystals for perpendicular geometry, whereas, for other inclined boundaries, markings were not found so prominently.

3. The cyclic motion was described as a combination of sliding and migration mechanism. The motion in first cycle (in perpendicular geometry) is distinctly different with regard to its activation energies.

4. In case of perpendicular and parallel geometry, for low angle grain boundary motion, activation energies were found somewhat larger than the grain boundary self diffusion enthalpies (0.6-0.9 eV), whereas, for high angle boundary migration, activation energies were found smaller than the grain boundary diffusion enthalpies.

5. In case of 45° inclined geometry, for low angle grain boundary motion, activation energies (129.1 ± 21.1 kJ/mole) were close to volume diffusion enthalpies (1.25 eV), and for high angle grain boundary motion (79.3 ± 12.5 kJ/mole), it was close to grain boundary diffusion enthalpy (0.6-0.9 eV).

6. There were two sharp region of misorientation angle dependence of activation energy estimated. However, the transition angle between low angle and high angle grain boundary misorientaion angle was reasonably similar to the shear stress driven boundary migration values, i.e in present study it was on the range of 13.6° to 15.5°, whereas 13.6° was estimated into the previous experiments [30-32].

7. Driving force for motion of boundary was calculated by dislocation dynamics approach. Formations of trace-mark during cyclic motion were found strongly dependent on the direction of force acting on dislocation rather than the mode of deformation or inclination of boundaries.
8. A relation between normal stress and shear stress were established in term of the force on dislocation acting at the boundary. The contribution of sliding part and motion part during the entire boundary motion could be approximately predicted by the force acting on dislocation during deformation.

5.3 Cyclic motion of <100> tilt boundary under elastic stress

5.3.1 Motion mechanism of <100> tilt grain boundaries with regard to kinetics

The experimental results have convincingly shown that the <100> symmetrical planar tilt boundary was also movable under a stress field of cyclic deformation at different temperatures irrespective of misorientation angle and mode of deformation. Identical results were presented under a pure shear stress field at higher temperatures [29], although the type of stress was different. A strong misorientation angle dependence of activation energy during cyclic motion of <100> planar tilt boundary was observed (Fig. 5.10). The activation parameters were not calculated for first cycle and the rest of the cycle motion separately (compared to the motion of <112> tilt boundary). Because it has been seen from the previous results that the displacement of grain boundary was always larger than the displacement during the rest of cycles, although, in the present case it was difficult to measure the displacements separately. However, in case of perpendicular geometry, for low angle grain boundary motion, activation energies (146.6 ± 11.3 kJ/mole for 7.8° and 128.1 ± 14.4 kJ/mole for 6.8° grain boundary) were found to be close to the volume self diffusion enthalpies (1.25 eV), whereas for the motion of high angle grain boundaries, the activation energies (77.36 ± 17.0 kJ/mole for 9.2°, 78.1 ± 4.8 kJ/mole for 17.5° and 81.4 ± 11.9 kJ/mole for 33.9°) were comparable to the grain boundary self diffusion enthalpies (0.6-0.9 eV). The measured activation energy for the motion of grain boundary 3.7° <100> (73.2 ± 4.812 kJ/mole), was found exceptional, which may be due limited number of experimental data. Similarly, in case of parallel geometry, for motion of low angle grain boundary (6.6°), the activation energy (129.4 ± 7.4 kJ/mole) was found to be close to the volume self diffusion enthalpy (1.25 eV), meanwhile, for the motion of high angle grain boundary (34.5°), the activation energy (72.9 ± 11.3 kJ/mole) was close to the grain boundary self diffusion enthalpy. Moreover, for parallel geometry, the motion of low angle and high angle grain boundary is quite consisting from the activation energy point of view (Fig. 5.10). Moreover,
the profile of activation energy vs misorientation angle, established a transition in the range of 7.8° and 11.7° of misorientation angle of grain boundary with respect to its motion (except 3.7° <100> boundary).

![Fig. 5.10. Misorientation angle dependence of activation energy during motion of <100> tilt grain boundary with perpendicular and parallel geometry.](image)

However, a few of the <100> grain boundaries have shown different trend with respect to measured activation energies of motion, nevertheless, majority of cyclic grain boundary motion have shown similar trend as that for a pure shear stress driven motion. In the context, the mechanism for their motion will also be similar. As it was formerly reported [33] that the mechanism for the motion of <100> tilt grain boundary under pure shear stress is attributed to a sequential motion of dislocations by glide process due to applied stress and climb in opposite sense, henceforth, the dislocations have to move by a nonconservative motion in order to make the entire boundary migrate and to maintain the spacing of the structural dislocations. The climb process requires diffusion which can only be accomplished by volume diffusion for low angle grain boundaries, but grain boundary diffusion is sufficient for the motion of high angle grain boundaries as revealed from the observed activation enthalpies. In case of low angle grain boundaries, volume diffusion is needed to provide the vacancies for dislocation climb. In the context, the motion of planar high angle grain boundaries can be associated with the movement of dislocations in boundary, if it forms a continuous dislocation slab, vacancies can also reach the dislocations via grain boundary diffusion. It was also reported [33] that the activation enthalpies for grain boundary motion (pure shear stress
5.3 Cyclic motion of $<100>$ tilt boundaries under elastic stress

