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# GRAIN BOUNDARIES AS DISLOCATION SOURCES

by

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## ABSTRACT

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An investigation was conducted into the role of grain boundaries as sources of lattice dislocations during yielding. An extensive review of the literature was conducted in order to establish a firm base for the study of this relatively new field. This included grain boundary structural models, the defect structure associated with the boundary, previous experimental confirmation of grain boundary source operation, and the proposed models for such sources, including the stress required and the means of enhancing the applied stress to this value.

An electron microscope examination was then conducted on several materials which possessed microstructures conducive to boundary sources and which had been strained to points well below and up to the yield point. Two techniques were developed to aid the thin foil examination. One led to a reduction in thin foil deformation due to foil handling, and the other was a thin foil mapping technique to provide comparative data on the densities and distributions of lattice defects pertinent to the

yield process.

The experimental results indicated that the great majority of boundary sources were nonregenerative in nature, and involved the nucleation and emission of both perfect and partial dislocations from grain boundary ledges. These dislocations were nucleated at low stresses and many were retained at the boundary. Emission occurred preferentially from triple points in the early stages of yielding. The proportion of strain contributed by boundary sources was negligible in high purity Al. In high purity Cu, the most extensively studied material, it was significant in only the first stage of what appeared to be a two-stage yielding process common to all the materials. In the second stage grain interior sources were predominant. In medium purity Ni, boundary sources were present in substantially larger numbers, and in Cu - 1 wt % Sn, they were present in sufficient numbers to control the major portion of the entire yield process. A number of factors, such as stacking fault energy or elastic anisotropy, appeared to influence boundary source characteristics or their operation, but the most important were the initial states of the boundaries and the distribution of solute or impurity atoms within the material.

With the experimental observations in mind, a detailed model of boundary sources was constructed. It

proposes dislocation nucleation from groups of small boundary ledges, with the ledge geometry providing the major portion of the necessary stress concentration for this nucleation. Additional stress concentration is supplied by interaction of the stress fields from the ledges within each group. The variation in the number, size and spacing of ledges within each group, along with the variation in individual ledge geometry, account for the observed non-homogeneous distribution of boundary sources. Several specific aspects of this model are then discussed, as well as its implications for other important facets of mechanical behavior.

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# TABLE OF CONTENTS

Page

Page

# LIST OF TABLES

# LIST OF MICRO-MAPS

# LIST OF FIGURES

Page

Page

Page

Page

## 1.0 INTRODUCTION

An overwhelming majority of engineering materials are used in the polycrystalline state, hence grain boundaries have always been considered to play an important role in plastic deformation. This role has centered around their ability to act as obstacles to the movement of dislocations. Thus, the classical concept has long been one of yield initiation via dislocation generation in grain interiors and subsequent dislocation pileups against the boundaries causing source activation in the next grain. However, during the past decade, an increasing amount of evidence has established that grain boundaries can also act as dislocation sources, particularly in the early stages of yielding. This region is generally referred to as the premacroyield strain region, and it extends from the first substantial movement of dislocations to the point where massive dislocation movement and multiplication is required to maintain the plastic strain rate imposed by the testing machine. It thus separates the microyield region (plastic strain of zero to around $1 \times 10^{-4}$) from the macroyield region (yield point).

The operation of grain boundary sources in the premacroyield region would have a number of consequences:

1) It might affect either the type, character or number per source of dislocations generated,
2) It would affect the work hardening behavior of materials in which cross-slip, and hence tangling, is relatively easy. This behavior would be affected by the changed location of these tangles from

the grain interior to the grain perimeter when boundary sources predominate,

3) The work hardening behavior with respect to solute atoms or particles hindering dislocation movement would be also affected, since solute levels and distribution at the grain boundary can be different from those of the grain interior in many materials.

In addition, the possibility of grain boundaries acting as dislocation sources may have implications for mechanical properties above the yield point, such as creep and fatigue strengths, or the amount of ductility a material possesses.

Although the concept of grain boundary dislocation sources has been formulated for some time, relatively little detailed experimental work has been conducted on their operation or their influence on the yielding process. Therefore this study was directed towards the following objectives:

1) To examine and correlate the many theoretical models for grain boundary structure and grain boundary lattice defects, dislocation nucleation at, and generation from these boundaries, the stresses required for operation of boundary sources and the means of obtaining these stresses,

2) To verify that grain boundary dislocation generation can occur in the premacroyield region of a pure FCC metal (Cu) in which it had not previously been observed, by designing a microstructure conducive to such generation,

3) To develop a method of extracting quantitative data (via electron microscope thin foil examination) concerning boundary source operation,

4) To use this method for measuring relative source densities and distributions in Cu at various strains in the premacroyield region,

5) To study some of the parameters, such as solute content, stacking fault energy, elastic anisotropy and initial boundary defect density, that should influence boundary source operation, by examining selected materials (Cu-1wt%Sn, Ni, Al) in a similar microstructural state to that of the Cu,

6) To examine the characteristics of individual sources in any material, for the purpose of establishing a boundary source model which will account for these characteristics and those determined in 4) and 5).

