Materials Characterization 117 (2016) 99–112

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Determination of grain boundary mobility during recrystallization by statistical evaluation of electron backscatter diffraction measurements

I. Basu *, M. Chen, M. Loeck, T. Al-Samman, D.A. Molodov

Institute of Physical Metallurgy and Metal Physics, RWTH Aachen University, 52056 Aachen, Germany

ARTICLE INFO

Article history:
Received 13 January 2016
Received in revised form 19 April 2016
Accepted 30 April 2016
Available online 3 May 2016

Keywords:
Grain boundary mobility
Recrystallization
Aluminium
Electron back scattered diffraction (EBSD)
Growth anisotropy

ABSTRACT

One of the key aspects influencing microstructural design pathways in metallic systems is grain boundary motion. The present work introduces a method by means of which direct measurement of grain boundary mobility vs. misorientation dependence is made possible. The technique utilizes datasets acquired by means of serial electron backscatter diffraction (EBSD) measurements. The experimental EBSD measurements are collectively analyzed, whereby datasets were used to obtain grain boundary mobility and grain aspect ratio with respect to grain boundary misorientation. The proposed method is further validated using cellular automata (CA) simulations. Single crystal aluminium was cold rolled and scratched in order to nucleate random orientations. Subsequent annealing at 300 °C resulted in grains growing, in the direction normal to the scratch, into a single deformed orientation. Growth selection was observed, wherein the boundaries with misorientations close to Σ7 CSL orientation relationship (38° ⟨111⟩) migrated considerably faster. The obtained boundary mobility distribution exhibited a non-monotonic behavior with a maximum corresponding to misorientation of 38° ± 2° about ⟨111⟩ axes ± 4°, which was 10–100 times higher than the mobility values of random high angle boundaries. Correlation with the grain aspect ratio values indicated a strong growth anisotropy displayed by the fast growing grains. The observations have been discussed in terms of the influence of grain boundary character on grain boundary motion during recrystallization.

1. Introduction

The exclusive property of grain boundaries, i.e. interfaces between crystallites of the same phase with different crystallographic orientations, is their ability to move in response to applied forces. Grain boundary motion, by which the crystalline solid is rearranged atom by atom, is the fundamental process of grain microstructure evolution in the course of recrystallization and grain growth during annealing subsequent to plastic deformation. The motion of grain boundaries is controlled by their mobility m and the applied driving force p. As was predicted theoretically [1,2] and corroborated by many experiments with different driving forces [3–9], the boundary migration rate v is proportional to the acting driving force, given as,

$$v = mp \tag{1}$$

Grain boundary motion is a thermally activated process and the boundary mobility thus shows an Arrhenius temperature dependence, i.e.

$$m = m_0 \exp\left(-\frac{H}{kT}\right) \tag{2}$$

where H is the activation enthalpy of grain boundary migration.

It is well-established that grain boundary mobility is to a great extent determined by the grain boundary character, which is commonly reduced to the orientation relationship between adjacent grains and the orientation of the grain boundary plane. The orientation dependence of grain boundary mobility was first evidenced by developing distinctive crystallographic textures during annealing of deformed polycrystals. The classical growth selection experiments in deformed Al single crystal by Lücke with co-workers [10–14] revealed that the grains with the fastest growth rate were found to be misoriented relative to the deformed matrix by a rotation angle of slightly >40° around an axis close to ⟨111⟩.

The well pronounced non-monotonic dependences of the grain boundary migration rate and the migration activation energy on the misorientation angle were also observed in experiments on bicrystals of various metals [9,15–26]. The higher velocity values and the lower activation energies were associated with the boundaries corresponding to the low Σ Coincidence Site Lattice (CSL) orientation relationships. Measurements of grain boundary motion in Al bicrystals [19,25] have shown that tilt grain boundaries with ⟨111⟩ rotation axis and rotation angle of about 40° have the highest mobility. According to the understanding that grain boundaries with highly periodic coincidence structure (low Σ CSL or special boundaries) move faster than off-coincidence (random) boundaries and due to the close vicinity of 40° ⟨111⟩ misorientation to the Σ7 CSL (Coincidence Site Lattice) orientation relationship, the Σ7

* Corresponding author.
E-mail address: basu@imm.rwth-aachen.de (I. Basu).

http://dx.doi.org/10.1016/j.matchar.2016.04.024
1044-5803/

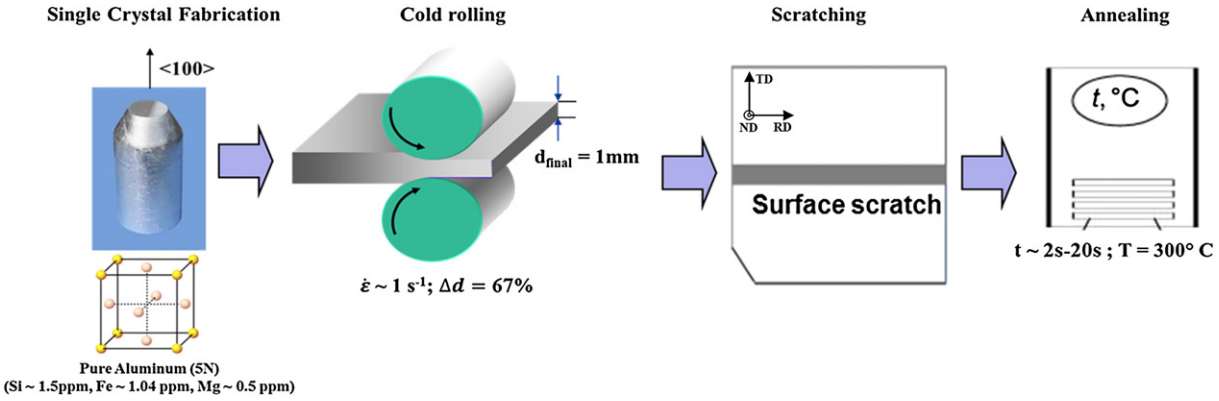

Fig. 1. Schematic illustration of the experimental procedure used for the recrystallization study.