experiments) is associated with the climb processes as the climb process is much slower than the glide processes as it determines the boundary velocity [33]. On the other hand, the activation energy for grain boundary motion was estimated to be 76.1 ± 5.0 kJ/mole as reported by Raman and Langdon [53, 54, 56] for motion of grain boundary in high purity polycrystalline Al under reverse bending fatigue tests and emphasised that the cyclic motion mechanism follows a combination mechanism of sliding as well as migration. Although several authors [71] have reported that the sliding mechanism is essentially based on the movement of lattice dislocations along the grain boundary plane by a combination of dislocation climb and glide process, McLean proposed [73] that the crystal lattice dislocations can be regarded as being dissociated into two component dislocations, one of them having the Burgers vector normal to the grain boundary plane and the other with Burgers vector parallel to the grain boundary plane and hence, the grain boundary sliding take place due to the motion of the dissociated dislocations, which are having Burger vector parallel to the grain boundary plane. Therefore, the presently measured activation energies for cyclic grain boundary motion can be described as a combination of sliding and migration rather than pure migration mechanism.

The discrepancies between earlier [53, 56] reported results and the results of the present investigations can be attributed to the fact that earlier studies were on polycrystals, which has some demerits with regards to the effects of other features of grain boundary. For example, in case of polycrystal experiments, the activation energy of motion were close to the motion of a curved grain boundary, whereas for the present case, a planar boundary were used. Therefore, the presently obtained value of activation energy was more realistic as it was performed in bicrystals and could not be compared with the reported value in [53]. Nevertheless, the present work also has some limitations with regard to the calculation of the activation energy, which is already mentioned in section 5.2. Meanwhile, it has to be recalled that the presently measured activation energies during cyclic motion in bicrystals were more close to that for the shear stress driven boundary motion [33].

5.3.2 Mechanism for the motion of perpendicular $<100>$ tilt boundaries with regard to dislocation theory

The experimental results on the high-temperature cyclic plastic deformation of Al bicrystals has been discussed in section 5.1, and it is already proven that the motion of grain boundary was attributed to the differences in dislocation density of the grains caused by plastic deformation. However, in the present experiment, an elastic amplitude of stress (± 0.5 MPa)
was applied, which is even smaller than the critical resolved shear stress (1 MPa) for high-purity Al single crystals [81], thus, the deformation was fully elastic (see section 3.1), which means, without generation of any new dislocation. The mechanism of grain boundary motion under such a cyclic elastic deformation would be completely different from that under cyclic plastic deformation. The driving force for these type of motion of grain boundary has already been postulated in section 3.3 in case of <112> tilt grain boundaries, similarly for <100> tilt boundary, the driving force for motion can be calculated as follows.

Fig. 5.11. Schematic diagrams for the forces acting on the dislocations of <100> tilt grain boundaries in perpendicular to the stress axis during cyclic deformation a) complete dislocation arrangement on the grain boundary b) dissociation of edge dislocation into two lattice dislocation component.

The dislocation structure in <100> tilt grain boundary has been shown in Fig. 5.11a and the edge dislocation with Burgers vector \( b = a<100> \) can be dissociated into a pair of lattice dislocations with Burgers vector \( b_1 = a/2<110> \) and \( b_2 = a/2<1-10> \), as this model was supported by the experiments of Viswanathan and Bauer [42] and was proved by transmission
5.3 Cyclic motion of $<100>$ tilt boundaries under elastic stress

... electron microscopic study of grain boundary [82]. The lattice dislocation pairs are 45° inclined to the generated shear stress directions (Fig 5.11b), which will exert maximum shear stress on the dislocation to move.

As the dissociated lattice dislocations are 45° inclined to the applied stress axis, a shear stress acts on the grain boundary plane (Fig. 5.11b).

Where, stress tensor can be project as

$$\sigma = \begin{pmatrix} \sigma_{xx} & \tau_{xy} & \tau_{xz} \\ \tau_{xy} & 0 & 0 \\ \tau_{xz} & 0 & 0 \end{pmatrix}$$ (5.22)

and the burger vectors $b_1$ and $b_2$ and line element of dislocation $s$ will be

$$b_1 = \begin{pmatrix} \frac{b}{\sqrt{2}} \cos(\pi/4 - \theta/2) \\ \frac{b}{\sqrt{2}} \sin(\pi/4 - \theta/2) \\ 0 \end{pmatrix}, \quad b_2 = \begin{pmatrix} \frac{b}{\sqrt{2}} \cos(\pi/4 + \theta/2) \\ \frac{b}{\sqrt{2}} \sin(\pi/4 + \theta/2) \\ 0 \end{pmatrix} \quad \text{and} \quad s = \begin{pmatrix} 0 \\ 0 \\ -1 \end{pmatrix}$$ (5.23)