# 2 GRAIN BOUNDARY STRUCTURE AND ASSOCIATED CRYSTAL DEFECTS

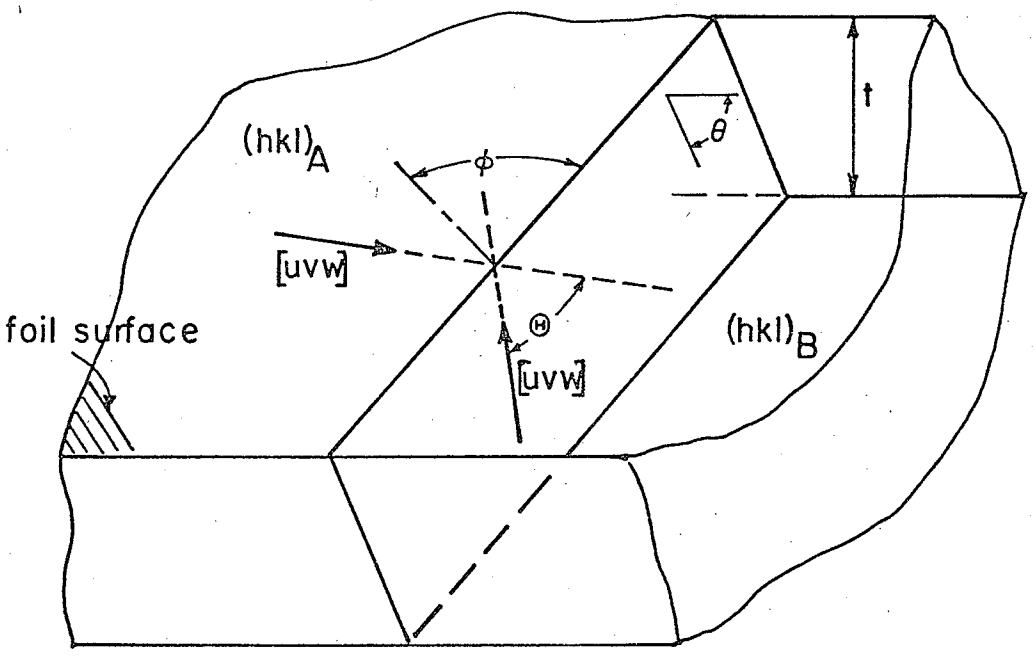
A grain boundary may be defined as the contact region between two crystals differing only in orientation (1). This definition, although relatively straightforward, sheds little light on the nature of this region, either on the scale of the individual atoms or the larger scale of the various defects present among the arrays of atoms. It is essential to realize that the strength of polycrystals indicates the presence of strong interatomic forces across this region. Thus it is not a "space" between two crystallites (as often represented in drawings) but a transition zone or a special lattice between the two misoriented crystal lattices. It accordingly possesses its own special characteristics, particularly with regard to the interaction of different types of dislocations. The character is also highly variable, and must change as the misorientation changes. This zone is spatially more restricted than our common notion of a lattice, being essentially a two-dimensional film curved so as to surround the grains in three dimensions. This latter point is particularly easy to forget since we are accustomed to observing "ribbons" of grain boundary in transmission electron micrographs.

## 2.1 GRAIN BOUNDARY PARAMETERS

Before studying the various models which have

been postulated to describe the structure of boundaries, it would be valuable to describe how a boundary is defined with respect to the crystallites it separates. If the crystallites are infinitely large, three angular parameters define their misorientation; one a rotation about an axis normal to the mirror plane separating them (twist component) and two about mutually perpendicular axes within this plane (tilt components). This mirror plane is normally the boundary plane (symmetric boundary), but does not necessarily have to be (e.g. in an asymmetric boundary). For the more normal case of a boundary in a thin foil, two additional parameters describe the orientation of the boundary plane to the foil (2). As shown in figure 1, these are $\theta$, denoting the inclination of the boundary to the foil surface, and $\phi$, denoting the angle between the boundary - foil intersection and $\theta$, the misorientation of a common crystallographic direction. This latter parameter encompasses the three angular parameters described above. This normally suffices to accurately reference the boundary, but with the advent of increasingly sophisticated models, Chalmers (3) has recently proposed that an additional three orthogonal inrotational translations are required to describe the relaxation of individual atoms at the boundary. However necessary these may be theoretically, they are as yet of

Figure 1. Geometrical conventions for characterizing a grain boundary (ABCD) in a thin foil of finite thickness, t (after Murr et al, 2).


