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X-RAY METHOD FOR CONTINUOUS TRACKING OF GRAIN BOUNDARY MOTION: INVESTIGATION OF GRAIN BOUNDARY MIGRATION IN Al-BICRYSTALS

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ABSTRACT

A method which enables continuous tracking of the migration of boundaries will be represented. It utilizes X-ray diffraction rather than optical methods to determine a grain boundary position and allows to define the velocity of GBM at any moment of experiment.
In the second part of the paper two example are presented to illustrate the capabilities of this method.

INTRODUCTION

Grain boundary (GB) migration strongly influences technologically important processes such like recrystallisation and grain growth and controls the evolution of texture. Despite long standing interest and research efforts on this process, there is a substantial lack of reliable data on grain boundary migration (GBM). This deficiency is mainly due to inappropriate experimental methods and the underrating of drag effects. The main disadvantage of the previously used experimental methods was the interruption of GBM to measure the GB position. Correspondingly, those methods provided only an average velocity of the GB and not the true velocity at every moment during the experiment. This has gravitated in a profound misunderstanding of the nature of GB motion in the past [1].
The experimental method introduced in this paper allows to measure the position and velocity of the GB at any time of the experiment, i.e. enables to measure the GBM in a steady state motion.

EXPERIMENTAL

The Sample

The experiments were carried out on bicrystals with well defined angle of misorientation φ and rotation axis <hkl> between the two grains. The motion of the boundaries were investigated under a constant driving force $\Delta F \sim a^{-1}$ and with the reduced mobility A:

$$A = v \cdot a \tag{1}$$

$$A = A_0 \cdot e^{-\frac{H}{kT}} \tag{2}$$

v: velocity of GBM
A_0: pre exponential factor
H: enthalpy of activation of GBM

Figure 1 shows the bicrystal and the shape of the boundary. The curved part of the GB moves along the axis of the sample parallel to itself. The total area of the boundary decreases and this provides the driving force for GBM. The shape of the moving boundary and, consequently, the orientation of the boundary with respect to the crystallographic axis remained constant during motion.

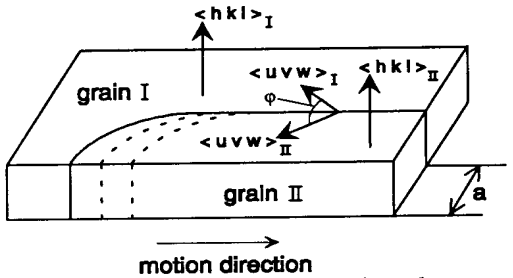

Figure 1: Bicrystal with a tilt grain boundary: Tilt axis <hkl> and angle of misorientation φ. The grain boundary at later moments is symbolized by the dashed lines.

Method of measurement

The basis of the method of continuous tracking of a moving boundary is illustrated in figure 2. The method employ X-ray diffraction to determine the GB motion. Before the measurement the bicrystal is rotated in a goniometer such that one grain is in Bragg-position while the other is not. Then the bicrystal will be displaced until the X-ray spot is located on the grain boundary, which will be identified by an intermediate intensity I_m recorded by a detector. When the boundary moves, the sample is concurrently shifted such that the reflected X-ray intensity remains constant during the measurement. So, the GBM velocity at any time is equal to speed of sample movement at this moment.

Figure 3 shows a schematic sketch of the device. The computer records the X-ray intensity from the detector and moves the sample with a longitudinal steping motor such that the recorded intensity remains constant. To account for the thermal expansion of the sample, the Bragg-angle is continuously corrected during the heating. The resolution of the longitudinal displacment by the steping motor is 5μm and the size of the X-ray spot is 0.37mm² in Bragg position. During the GB motion experiment the temperature is kept constant within ±0.3°C. To avoid thermal grooving, the sample and the hot stage are exposed to a nitrogen atmosphere.

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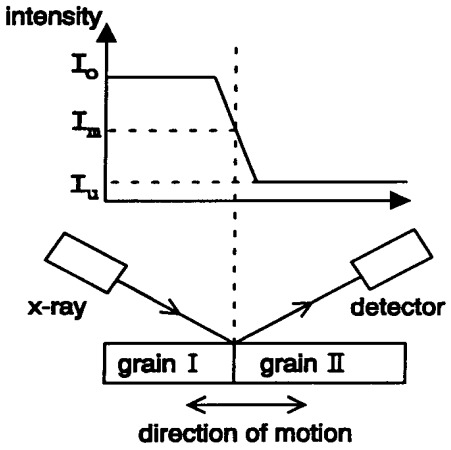

Figure 2: The scheme of the method of measurement.
The X-ray spot is located on the GB such that a half of the spot is on grain I while the other is on grain II.