From the equations (5.22) and (5.23) the force on the dislocations can be summarised as

$$F_{1\text{dis}} = \sigma_{xx} \frac{b}{\sqrt{2}} \cos(\pi/4 - \theta/2) - \tau_{xy} \frac{b}{\sqrt{2}} \sin(\pi/4 - \theta/2)$$

and

$$F_{2\text{dis}} = \sigma_{xx} \frac{b}{\sqrt{2}} \cos(\pi/4 + \theta/2) - \tau_{xy} \frac{b}{\sqrt{2}} \sin(\pi/4 + \theta/2)$$ (5.25)

From the equation (5.25) it is clear that the force on dislocations acts only in x-direction and y-directions, therefore, the motion of dislocations are possible only either parallel and/or perpendicular to the grain boundary plane. Since, in experiments on perpendicular geometry, the grain boundary displacement was possible only along the x-direction and grain boundary sliding may take place along y-direction. Thus, the product of $(F_{1\text{dis}})$ and $(F_{2\text{dis}})$ as well as $(F_{y\text{dis}})$ and $(F_{y\text{dis}})$ should be positive, i.e., $(F_{x\text{dis}})_1 \cdot (F_{x\text{dis}})_2 \geq 0$ and $(F_{y\text{dis}})_1 \cdot (F_{y\text{dis}})_2 \geq 0$.

In case of $(F_{x\text{dis}})_1 \cdot (F_{x\text{dis}})_2 \geq 0$, with taking $\tau_{xy} \equiv \tau$, the force on dislocations...
5.3 Cyclic motion of \(<100>\) tilt boundaries under elastic stress

\[
\left( \tau - \frac{b}{\sqrt{2}} \cos(\pi/4 - \theta/2) \right) \left( \tau - \frac{b}{\sqrt{2}} \cos(\pi/4 + \theta/2) \right) \geq 0
\]

(5.26)

The solution of the equation (5.26) will be

\[
\frac{\tau^2 b^2}{4} \left( \cos(\pi/2 + \cos \theta) \right) \geq 0 \quad \text{or} \quad \frac{\tau^2 b^2}{4} \cos \theta \geq 0
\]

(5.27)

Equation (5.27) is a preliminary condition for the motion of perpendicular \(<100>\) tilt boundary along the x-direction.

As we know that \(\sigma_{xx} \neq 0\), therefore, there is always a possibility that a force acting along the y-direction results in the sliding of boundary along y-direction. Thus, in case of \((F_y^{dis})_1\),

\[
(F_y^{dis})_x \geq 0, \text{ taking } \tau_{xy} = \tau, \text{ and } \sigma_{xx} = \sigma, \text{ the force on dislocations}
\]

\[
\left( \sigma - \frac{b}{\sqrt{2}} \cos(\pi/4 - \theta/2) - \tau \frac{b}{\sqrt{2}} \sin(\pi/4 - \theta/2) \right) \left( \sigma - \frac{b}{\sqrt{2}} \cos(\pi/4 + \theta/2) - \tau \frac{b}{\sqrt{2}} \sin(\pi/4 + \theta/2) \right) \geq 0
\]

(5.28)

Taking the simplified form of the equation (5.28), we can get

\[
(\sigma^2 + \tau^2) \cos \theta - 2\sigma \tau \geq 0
\]

(5.29)

Similarly, the equation (5.29) is a preliminary condition for sliding of perpendicular \(<100>\) tilt grain boundary along the y-direction.

As we have already seen, in particular, under the influence of a pure shear stress field, the grain boundary motion was basically associated by the motion of structural dislocations of grain boundaries. It is firmly believed that such a hypothesis also applies to the present situation, and the \(<100>\)-tilt GB migration behaviour under cyclic elastic deformation should be discussed in terms of properties of dislocation arrays of a [100](010), (Fig. 5.11) which comprise grain boundary, as described in Section 3.1. However, owing to rather different loading modes applied, i.e. the constant shear stress and alternating cyclic stress, the GB migration features and mechanisms are definitively distinctive for the both cases.