(hkl)A
φ
θ
t
[uvw]
foil surface
Θ
(hkl)B
[uvw]

limited importance experimentally, thus the first five parameters are considered sufficient for most boundary descriptions. It should be pointed out that the above symbols are somewhat confusing in that a great many authors use the lower case theta ($\theta$) to denote the misorientation angle and the lower case alpha ($\alpha$) to denote the inclination angle of the boundary with the foil surface.

## 2.2 MODELS OF GRAIN BOUNDARY STRUCTURE

There are two basic approaches to considering models for grain boundaries (4). In one approach the boundary is viewed as a smooth, homogeneous layer purely for thermodynamic purposes. In the other, the basic structure of this layer is described. This study deals with the latter. Gifkin states (4) that a model must satisfy the following criteria to be completely acceptable:

a) it must result in a boundary width of three to four atom diameters to correlate with experimental observations.

b) it must produce a reasonable value of grain boundary energy and the orientation and temperature dependence thereof.

c) it must account for the change in orientation between the two grains while still taking into

account individual atom interactions.

d) it must be able to explain, to at least some degree, a host of properties such as boundary sliding, migration, segregation, corrosion, melting and low temperature mechanical behaviour.

This last requirement is the most difficult to fulfill. The main difficulty to date appears to be that a given model may elegantly account for the behaviour of one or two boundary properties, yet break down completely when the others are considered. For this reason, plus the fact that some models have not yet been evaluated with respect to even the first three criteria, it is impossible to rigorously assess their shortcomings and merits. Thus they will be presented only briefly, with perhaps some indication of current popularity, in order to establish a physical basis for the consideration of dislocation generation from grain boundaries.

One problem endemic to most models is that they were originally constructed to explain the structure of simple "special" boundaries, such as low angle pure tilt or twist boundaries or coincidence boundaries. Ultimately, however, they should be able to give at least an approximate picture of that most common of boundaries in normal materials, the random, high angle (high $\theta$) grain boundary.

### 2.2.1 AMORPHOUS MODEL

This model, developed by Rosenhain and co-workers (5, 6) was the first real attempt to account for boundary properties. In essence, it considered the boundary as an amorphous, undercooled liquid, i.e. possessing no long range periodic structure. Although this could qualitatively explain such phenomena as grain boundary sliding and brittleness at high and low temperatures, respectively, it has been generally discounted for a number of reasons (1). A calculation of the excess internal energy based on this model leads to calculated values of boundary thickness much larger than observed experimentally. Also, it seems inherently unreasonable that so thin a region would not be influenced at all by the periodic crystal structures on either side of it. Most important, such a model completely fails to account for the variation in many boundary properties with both orientation and/or inclination.

The concept on which the model is based has some value with regard to such matters as calculation of grain boundary energy. Also, as McLean comments (7), the boundary layer bears some similarity to an amorphous layer in that both are regions in which the perfect crystal structure is forbidden, thus tempering somewhat the comparison of this region to a special lattice. Aaron and Bolling (8)

have considered grain boundary energy using somewhat of an amorphous model, that is, the boundary structure with the lowest free volume possesses the lowest energy boundary. They conclude that high angle boundaries are most likely constructed according to a "structureless" model built on the random close-packing of atoms. As we shall soon see, however, there is a good chance that this could correspond to a structured model which allows individual atom relaxation into the lowest energy configuration.

### 2.2.2 DISLOCATION MODELS

These models consider grain boundaries as planar arrays of dislocations. It is well established that low angle boundaries (9, 10), i.e. low $\theta$ values, are composed of arrays of lattice edge dislocations for a pure tilt misorientation, screw dislocations for a pure twist misorientation, or, as is most often the case, dislocations of mixed character for a mixed orientation. This is illustrated in figure 2, with the twist segment on top, the tilt segments on the sides and a mixed segment denoted DCH.

The spacing of these lattice dislocations decreases with increasing misorientation according to $d = \underline{b}/\theta$ (b = Burgers vector, d = spacing), hence the boundary

Figure 2. Illustrating symmetric tilt, twist and mixed low angle grain boundaries for the case of one grain entirely surrounded by another grain, with both sharing a common <001> axis (Bishop and Chalmers, 35).

Figure 3. (a) Dislocation model of a symmetric $53^{o}$ tilt boundary. (b) Dislocation model of a symmetric $60^{o}$ tilt boundary (Gleiter, 1).