

## Investigation of Grain Boundary Migration in Al-Bicrystals by Continuous Tracking with X-Rays

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**Abstract.** The measurement of grain boundary motion [in pure Al bicrystals] by X-ray continuous interface tracking is introduced. The method provides information on the grain boundary mobility without interfering with the process of migration itself. The dependence of grain boundary motion on orientation and materials purity was addressed. It was found that the misorientation affects both the activation enthalpy of grain boundary motion and preexponential mobility factor. This causes the exact  $\Sigma 7$  boundary to move fastest at low temperatures, however, the  $40.5^\circ \langle 111 \rangle$  boundary is most mobile at high temperatures. Impurities were found to reduce the grain boundary mobility by rising the migration activation enthalpy, while the preexponential factor depends only mildly on impurity content.

### 1. Introduction

The migration of grain boundaries is the dominating process of microstructure and texture evolution during recrystallisation and grain growth. Despite long standing interest and research efforts in this process, there is substantial lack of reliable data on grain boundary migration. The main reason for this deficiency is the difficulty to experimentally determine grain boundary mobility.

There is evidence that grain boundaries properties depend on grain boundary structure, i. e. on its crystallography and chemical composition. Experiments on polycrystals can only yield average properties of the polycrystalline assembly. The structural dependence of boundary mobility can only be retrieved from investigations of defined grain boundaries in material with controlled purity. The current study will focus on the orientation dependence of grain boundary mobility in bicrystals of differently pure aluminium.

### 2. Experimental

The motion of  $\langle 111 \rangle$  tilt boundaries with misorientation angles in the vicinity of the special misorientation  $\Sigma 7$  ( $\varphi = 38.2^\circ$ ) was studied. The experiments were carried out on bicrystals produced from high purity aluminium of different origin with total impurity content of 0.4 - 7.7 ppm. The impurity concentration in used materials was determined by glow discharge mass-spectrometry. The total impurity content in each material was defined as the sum of the concentration of all found elements.

For measurements of grain boundary mobility, the motion of grain boundaries under the action of a costant driving force was recorded (Fig. 1). The driving force was provided by the surface tension of a curved grain boundary:  $p = \sigma/a$ , where  $\sigma$  is the grain boundary surface tension, and  $a$  the width

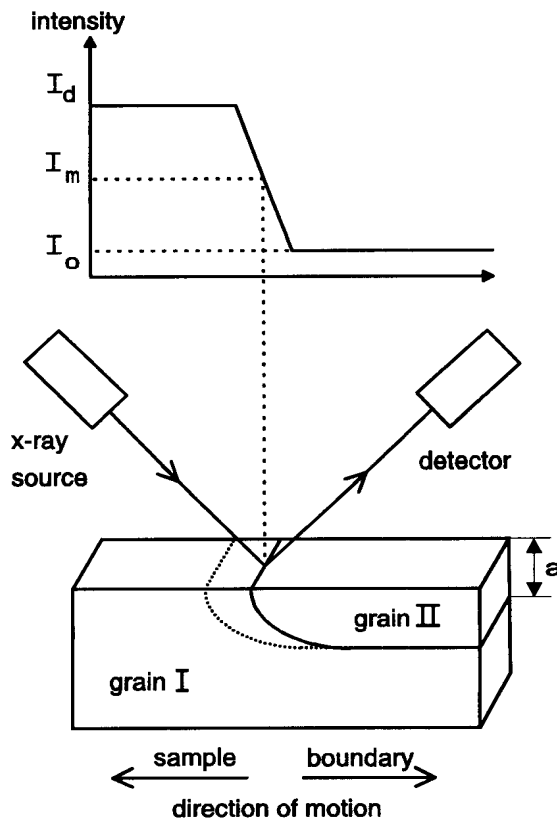


Fig.1: Geometry of used bicrystals and measuring principle of the XICTD [3].

of the shrinking grain. Details of bicrystal growth and sample preparation are given elsewhere [1, 2]. The velocity of grain boundary motion was measured using a specially designed X-ray device for continuous tracking of moving grain boundary (XICTD) with an accuracy not worse than 2% [3]. The method of operation is illustrated in Fig. 1. A sample, which contains two differently oriented crystals is mounted such that under the incident X-ray beam one crystal is in Bragg-position while the other is not. A scan of the X-ray beam along the sample surface would give the intensity distribution as shown in Fig. 1. The maximum intensity  $I_d$  is recorded as long as the X-ray spot is entirely located on the surface of crystal 1. When the X-ray spot has completely moved over to crystal II the detected intensity attains the value  $I_0$ . The boundary position can be associated with the position where the intermediate intensity  $I_m$  is detected. When the boundary moves, the sample is concurrently shifted such that the reflected X-ray intensity remains constant during the measurement. Thus, the velocity of the moving grain boundary is equal to the speed of sample movement at any moment during the experiment.