Another prominent difference in the motion feature is that some displacement markings can always be detected during cycling in the perpendicular geometry of boundary, but never seen in the parallel geometry (except 34.5° and 42.5° grain boundary motion) as well as during constant pure shear stress induced grain boundary motion [33]. The force component parallel to the grain boundary plane, as expressed by Equation (5.25), which would impel a dislocation sliding along the grain boundary plane. As it is well known, the migration markings are due to steps produced at the specimen surface by grain boundary sliding and
deduced an optimum ratio of normal stress and shear stress in terms of dislocation arrangement, which can give a small displacement of crystal perpendicular to the specimen surface. It is thereby demonstrated that, besides grain boundary motion, sliding of boundary has occurred concurrently under the action of a cyclic stress which may not be present under the action of a constant shear stress or was very less for parallel geometrical boundary motion, as force component parallel to the boundary was balanced to each other. In contrast, in the case of motion of grain boundary under the action of a constant pure shear stress, the derived force component along the grain boundary plane is nearly close to zero. Therefore, it is easy to understand why the GB sliding occurred in the case of perpendicular geometry but did not prominently take place under the application of constant pure shear stress or in parallel geometry. Such a difference of grain boundary sliding would undoubtedly lead to different motion behaviour, because it was already reported that cyclic motion of grain boundary is a combination of migration and sliding [46, 49]. In addition, it should be mentioned that an approximate one-to-one correspondence between the number of visible migration markings on the specimen surface and the number of imposed cycles, which was often seen in the cyclic grain boundary motion in pure Al under reverse bending fatigue tests [46-49].

5.3.3 Mechanism for the motion of parallel <100> tilt boundaries with regard to dislocation theory

Grain boundary motions of parallel geometry were also found for all type of <100> tilt boundaries. The detailed mathematical calculation for the stress components acting on dislocations and the possible driving force can be measured in the following way. The dislocation array, the dissociated lattice dislocations and the direction of forces acting on the dislocation on the grain boundary is illustrated in (Fig. 5.12a and b). The applied force on the grain boundary is assumed to be 45° inclined to the dissociated lattice dislocations. As the dissociated lattice dislocations will be 45° inclined to the applied stress axis, an amount of shear stress acts on the grain boundary plane (Fig. 5.12b). The stress tensor can be written as

$$\sigma = \begin{pmatrix} \sigma_{xx} & \tau_{xy} & \tau_{xz} \\ \tau_{xy} & 0 & 0 \\ \tau_{xz} & 0 & 0 \end{pmatrix}$$

(5.30)

and the burger vectors $b_1$ and $b_2$ and line element of dislocation $s$ are given by
5.3 Cyclic motion of $<100>$ tilt boundaries under elastic stress

Fig. 5.12. Schematic diagrams for the forces acting on the dislocations of $<100>$ tilt grain boundaries in parallel to the stress axis during cyclic deformation a) complete dislocation arrangement on the grain boundary b) dissociation of edge dislocation into two lattice dislocation component.

From the equations (5.30) and (5.31) the force on the dislocations can be summarised as

$$F_{1 \text{dis}} = \begin{pmatrix} \tau_{xy} \frac{b}{\sqrt{2}} \sin(\pi/4 - \theta/2) \\ - \sigma_{xx} \frac{b}{\sqrt{2}} \sin(\pi/4 - \theta/2) + \tau_{xy} \frac{b}{\sqrt{2}} \cos(\pi/4 - \theta/2) \\ 0 \end{pmatrix}$$

and

$$F_{2 \text{dis}} = \begin{pmatrix} \tau_{xy} \frac{b}{\sqrt{2}} \sin(\pi/4 + \theta/2) \\ - \sigma_{xx} \frac{b}{\sqrt{2}} \sin(\pi/4 + \theta/2) + \tau_{xy} \frac{b}{\sqrt{2}} \cos(\pi/4 + \theta/2) \\ 0 \end{pmatrix}$$

From the equation (5.32) it is clear that the force acts on dislocations only in x-direction and y-directions, therefore, the motion of dislocations are possible only in a direction parallel and/or perpendicular to the grain boundary plane. Since, in experiments on parallel geometry, the grain boundary displacement is possible only along the y-direction and grain boundary
sliding may take place along x-direction. Thus, the product of \((F_{x_1}^{\text{dis}})\) and \((F_{x_2}^{\text{dis}})\) as well as \((F_{y_1}^{\text{dis}})\) and \((F_{y_2}^{\text{dis}})\) should be positive, i.e., \((F_{x_1}^{\text{dis}}) \cdot (F_{x_2}^{\text{dis}}) \geq 0\) and \((F_{y_1}^{\text{dis}}) \cdot (F_{y_2}^{\text{dis}}) \geq 0\).

In case of \((F_{x_1}^{\text{dis}}) \cdot (F_{x_2}^{\text{dis}}) \geq 0\), with taking \(\tau_{xy} \equiv \tau\), the force on dislocations

\[
\left\{ \frac{b}{\sqrt{2}} \sin(\pi/4 - \theta/2) \right\} \left\{ \frac{b}{\sqrt{2}} \sin(\pi/4 + \theta/2) \right\} \geq 0
\]

(5.33)

The solution of the equation (5.33) will be

\[
\frac{\tau^2 b^2}{4} \left( \cos \theta - \cos \pi/2 \right) \geq 0 \quad \text{or} \quad \frac{\tau^2 b^2}{4} \cos \theta \geq 0
\]

(5.34)

therefore, the equation (5.34) is a preliminary condition for the sliding of parallel <100> tilt boundary along the x-direction, which is valid for each grain boundary with \(0 \leq \theta \leq \pi/2\) of misorientation angle.