(38.2° ⟨111⟩) tilt boundary was identified as the most mobile boundary in Al [27,28]. However, as was pointed by Lücke [14], the overwhelming statistics of growth selection experiments substantiated that the rotation angles for the fastest moving ⟨111⟩ boundaries were even larger than 40°: "The orientation for the maximum growth rate is definitely not the 38°<111> rotation", "… the angle of rotation lies between 41° and 42°, where practically no coincidences exist" [14].

Later investigations on the dependence of the mobility of ⟨111⟩ tilt boundaries in Al bicrystals upon angular misorientation values confined between 37° and 43° [29–31], revealed that the temperature dependence of boundary mobility is different for the boundaries with different misorientation angles, and there is a temperature, referred to as the compensation temperature *Tc*, where the mobility values of different boundaries were similar. Both the activation energy and the pre-exponential factor were obtained to be maximum for a misorientation angle of 40.5° and minimum for the exact Σ7 orientation (38.2°). Correspondingly, at temperatures lower than *Tc*, which is about 450 °C in high purity Al [29–31], the mobility was higher for grain boundaries with lower activation energy, in particular it was at maximum for the exact Σ7 boundary, whereas in the high temperature range the maximum misorientation shifted to 40.5° [29–31]. These results directly explained why growth selection experiments [10–13], which were performed at temperatures above 600 °C, distinctly identified a 40° ⟨111⟩ boundary as having the highest mobility.

For modeling the microstructure and texture evolution during recrystallization, it is indispensable to determine the migration rate of the moving boundaries of the new recrystallized grains/nuclei, by using Eq. (1) for example. Therefore, knowledge of boundary mobility and estimating its orientation dependence are decisive in accurately modeling recrystallization. It can also be important to extract boundary mobility data from recrystallization experiments because the mobility of stored-energy-driven boundaries can be expected to differ from that of curvature-driven boundaries [7].

For the measurements of grain boundary mobility during recrystallization, an experimental procedure was utilized, wherein grains are allowed to grow from a scratch into a single deformed orientation. The scratch enables nucleation of random orientations thus negating oriented nucleation effects. Huang and Humphreys [7,32,33] utilized this approach combined with in situ annealing in a scanning electron microscope [34] to measure grain boundary mobility during recrystallization of a single-phase Al-0.05 wt.% Si alloy. The obtained maximum mobility values corresponded to boundaries with misorientation angles in the range between 35° and 45° about axes within ± 10° of ⟨111⟩. The difference between this broad peak and the sharp peaks found in experiments with curvature-driven boundaries was attributed to local porosity or excess volume associated with interaction of migrating grain boundaries with point defects and dislocations [7]. A similar investigation on single crystal Al-Zr alloy, conducted by Taheri et al. [35] confirmed that ⟨111⟩ high angle boundaries in Al are the most mobile in general. In good agreement with the measurements of boundary mobility, where curvature was utilized as a driving force [29–31], a minimum in migration activation energy and maximum mobility at low temperatures were observed for the 38° ⟨111⟩ boundary. Also, as it was found in experiments on bicrystals, at high temperatures the mobility of this boundary becomes a local minimum [35].

In previous investigations [7,32,33,35] grain boundary mobility measurements during recrystallization have primarily employed in-situ annealing procedure. Even though in-situ measurements provide the necessary spatial and temporal evolution of the microstructure, the obtained datasets lack statistical sufficiency due to the associated experimental difficulties. However, considering that both nucleation and growth of new grains are essentially stochastic processes on a large scale, i.e. depend more upon the local environment than the global, statistical corroboration of such measurements becomes imperative. The present study hence proposes a new approach, whereby the spatial and temporal effects on determining the boundary mobility are compensated by measuring substantially larger data sets, which allow obtaining statistically more reliable mobility values.

In order to test the validity and efficiency of the aforementioned method under practical considerations, aluminium, as a model material, was employed in the current study due to sufficiently available literature data on grain boundary mobility and its misorientation dependence in face-centered-cubic (fcc) metals. Future investigations would aim at application of this method upon materials with low symmetry crystal structures, such as hexagonal-close-packed (hcp) metals, whereby statistically more accurate and reliable grain boundary mobility data can be generated. Such information is essential in order to design optimized processing techniques resulting in desirable textures and microstructures.

2. Material and experiments

Conically shaped high purity aluminium (99.999%) single crystals with two different orientations were fabricated using vertical Bridgman technique. Orientation determination by means of standard Laue technique revealed respective initial orientations given by, (i) [311] direction deviating by ~5° from sheet normal (ND) and [152] aligned with rolling direction (RD) denoted as 'Type-I'; and (ii) [111] direction deviating from the ND by about 7°, with [110] along RD labeled as 'Type-II'. Monocrystalline specimens of 65 mm length, 25 mm width and 3 mm thickness were machined from the grown single crystals by electro-discharge machining. These specimens were then subjected to multi-pass cold rolling treatment at different strain values, such that the specimens conforming to 'Type-I' orientation were rolled to a final thickness