### 3. Results

#### 3.1. Orientation Dependence of Grain Boundary Migration

During the experiment the boundary displacement is recorded. Its derivative with regard to time is the velocity  $v$  of grain boundary motion, which is related to the driving force  $p$  by the boundary mobility  $m = v / p$ . For convenience we use the reduced boundary mobility

$$A \equiv v \cdot a = A_0 \exp\left(-\frac{H}{kT}\right) = m\sigma, \quad (1)$$

where  $H$  is the activation enthalpy of migration and  $A_0$  the pre-exponential mobility factor.

The orientation dependence of boundary migration parameters reveal that both the activation enthalpy and the preexponential factor are at maximum for misorientation angle  $\varphi=40.5^\circ$  and at minimum for the exact  $\Sigma 7$  orientation (Fig. 2). The temperature dependence of boundary mobility varies with misorientation, and there is a temperature, the so-called compensation temperature  $T_c$ , where the mobilities of the boundaries of differently misoriented grains are the same. As a result,

for  $T > T_c$ , the mobility is higher for grain boundaries with higher activation energy, in particular it is at maximum for  $\phi = 40.5^\circ$ , while for  $T < T_c$  the exact  $\Sigma 7$  boundary moves fastest (Fig. 2). This result easily explains the finding of the growth selection experiments of Lücke *et al.* [4], which clearly yielded a maximum growth rate at the rotation angle of slightly more than  $40^\circ$ , while the measurements on bicrystals reveal the  $\Sigma 7$  boundary as the fastest [5]. Growth selection experiments were conducted at very high temperatures (above  $600^\circ\text{C}$ ), i. e. in the temperature regime, where, according to results of the current study, the mobility of the  $40.5^\circ <111>$  boundary is the highest. The reason for the different maximum mobility orientation in different temperature regimes is obviously the orientation dependence of both, the activation enthalpy and the preexponential factor, and the linear relationship between  $H$  and  $\log A_0$  - the compensation effect (Fig. 3).

### 3.2. Effect of impurity on grain boundary mobility

There is evidence that grain boundary motion is slowed down by impurity atoms. But very little is known how impurities affect the activation enthalpy of grain boundary migration and the preexponential mobility factor and how much this effect depends on the grain boundary crystallography. As expected, in our experiments impurities were found to reduce grain boundary mobility (Fig. 4). But in contrast to the impurity drag theory [6, 7] the mobility does not decrease due to a reduction of the preexponential factor, but due to a considerable rise of the activation enthalpy (Fig. 4). The preexponential factor was found to be only mildly dependent on the impurity concentration.

As a result, the migration activation enthalpy is strongly affected by both, the boundary crystallography and material purity. However in the former case with increasing  $H$  the preexponential factor  $A_0$