As we know that \(\sigma_{xx} \neq 0\), therefore, there is always a possibility that a force acting along the y-direction leads the boundary to move along y-direction. Therefore, in case of \((F_{y_1}^{\text{dis}}) \cdot (F_{y_2}^{\text{dis}}) \geq 0\), taking \(\tau_{xy} \equiv \tau\), and \(\sigma_{xx} = \sigma\) the force on dislocations

\[
\left\{ \frac{b}{\sqrt{2}} \cos(\pi/4 - \theta/2) - \sigma \frac{b}{\sqrt{2}} \sin(\pi/4 - \theta/2) \right\} \left\{ \frac{b}{\sqrt{2}} \cos(\pi/4 + \theta/2) - \sigma \frac{b}{\sqrt{2}} \sin(\pi/4 + \theta/2) \right\} \geq 0
\]

(5.35)

Taking the simplified form of the equation (5.35) we can get

\[
(\sigma^2 + \tau^2) \cos \theta - 2\sigma \tau \geq 0
\]

(5.36)

Similarly, the equation (5.36) is a preliminary condition for displacement of parallel <100> tilt grain boundaries along the y-direction.

In this case, the displacement-markings were not so clearly seen within the resolution range of optical microscope. By virtue of the nature of parallel boundary motion being very much similar to the shear stress driven boundary motion. For instance only the initial and final position of the grain boundary could be seen into the microstructures.

### 5.3.4 Summary of the section
1. The motions of $<100>$ symmetrical tilt grain boundaries with two specific geometry (perpendicular and parallel) were investigated under the influence of cyclic stress at elevated temperatures.

2. The nature of grain boundary displacement is not different for differently inclined boundary as compared to the $<112>$ boundaries.

3. The cyclic motion was described as a combination of sliding and migration mechanism. The measured activation energy for motion of $<100>$ tilt grain boundary has shown a trend similar to pure shear stress driven grain boundary motion.

4. In case of perpendicular geometry, for low angle grain boundary motion, activation energies ($146.6 \pm 11.4$ kJ/mole for $7.8^\circ$ and $128.15 \pm 14.4$ kJ/mole for $6.8^\circ$ grain boundary) were found to be close to the volume self diffusion enthalpies ($1.25$ eV), whereas for the motion of high angle grain boundaries, the activation energies ($77.3 \pm 17.1$ kJ/mole for $9.2^\circ$, $78.1 \pm 4.8$ kJ/mole for $17.5^\circ$ and $81.4 \pm 11.9$ kJ/mole for $33.9^\circ$) were found closely comparable to the grain boundary self diffusion enthalpies ($0.6-0.9$ eV).

5. In case of parallel geometry, the activation energy for motion of low angle grain boundary ($129.5 \pm 7.4$ kJ/mole for $6.6^\circ$ grain boundary) was found to be close to the volume self diffusion enthalpy ($1.25$ eV), whereas, for the motion of high angle grain boundary ($34.5^\circ$), the activation energy ($72.9 \pm 11.3$ kJ/mole) was found to be close to the grain boundary self diffusion enthalpy.

6. Two sharp regions of misorientation dependence of activation energy were also estimated for the motion of $<100>$ tilt boundary. The transition angle between low angle and high angle grain boundary misorientation was estimated in the range of $7.8^\circ$ to $11.7^\circ$, whereas, $8.6 \pm 0.15^\circ$ was estimated for the pure shear stress driven grain boundary motion [33].

7. Driving force for motion of boundary was calculated using dislocation dynamics approach. Formation of trace-mark during cyclic motion were found to be strongly dependent on the direction of force acting on dislocations.

8. A relation between normal stress and shear stress can be established in terms of dislocation arrangement of grain boundaries. The contribution of sliding part and movement part during the entire boundary motion could roughly be predicted by the force acting on the dissociated dislocation during deformation.
5.4 Cyclic motion of <100> twist boundary under elastic stress