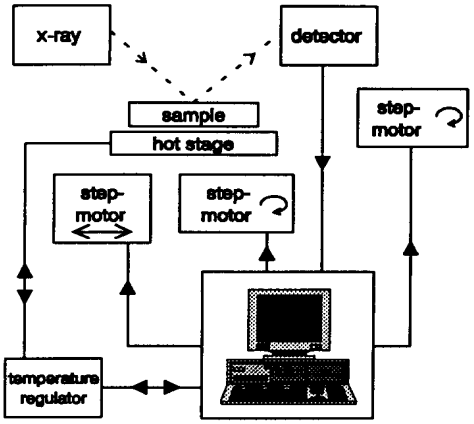

Figure 3: Block scheme of the goniometer. The computer records the detected intensity and shifts the sample by the longitudinal steping motor. During the heating the computer controls the temperature by a temperature regulator and corrects the Bragg angle by steping motors.

First Results

In this chapter two examples will be shown to illustrate the possible application of the device.

Kinetics of Phase Transition of GBM

The detachment of a moving GB from an impurity cloud is an interesting and important physical phenomenon. Measurement of this effect permits us to determine the adsorption properties of the GB: the energy of interaction between the GB and impurity atoms, the number of adsorption sites, the activation energy for the diffusion of impurity atoms together with moving GB [2].

Figure 4 shows the temperature dependence of the mobility of tilt GB, φ=46.8° in iron-doped Al bicrystals. In contrast to the undoped sample (I) the iron doped samples (II-IV) exhibits the mobility of discontinuities at two different temperatures which results in a drastic alteration of the migration activation energy. An analysis of the data has shown that the observed discontinuous changes of GBM are related to the break-away of the migrating GB from adsorbed iron atoms, and the sequence of the break-away can be attributed to the existence of two types of adsorption centres in low Σ grain boundaries.

The and measurement of this effect needs a very sensitive technique, which enables to observe the moving GB displacement with a high resolution. The method of GB X-ray tracking meets this requirement.

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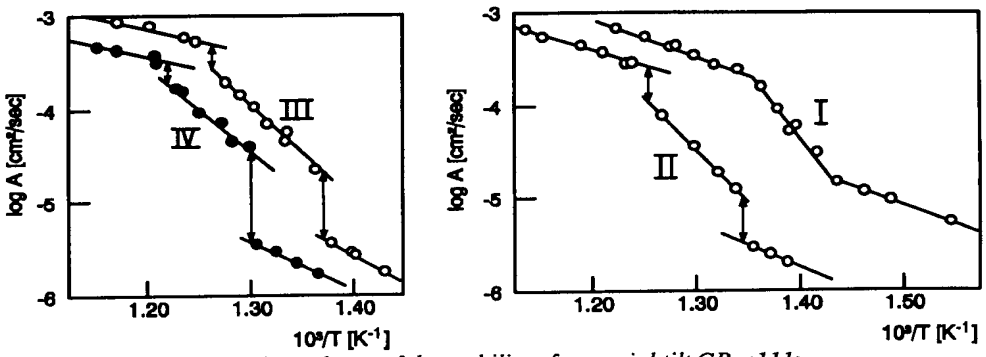

Figure 4: Temperature dependence of the mobility of a special tilt GB <111> (φ =46.5° ± 0.5°) in iron-doped aluminium samples. I denotes the undoped aluminium and II-IV identify the doped material with increasing content of iron.

Orientation dependence of GBM

The second example shows the dependence of the activation energy for the mobility of <100> tilt grain boundaries on misorientation in Al with different content of impurities (figure 5). For small angle boundaries the activation energy decreases with increasing misorientation. For high angle boundaries (φ≥20°) the activation energy depend strongly on impurity level. For both ultra pure (99.99995%) and low purity (99.98%) material, the activation energy does not depend on angle of rotation, but the absolute value of the activation energy is by a factor of three higher for the impure material. For less than ultra pure material (99.9992%, referred to as high purity material), the activation energy oscillates and attains minima for special angles of rotation, which correspond to low Σ CSL rotations. For these special misorientations the activation energy is practically identical for high purity and ultra pure material. Thus, experimental results on single GBs qualitatively confirm the findings on polycrystals [3-5] that in high purity materials special boundaries have a higher mobility than random boundaries.

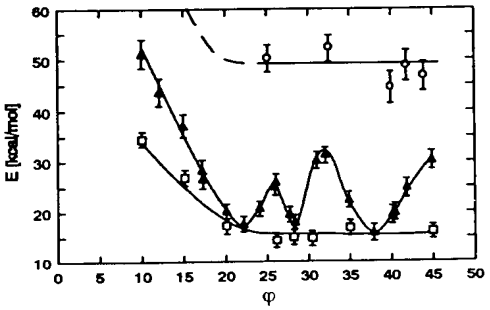

Figure 5: The dependence of the activation energy of migration for <100> tilt grain boundaries in Al of different purity □-99.99995 at.%; ▲-99.9992 at.%; ○-99.98 at%.

ACKNOWLEDGEMENT

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