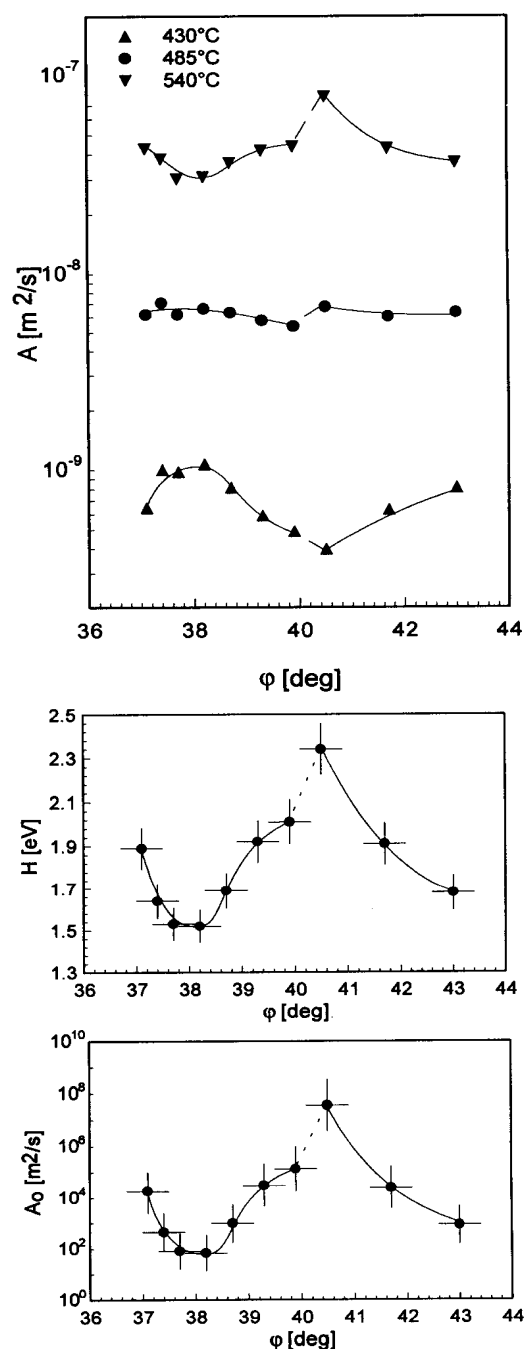


Fig. 2: Mobility  $A$ , activation enthalpy  $H$  and preexponential factor  $A_0$  for  $<111>$  tilt boundaries in pure Al with 1.0 ppm total impurity content.

risers by several orders of magnitude, while in the latter case  $A_o$  remains at the same level. Therefore, the preexponential factor  $A_o$  in the investigated impurity concentration interval was found to be much less sensitive to the material purity than to a change of the boundary crystallography. This result allows to conclude that the observed orientation dependence of mobility (Fig. 2), determined by both  $H$  and  $A_o$ , does not only reflect the different segregation behavior of coincidence and random boundaries, as frequently proposed [8], rather it provides evidence for an intrinsic dependence of grain boundary mobility on grain boundary structure.

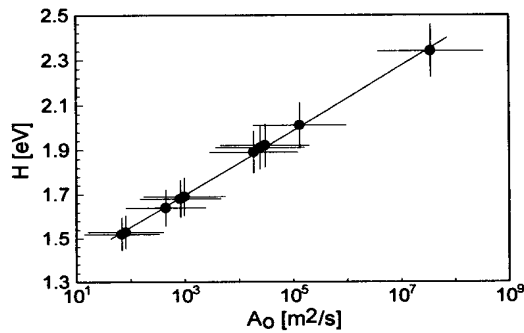


Fig. 3: Dependence of migration activation enthalpy on preexponential mobility factor.

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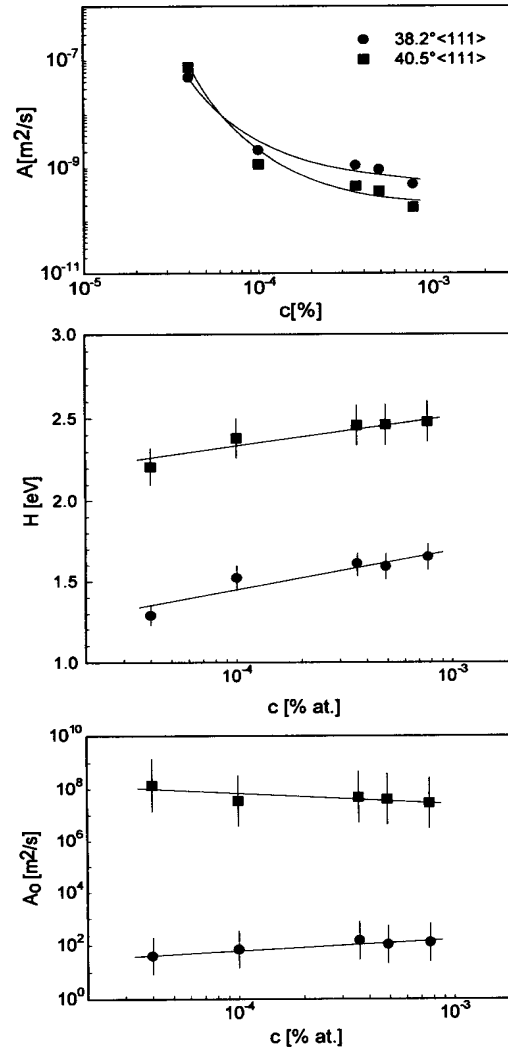


Fig. 4: Dependence of grain boundary mobility  $A$ , activation enthalpy  $H$  and preexponential mobility factor  $A_o$  on total impurity content in pure Aluminium.