5.4.1 Motion mechanism of <100> twist grain boundaries with regard to kinetics

The observed results have shown that the <100> symmetrical planar twist boundary was also mobile under a stress field of cyclic deformation at different temperatures irrespective of misorientation angle and mode of deformation, although, the displacement of twist boundary was very much scattered with respect to tilt boundaries. The results from pure shear stress experiments [74] indicate a lower range of activation enthalpies as compared to the formerly measured activation enthalpies for tilt boundary motion. However, the present result is limited to low misorientation angle regime with 5.9° to 11.2°, majority of the measured activation energies (both in perpendicular and parallel geometry) have shown identical value of activation enthalpies (Table 5.3). Most of the grain boundaries have shown non-uniform motion at higher temperature conditions. Nevertheless, a misorientation angle dependence of activation energy during cyclic motion of <100> planar twist boundary is also presented (Fig. 5.13). The activation parameters were not calculated separately (compared to the motion of <112> tilt boundary) for first cycle and the rest of the cycle motion. In general, previous results have shown that the displacement of grain boundary was always larger than the displacement during the rest of cycles. In the present case, however, it was difficult to measure the displacements separately. In case of perpendicular geometry, the activation energies for the motion of low-angle twist boundary were measured 74.3 ± 0.4 kJ/mole for 5.9° and 60.8 ± 16.4 kJ/mole for 10.5° grain boundary, whereas for the motion of comparatively large angle grain boundaries, the activation energy was 110.8 ± 10.3 kJ/mole for 11.2°. Similarly, in case of parallel geometry, the activation energy for motion of 9.3° <100> and 10.9° <100> twist grain boundaries was found 78.4 ± 19.7 kJ/mole and 93.8 ± 12.6 kJ/mole respectively. Correspondingly, under constant shear stress [74], the activation enthalpy for the motion of the low-angle boundary was measured \( \Delta H = 0.48 \text{ eV} \pm 0.04 \text{ eV} \) and for the high-angle grain boundary \( \Delta H = 0.25 \text{ eV} \pm 0.02 \text{ eV} \) and the transition angle between low angle and high angle was found at misorientation angle of 9.8°. The most probable reason for this may be due to reaction of screw dislocation under cyclic deformation being different, which may cause the motion of twist boundary very much non-uniform, even sometimes bulging of boundary (see section 4.4). As the present study was limited to the
motion of low angle grain boundary (Table 5.3), transition angle between low angle and high angle grain boundary with respect to activation energy was not established.

Fig. 5.13. Misorientation angle dependence of activation energy during motion of <100> twist grain boundary with perpendicular and parallel geometry.

However, a comparison of activation energies with different grain boundary has been presented in Table 5.3. A method to realise a constant driving force concept for planar grain boundaries was given by Schönfelder et. al. [75-76] where for the first time grain boundary motion of a planar $\Sigma 29$ <100> twist grain boundary due to an elastic driving force was reported in a computer simulation study of Cu bicrystals. Comparing the results of the present work and the data from the work of Nomura and Adams [77], a similar orientation dependence of the activation energy was observed, i.e. the activation energy for the motion of comparatively larger angle grain boundaries have shown larger values, which were different from the measured activation energy in case of constant shear stress experiments [74].

5.4.2 Mechanism for the motion of twist grain boundaries with regard to dislocation theory

The measured activation parameters for twist grain boundary motion was comparatively larger than the reported results in the literature [74, 76], in contrast, it was also demonstrated that especially for the $\Sigma 181$, $\Sigma 85$, and $\Sigma 41$ twist grain boundaries, which have comparatively lower misorientation angle (e.g. 6.03°, 8.8° and 12.7° respectively), the activation enthalpies
lie in the range of 0.507 ±0.02 eV to 0.585 ± 0.081 eV. These values are more comparable to the presently obtained activation enthalpies; therefore, the mechanism of motion of the presently investigated grain boundaries under cyclic deformation should follow a similar mechanism. The atomistic simulation on the migration of twist grain boundaries by Schöpfelder et. al. [76] reported that the low angle twist boundaries consisted of discrete screw dislocation networks that remained stable at higher temperature range. The discrete cores of the screw dislocations of the low angle twist boundaries were identified by utilising the CNA (common-neighbour structural analysis) scheme which showed that the crystal lattice does continue smoothly across the grain boundary region outside of the screw dislocation cores in the boundary.

Table 5.3 Comparison of activation energies with the activation enthalpies of diffusion during motion of planar symmetrical <100> twist boundaries. GB plane was perpendicular and parallel to the stress axis.

<table>
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<th>Initial θ(°)</th>
<th>Q_CGMB (D_N) [kJ/mole]</th>
<th>GB Geometry</th>
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<tr>
<td>5.9° ± 0.2</td>
<td>74.34 ± 0.4</td>
<td>Perpendicular Geometry</td>
</tr>
<tr>
<td>7.9° ± 0.5</td>
<td>61.55 ± 7.2</td>
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<td>9.2° ± 0.5</td>
<td>69.4 ± 10.3</td>
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<tr>
<td>10.5° ± 0.1</td>
<td>60.8 ± 16.4</td>
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</tr>
<tr>
<td>11.2° ± 0.1</td>
<td>110.8 ± 10.3</td>
<td></td>
</tr>
<tr>
<td>9.3° ± 0.5</td>
<td>78.4 ± 19.7</td>
<td></td>
</tr>
<tr>
<td>10.9° ± 0.1</td>
<td>93.8 ± 12.7</td>
<td>Parallel Geometry</td>
</tr>
</tbody>
</table>

The structural analysis revealed that the migration mechanism of a low angle twist boundaries was linked to a movement of the screw dislocation network itself. In contrast, the high angle twist boundaries moved in the low temperature regime by collective shuffle mechanisms, which were basically in-plane displacements. Correspondingly, a more realistic approach of the motion mechanism of twist boundary with regard to the movement of screw dislocations by cross slip has been proposed by Winning [74], during the motion of twist boundary under a constant shear stress. The mechanism of motion of the twist boundary already discussed was based only under a constant shear stress and from computer simulation results. At the same time, the mechanism was not completely understood. The present study was even more complicated as the applied stress was cyclic normal stress.

