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In situ observation of triple junction motion during recovery of heavily deformed aluminum

Tianbo Yu,* Darcy A. Hughes,[1] Niels Hansen and Xiaoxu Huang

Danish–Chinese Center for Nanometals, Section for Materials Science and Advanced Characterization, Department of Wind Energy, Risø Campus, Technical University of Denmark, DK-4000 Roskilde, Denmark

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Abstract—Microstructural evolution during in situ annealing of heavily cold-rolled aluminum has been studied by transmission electron microscopy, confirming that an important recovery mechanism is migration of triple junctions formed by three lamellar boundaries (Y-junctions). The migrating Y-junctions are pinned by deformation-induced interconnecting and lamellar boundaries, which slow down the recovery process and lead to a stop–go migration pattern. This pinning mechanism stabilizes the deformation microstructure, i.e. the structure is stabilized by balancing the driving and pinning forces controlling the rate of triple junction motion. As a consequence, recovery and the subsequent recrystallization are strongly retarded. The mechanisms underlying Y-junction motion and its pinning are analyzed and discussed.

Keywords: Aluminum; Annealing; Deformation structure; Transmission electron microscopy (TEM); Triple junction

1. Introduction

Strengthening of metals by plastic deformation is of universal interest, with the current focus being on deformation to high strains to produce materials with ultrafine structures and high strengths. This strength has its origin in a high concentration of structural defects exemplified by narrowly spaced low- and high-angle deformation-induced boundaries [1–4]. These boundaries together with other defects raise the stored energy and the driving pressure for boundary migration, reducing the thermal stability of the strong materials [5–8]. For example, high-purity Al (⩾99.99%) deformed by torsion to various strains in liquid nitrogen was reported to recover and recrystallize below room temperature [9,10], and high-purity Cu (99.96%) deformed by equal-channel angular extrusion to a high strain was reported to be partially recrystallized after long-term storage at room temperature [11]. It has also been found, however, that relatively high thermal stability can be achieved with the addition of some impurities. For example, only a slight decrease in hardness was observed when commercial-purity Al (99.2%) was annealed at 100 °C after deformation by accumulative roll bonding to a large strain [8], and heavily cold-rolled Al (99.5%) remained at the onset of recrystallization after annealing for 10 min at 300 °C (~0.6 in terms of homologous temperature) [12]. Such a stabilization may have its cause in the retardation of boundary migration by solute drag or particle pinning [13–16]. Other stabilizing methods may be found and utilized, in addition to stabilizing by solutes and particles, by examining the fundamental recovery and pinning mechanisms inherent in the structure of heavily deformed metals. Pinning interactions may depend on the properties of the deformation-induced boundaries, but may also depend on their morphology and their surrounding structures. For example, it has been observed that migration of one type of triple junction, the Y-junction, is an important recovery mechanism in heavily deformed Al [17,18], leading to removal of boundaries and coarsening of a lamellar microstructure prior to recrystallization.

The migration of a triple junction in a heavily deformed structure adds to such well-known phenomena as triple junction motion during recovery [19] and creep [20] of metals which are slightly deformed. In the latter two cases the interacting boundaries are of very low angle, about a few degrees, whereas the observed motion of Y-junctions [17,18] typically involve one to three high-angle boundaries (>15°). In addition, the misorientation angle of the extending boundary is not necessarily larger than those of the receding boundaries as in the model proposed by Li [19]. The present situation is thus different from early considerations of triple junction motion as a recovery mechanism, where two low-angle dislocation boundaries zip up [19,21]. The migration of a triple junction also differs from observations of triple junction motion during normal grain

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* Corresponding author. Tel.: +45 4677 4706; fax: +45 4677 5758; e-mail: tiyu@dtu.dk

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growth [7,22] after recrystallization in medium- to coarse-grained samples. The heavily deformed structure contains a high stored energy, e.g. $\sim$2 MJ m^{-3} in Al [8], and typically the driving force for Y-junction motion is significantly larger than that for grain growth. During Y-junction motion, both the mobility of Y-junctions and the mobility of boundaries may control the overall kinetics, whereas during grain growth the kinetics may be controlled solely by the boundary mobility. Moreover, the apparent activation energy increases significantly from 110 to 240 kJ mol^{-1} during Y-junction motion in Al [18] in line with Kuhlmann-Wilsdorf's suggestion [23], whereas during grain growth it is commonly found to be constant (e.g. [24]). On the other hand, some aspects of Y-junction motion may be similar to the coarsening of nanocrystalline materials with grain sizes below 100 nm, where there is also a large driving force and the kinetics are affected by triple junction mobility [25]. However, the structural morphology and size scale are significantly different between the current and the nanocrystalline case.

