PERGAMON

Acta mater. 48 (2000) 2017–2030

Acta MATERIALIA

www.elsevier.com/locate/actamat

SUBGRAIN GROWTH AND LOW ANGLE BOUNDARY MOBILITY IN ALUMINIUM CRYSTALS OF ORIENTATION {110}⟨001⟩

Y. HUANG and F. J. HUMPHREYS†

Manchester Materials Science Centre, Grosvenor Street, Manchester M1 7HS, UK

(Received 17 September 1999; accepted 7 November 1999)

Abstract—Single crystals of orientation {110}⟨001⟩ of a high purity Al–0.05% Si single-phase aluminium alloy have been deformed in channel die plane strain compression at room temperature and 350°C. The specimens were annealed at temperatures between 250 and 400°C and detailed measurements have been made of the extensive subgrain growth which occurs in these crystals. It was found that subgrain growth tended to be discontinuous, confirming earlier experimental and theoretical work, and showing that subgrain growth is quite different from normal grain growth. The mean misorientation between subgrains decreased during annealing and this was shown to have a strong effect on the kinetics of subgrain growth. The mobilities of low angle boundaries ($2.6^\circ < \theta < 5.6^\circ$) at temperatures between 250 and 400°C were determined from the subgrain growth kinetics and the activation energies for migration found to be consistent with control by lattice diffusion of solute. The boundary mobilities were found to increase rapidly with increasing misorientation and the results have been compared with the predictions of current theories.

Keywords: Electron diffraction; Scanning electron microscopy; Aluminum; Recrystallization & Recovery; Subgrain growth

1. INTRODUCTION

The recovery processes which occur during the early stages of the annealing of deformed metals are probably not as well understood as the subsequent processes of recrystallization and grain growth [1, 2]. In alloys with a high stacking fault energy and low solute content, such as dilute aluminium alloys, the microstructures after deformation at ambient or elevated temperatures can usually be described as a cellular or subgrain structure (e.g. Refs [3, 4]). On annealing, the main recovery process is then subgrain growth, which occurs by the migration of low angle grain boundaries (see, e.g. Ref. [4]). Subgrain growth in regions of the microstructure where there are large orientation gradients may also lead to the initiation of recrystallization.

Our current understanding of subgrain growth and knowledge of the early stages of recrystallization and our ability to model these processes are primarily limited by a lack of information about the mobility of low angle grain boundaries [4, 5]. Although there have been several recent investigations of the migration and mobility of high angle boundaries during grain growth [6] or recrystallization [7], it has proved difficult to investigate the movement of low angle boundaries during recovery, because there is often little low angle boundary migration before the recovered microstructure is obliterated by recrystallization. Recrystallization may be suppressed if there are no prior grain boundaries and if the deformation is such as to introduce no large orientation gradients, and Ferry and Humphreys [8] showed that single crystals of the {110}⟨001⟩ or Goss orientation, deformed in plane strain compression, could undergo extensive subgrain growth on subsequent annealing. They also showed that the annealing of such crystals was capable of yielding some information about low angle boundary mobility. The present investigation that is essentially an extension of this earlier work, was carried out specifically to study low angle grain boundary migration and the effects of temperature and misorientation on boundary mobility in dilute aluminium alloys. The availability of automated EBSD methods which were not developed at the time of the earlier investigation, means that it is now possible to characterize subgrain orientations and misorientations much more accurately and extensively than was then possible.

† To whom all correspondence should be addressed.

1359-6454/00/$20.00
PII: S1359-6454(99)00418-8

2. MATERIALS AND METHOD

2.1. Material

The material used in the investigation was a high-purity Al–0.05% Si alloy (total impurity level 0.001 wt%) supplied as a DC cast billet by Alcan International, Banbury, U.K.. The small amount of silicon, which is close to the limit of solid solubility at ~300°C, served to pin dislocations and slightly solution harden the material, thus making the crystals easier to handle than for high purity aluminium. Similar material has been used extensively for other investigations (e.g. Refs [7, 8]) and because silicon does not readily precipitate in aluminium, no precipitation has been observed in this alloy during the present or previous investigations during annealing at temperatures down to 250°C.