The results of the present investigation can be rationalise by our text book knowledge as follows. The twist grain boundaries essentially consist of a network of screw dislocations and the screw dislocations can only move by cross slip process, as its Burgers vector are parallel...
to the line of dislocations so as to glide on any plane as long as it moves parallel to its original orientation. The slip plane of screw dislocation can be any plane containing the dislocation, and it can cross slip from one plane to another just so long as both planes contain a common slip direction. If cross slip of screw dislocations could not occur, dislocation motion would be impeded during the deformation process. In general, the formation of jogs in screw dislocations impedes their motion and may even lead to the formation of vacancies and interstitial if the jogs are forced to move non-conservatively. However, for the screw dislocations, the jogs all have edge orientation, and since edge dislocations move freely on its slip line and along the Burgers vector, the only way a jog can move by slip is along the axis of screw dislocation. The possibility that the screw dislocation can move with its jog is by a non-conservative process like climb process. In principle, dislocation climb is a thermally activated process; therefore the movement of jogged screw dislocation will be temperature dependent, henceforth, at the temperature where climb cannot occur, the motion of screw dislocation will be hindered by jog formation. Therefore, the movement of screw dislocation through crystal is much slower than an edge dislocation, which was experimentally observed by Chen and Pond [78]. The present result also shows a slower velocity of grain boundary motion, in turn, the measured activation energies were found larger.

5.4.3 Summary of the section

1. The motion of <100> twist grain boundary was also possible under a elastic cyclic stress at higher temperatures, indeed, the motion was very much scattered and non-uniform.
2. The activation energy was also measured from the displacement data to investigate the kinetics of the twist boundary and compared to the formally done experimental results on tilt boundaries.
3. In case of perpendicular geometry, for low angle twist grain boundary motion, activation energies (74.3 ± 0.4 kJ/mole for 5.9° and 60.8 ± 16.4 kJ/mole for 10.5° grain boundary) was close to the grain boundary self diffusion enthalpies (0.6-0.9 eV), whereas for the motion of comparatively large angle grain boundaries, the activation energies (110.8 ± 10.3 kJ/mole for 11.2°) were comparable to the volume self diffusion enthalpies (1.25 eV).
4. In case of parallel geometry, the activation energy for motion of 9.3° <100> and 10.9° <100> twist grain boundaries was found to be 78.4 ± 19.7 kJ/mole and 93.8 ± 12.6 kJ/mole respectively, which was close to the grain boundary self diffusion enthalpy (0.6-0.9 eV).
5. The transition angle between low angle and high angle grain boundary was difficult to establish due limited number of samples. Nevertheless, the average measured activation energy was found to be comparable to the activation enthalpies of grain boundary self diffusion.

6. The motion mechanism of the \(<100>\) twist boundary was discussed with respect to the movement of structural screw dislocation by cross slip.
Chapter 6

Summary and Conclusions

The motion for <112> tilt, <100> tilt and <100> twist grain boundaries were investigated under two specific conditions of cyclic deformation. The first part of the investigation was performed under plastic amplitude of stress and the second part was performed completely under elastic amplitude of stress. Although, the motion of grain boundary was found in both the case, nevertheless, the mechanism, kinetics and the source of driving force was different. The conclusion from this work would be postulated in two parts.

6.1 Conclusions from Part-1

1. The motion of planar, symmetrical <112> tilt low angle and high angle grain boundaries in aluminium was possible under an influence of high temperature cyclic stress field (plastic range) with different combinations of deformation parameters. For low angle grain boundaries, the movement of grain boundaries was not possible as long as the misorientation angle of the boundary was below a critical range of 13.8°. Grain boundaries with misorientation angle above 13.8° were found to be mobile under cyclic deformation conditions.

2. The deformed structure of bicrystal shows that a heterogeneous deformation into the grains and generate a difference in dislocation density between the grains, which was the driving force for motion.

3. The differences of slip density ($\Delta f_i$) and Schmid factors between the two grains of the deformed structures of bicrystal were observed most accountable for indexing of driving force of cyclic motion of grain boundaries.

4. The effect of cyclic stress was relatively more clear and comparable. Larger stress reduces the chance for grain boundary motion due to homogeneous deformation structure in the two grains.
6.2 Conclusions from Part-2

1. The motion of <112> and <100> symmetrical tilt and <100> twist grain boundaries with different geometry was investigated under the influence of cyclic stress at elevated temperatures.