In previous studies [17,18] of triple junction motion during recovery, it was also observed that migrating Y-junctions interacted with the surrounding deformation structure and that these Y-junctions appeared to be pinned when meeting interconnecting and lamellar boundaries. Such a pinning mechanism adds to well-known mechanisms such as solute drag and particle pinning. Therefore this structural pinning mechanism is important not only fundamentally but also technologically, as it may retard or inhibit Y-junction motion and thereby increase the stability of strongly deformed pure metals and their alloys. In order to study the mechanisms underlying migration and pinning of Y-junctions in heavily deformed Al, we have chosen in the present study to use in situ annealing of samples in a transmission electron microscope.

2. Structural characterization

The structural morphology of highly strained metals is typically characterized by the presence of lamellar boundaries and interconnecting boundaries, which subdivide the structure. Based on their origins, these boundaries have been classified as geometrically necessary boundaries (GNBs) and incidental dislocation boundaries (IDBs), respectively [1,26]. The misorientation angle of GNBs increases more rapidly than that of IDBs during deformation, and after a high strain, the GNBs are typically of medium to high angle (>5°) and the IDBs are typically of low angle (<5°) [1,2]. In this structure the triple junctions were classified into three categories based on their morphology: those joining three lamellar boundaries were classified as Y-junctions; the other two are H- and r-junctions which involve interconnecting boundaries [17].

The material examined was commercial-purity Al AA1050 (99.5% purity with an initial grain size of $\sim$100 μm) cold rolled to true strains of 2, 4 and 5.5 at room temperature, where the smallest strain $\varepsilon = 2$ is near the lower bound for a completely lamellar deformation structure. Since the lamellar boundaries are parallel to the rolling plane [1,2], we choose the longitudinal section, containing the rolling direction (RD) and the normal direction (ND), for all transmission electron microscopy (TEM) observations. As a result, the quasi two-dimensional deformation structure is well represented in TEM thin foils, making it possible to observe characteristic structural changes taking place during annealing. The TEM foils were prepared using a modified window technique [27], and the thickness of thin areas for observation was about 0.2–0.3 μm.

2.1. In situ characterization

The in situ experiments were carried out in a JEM 2100 transmission electron microscope, which was operated at a relatively low accelerating voltage of 120 kV in order to reduce radiation damage. TEM foils of deformed states were mounted in a double-tilt holder. After the deformation structure was characterized, the foil was heated to 100 °C followed by a holding period, and then the temperature was further increased in steps of 20 °C at a heating rate of 0.2–0.4 °C s^{-1} with a holding period of 3–5 min between two temperature increases. The maximum temperature reached was typically 300 °C. During both heating and holding periods, the microstructural changes were recorded by a TVIPS FastScan camera with a maximum frame rate of 12 fps at full resolution. For some samples, the heating program was interrupted by cooling the sample to room temperature in order to better characterize the microstructure of intermediate states. A Kikuchi diffraction method [28] was used to determine the misorientation angles for boundaries of interest.

2.2. Stationary characterization

The quasi two-dimensional boundary structure can be well characterized in our TEM foils and the in-plane migration of Y-junctions is driven by in-plane driving forces, so the effect of free surfaces can be considered to be small for a qualitative study of this motion. However, minor processes occurred in these foils due to the free surfaces [29], and the kinetics of structural evolution is different from that in the bulk interior [17]. In order to study the partially recovered structure in the bulk interior, Al following a strain of 5.5 was annealed at 180 °C for 1 h, and the microstructure after annealing was examined in a JEM 2000FX transmission electron microscope, which was also operated at 120 kV.

3. Results

3.1. In situ observations

When annealed below 160 °C, the deformation structure in the TEM foil was stable and no structural changes were directly observed during in situ observation. After the annealing temperature was further increased, both dislocation activity and Y-junction motion coupled to local boundary migration became active. Most of the microstructural changes took place during short temperature increases, whereas only limited changes were observed during long temperature holding periods, in agreement with the quasi-logarithmic time dependence of recovery [30,31]. Both dislocation activity and Y-junction motion led to a decrease in the density of loose dislocations. Only Y-junction motion, however, changed the structural morphology. An overview of the structural change is shown in Fig. 1.