2.2. Single crystal growth

Cylindrical single crystals of diameter 16 mm were grown from the melt in graphite moulds under a vacuum of 10^{-5} Torr from seed crystals of ⟨100⟩ orientation. The orientations of the crystals were checked by Laué back-reflection X-ray diffraction and the crystals were oriented and carefully machined, ground and polished to produce rectangular crystals. The orientations of the specimens were checked with X-ray diffraction and found to be within 1–2° of the required orientation. The crystals were heat treated for 4 h at 580°C, furnace cooled at 10°C/h to 300°C and then water quenched.

2.3. Deformation and annealing

The crystals, of approximate dimensions 12 mm (ND) × 15 mm (RD) × 10 mm (TD), were surrounded with thin PTFE sheet to provide lubrication and placed in a steel channel die as in the earlier investigation [8]. The die was placed between parallel platens and the specimens deformed at a nominal strain rate of 10^{-3}/s in an Instron testing machine. During testing, the PTFE lubricant was replaced at increments of ~20% reduction to reduce frictional constraint between the specimens and the compression jig. To maintain consistency with terminology for rolled materials, the directions with respect to the die are referred to as ND, TD and RD. Compressive reductions of 30 and 70% were made at room temperature, and samples were also deformed 70% at 350°C. This provided three different starting microstructures for annealing and these will subsequently be referred to as 30C, 70C and 70H, respectively. Slices at least 1 mm thick parallel to both ND and TD planes were cut from the as-deformed crystals. The surfaces of these slices were then mechanically ground and polished, and all specimens were subsequently deeply etched in Kellers reagent to produce strain-free surfaces in order to minimize the likelihood of surface nucleation of recrystallized grains during subsequent annealing. Recrystallization was deliberately induced in some specimens by lightly abrading the surfaces with #1200 SiC paper after deformation. Specimens were subsequently annealed at temperatures ranging from 250 to 400°C either in a salt bath or an air furnace.

2.4. Microstructural characterization

As similar crystals had been previously characterized by optical and transmission electron microscopy and X-ray texture methods [8], these techniques were not used in the present work, and samples were only investigated by scanning electron microscopy (SEM). The annealed crystal slices were mechanically polished to remove any surface-induced annealing effects and then electropolished to produce a strain-free surface. The specimens were examined in either a JEOL 6300 W-filament SEM or a Philips XL30 field emission gun SEM using backscattered electrons at 8–10 keV to reveal crystallographic contrast. Subgrain sizes (which throughout this paper are quoted as mean linear intercepts) and size distributions were measured from these images.

Both scanning electron microscopes were equipped with identical facilities for electron backscatter diffraction (EBSD). The EBSD patterns were detected by a Nordif CCD camera, and background subtraction and frame averaging were carried out using a Hamamatsu Argus 20 image processor. Microscope control, pattern acquisition and solution were carried out with the HKL Channel system. The diffraction data were usually acquired as orientation maps from raster scans ~100 × 75 μm with step sizes of 0.2–1 μm and an acquisition time of ~0.2 s/pattern. The resolution for electron channelling contrast images in the FEGSEM is some ten times better than for the W-filament microscope and the EBSD spatial resolutions of the FEGSEM is some four times better [9]. With the FEGSEM it was therefore possible to quantitatively characterize subgrains of sizes down to ~0.2 μm [9]. The EBSD data were analysed using VMAP, an in-house software development, to obtain subgrain misorientations and misorientation distributions. Subgrains may be measured from EBSD maps in two ways [10]. The simplest is to carry out linear intercepts of the data from which size and misorientation information may be obtained. An alternative procedure is to reconstruct the subgrains from the data in a manner similar to that employed in image analysis. Because each subgrain contains several data points, more accurate orientations may be obtained by this method, and individual misorien-