2. The nature of <112> and <100> tilt grain boundary displacement was different for different geometry. The displacement marks of grain boundary during deformation were seen distinctly on the surface of the bicrystals for perpendicular geometry, whereas, for other inclined boundaries, markings were not found so prominently. At the same time, grain boundary motion of <100> twist boundaries were completely different in nature as compared to the tilt boundaries. Boundary displacement was very much scattered along the grain boundary with some kind of bulging and snaky motion.

3. The cyclic motion of tilt boundary was described as a combination of sliding and migration mechanism. The motion of <112> tilt boundary in first cycle (in perpendicular geometry) is distinctly different with respect to measured activation energies. The activation energy for <100> tilt grain boundary motion in first cycle was not separately calculated as the displacement marking was not seen separately in microstructure.

4. In case of perpendicular and parallel geometry (for <112> tilt), for low angle grain boundary motion, the measured activation energies were found somewhat larger than the grain boundary self diffusion enthalpies (0.6-0.9 eV), whereas, for high angle boundary migration, activation energies were found somewhat smaller than the grain boundary diffusion enthalpies.

5. In case of 45° inclined geometry (for <112> tilt), for low angle grain boundary motion, activation energies (129.1 ± 21.1 kJ/mole) were close to volume diffusion enthalpies (1.25 eV), and for high angle grain boundary motion (79.3 ± 12.5 kJ/mole), it was close to grain boundary diffusion enthalpy (0.6-0.9 eV).

6. In case of perpendicular geometry (for <100> tilt), for low angle grain boundary motion, activation energies (146.6 ± 11.3 kJ/mole for 7.8° and 128.15 ± 14.4 kJ/mole for 6.8° grain boundary) were found to be close to the volume self diffusion enthalpies (1.25 eV) whereas for the motion of high angle grain boundaries, the activation energies (77.4 ± 17.1 kJ/mole for 9.2°, 78.1 ± 4.8 kJ/mole for 17.5° and 81.4 ± 11.9 kJ/mole for 33.9°) were found comparable to the grain boundary self diffusion enthalpies (0.6-0.9 eV).

7. In case of parallel geometry (for <100> tilt), the activation energy for motion of low angle grain boundary (129.5 ± 7.4 kJ/mole for 6.6° grain boundary) was found close to the
volume self diffusion enthalpy (1.25 eV). For the motion of high angle grain boundary (34.5°), the activation energy (72.9 ± 11.3 kJ/mole) was found close to the grain boundary self diffusion enthalpy.

8. In case of perpendicular geometry (for <100> twist), the activation energies for motion of twist boundaries, (74.34 ± 0.4 kJ/mole for 5.9° and 60.8± 16.4 kJ/mole for 10.5° grain boundary) were found close to the grain boundary self diffusion enthalpies (0.6-0.9 eV). For the motion of comparatively large angle grain boundaries, the activation energy (110.8 ± 10.3 kJ/mole for 11.2°) was found comparatively larger than the grain boundary self diffusion enthalpies (1.25 eV). Similarly, in case of parallel geometry, the activation energy for motion of 9.3° <100> and 10.9° <100> twist grain boundaries was found 78.4 ± 19.7 kJ/mole and 93.8 ± 12.7 kJ/mole respectively.

9. Two distinct regions of misorientation angle dependence of activation energy were estimated for <112> and <100> tilt boundary motion irrespective of geometry. In case of <112> tilt boundary, the transition angle between low angle and high angle grain boundary misorientation were reasonably similar to the shear stress driven boundary motion values. In the present study it was on the range of 13.6° to 15.5°, whereas 13.6 ± 0.5° was found in the previous experiments [30-32]. Similarly, in case of <100> tilt boundary, the transition angle between low angle and high angle grain boundary misorientation was estimated in the range of 7.8° to 11.7°. For the pure shear stress driven grain boundary motion experiments [33], 8.6 ± 0.15° was estimated. For twist boundary motion, in general, transition angle between low angle and high angle grain boundary was difficult to establish with respect to the measured activation energy due to limited number of samples. Nevertheless, the average measured activation energy for the motion of twist boundary was found to be comparable to the activation enthalpies of grain boundary self diffusion.

10. Driving force for motion of <112> and <100> tilt boundaries were calculated by dislocation dynamics approach, assuming that the grain boundary is basically controlled by the movement of structural edge dislocations. Formations of trace-mark during cyclic motion were found strongly dependent on the direction of acting force on dislocation rather than the mode of deformation. The motion mechanism of the <100> twist boundary was discussed with respect to movement of structural screw dislocation by cross slip.

11. A relation between normal stress and shear stress were established in term of the dislocation arrangement of the grain boundary. The contribution of sliding part and
movement part during the entire boundary motion can approximately be predicted by the force acting on dislocation during deformation.

12. Finally, inclination of grain boundaries with stress axis has a significant role during cyclic motion of grain boundaries with regards to its kinetics.
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**LEBENSLAUF**

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Zuständig für die Übung und Praktikum des Faches Metallkunde für die Bachelor-Studenten.

Aachen, November 2004