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Effect of annealing treatment on the microstructure and mechanical properties of ultrafine-grained aluminum

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ABSTRACT

Microstructural and property evolution of commercial pure Al subjected to multi-axil compression (MAC) and subsequent annealing treatment were investigated. After series of MAC pressings up to 15 passes, the samples were annealed at different temperatures. The deformed and deformed with sequent annealing treatment samples were characterized by X-ray diffraction, electron back scatter diffraction (EBSD), transmission electron microscopy (TEM) and tensile tests. The present results showed that on annealing the grain structures coarsen and transform from lamellar to equiaxed ones. Remarkably, the fraction of high angle grain boundaries drastically increases from 29.3% to 76.3% after annealing at 60 °C. Meanwhile, a significant decrease of lattice microstrain is observed after annealing, from 0.0839% to 0.0731% at 130 °C. A controlled 30 min annealing treatment on ultrafine-grained (UFG) Al at 60 °C can result obviously in a higher strength and a lower elongation, which may be associated with the nucleation and subsequent motion of dislocations in grain boundaries. As the annealing temperature is above 60 °C, the yield strength decreases and elongation increases gradually, which is attributed to the grain coarsening and microstructural enhancement.

1. Introduction

The production of bulk materials with nano-sized grains, especially through severe plastic deformation (SPD) processes has received considerable attention over the past decades. Metals and alloys processed by SPD exhibit much higher strength than those of conventional materials [1,2]. Additionally, much attention has been paid to the SPD methods because they are convenient ways to produce an ultrafine-grain (UFG) size in a bulk material. Multi-axial compression (MAC) as a novel technique in various SPD processings shows an advantage to fabricate bulk UFG material handleadility. So far, UFG materials produced by MAC have been extensively investigated because of their unique microstructure and excellent mechanical properties compared with conventional materials [3–6]. However, the novel properties are caused by non-equilibrium grain boundaries with extrinsic grain boundary (GB) dislocations [7,8], which leads to the instability of UFG materials. The microstructures of such UFG materials caused by storage and dynamic recovery of dislocations are at most in a metastable non-equilibrium state with limited thermal and mechanical stability [9,10]. That is to say, such microstructures can be easily changed by dynamic recovery and recrystallization under an applied loading even at room temperature, which is a critical problem for their practical engineering applications. Some studies [11,12]on the mechanical properties of UFG materials have presented the instability of their microstructure in the form of grain coarsening. Therefore, to take full advantage of the excellent mechanical properties, enhancing the thermal stability of SPD materials, annealing treatment has shown to be promising in reducing the macrostructural instabilities. However, it is incompatible to reduce the instabilities and maintain the excellent mechanical properties at the same time. Tremendous efforts have been making to set appropriate annealing temperature and time, which is a new attempt to UFG materials as potential engineering materials [3,6,12].

Akbari Mousavi et al. [3] discussed that annealing of deformed sample at 463 K for 10 min is responsible for new formed grains and the ultimate strength is enhanced about 45 MPa. They draw the conclusion that the enhancement of ultimate strength is attributed to the appropriate combination of temperature and time to annealing, which restrict apparent growth of grain. More recently, Huang et al. [13]observed that a nanostructured aluminum can be hardened by annealing treatment, accompanying with a reduction of ductility. They attributed this phenomenon to the variation of microstructural. Similarly, the work by Valiev et al. [14] indicated that controlled annealing treatment could bring about a 30% increase in strength of nanostructured SPD titanium simultaneously with enhanced ductility as compared to the case of as-produced state. Though the investigators have given some explanations to

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the above results [3,13,14], the effect of annealing treatment on microstructures and properties of UFG materials has not yet been studied systematically. Therefore, the study on annealing behavior of the UFG materials are expected to throw some light on the specific role of annealing temperature in microstructures and mechanical properties, to optimize the mechanical properties, especially to balance strength and ductility of nanocrystalline materials, along with reducing the instabilities.

This work focuses on the effect of annealing temperature on the microstructure and mechanical properties of the MACed samples. The commercially pure Al was first deformed by MAC at room temperature to obtain a submicron starting grain structure, followed by annealing at different temperatures for 30 min. The microstructure after annealing was analyzed in detail by electron backscatter diffraction (EBSD), transmission electron microscopy (TEM) and X-ray diffraction (XRD).

2. Experimental procedures

Commercial pure aluminum with the chemical composition given in Table 1 was used in this work to avoid the influence of phase transformation during the deformation and annealing treatment. The row commercial pure Al ingot was cut into 36 mm × 36 mm × 24 mm blocks and then annealed at 350 °C for 1 h before MAC to minimize the residual stresses in the samples with a grain size of about 20 μm.

To study the effect of annealing temperature on microstructures and properties of the as-MACed samples, all the samples were subjected to multiple pressing through a MAC mould at room temperature. MAC was carried out with a constant speed of 0.2 mm/s. All of the samples were pressed for 15 passes. The accumulated equivalent strain in MAC was 600%. Finally, annealing treatment was performed for 30 min at three different temperatures of 60 °C, 130 °C and 200 °C, which are around its recrystallization temperature (120 °C). All examinations were performed approximately at the middle of thickness. TEM observation was conducted on a JEOL JEM-200CX transmission electron microscopy at an accelerating voltage of 200 kV. Thin foils in the transverse cross section of the sheets were prepared by a twin-jet polishing technique in a mixture of 30% nitric acid and 70% carbinol at −30 °C. For EBSD measurements were carried out by using the program TSL-OIM in a Philips XL 30S SEM machine with a FE gun operated at 15 kV. Prior to EBSD analysis the surface of the sample was polished by a twin-jet polishing technique in a 10% perchloric acid and 90% carbinol at −20 °C. The step size and scan area for EBSD mapping were chosen as 0.1 μm and 25 μm × 25 μm, respectively. X-ray diffraction measurements were performed using a Scintag XDS2000, at room temperature with a diffractometer in a step scanning mode using Co Kα radiation. Specimens of ∼10 mm × 10 mm × 2 mm were cut from the central part of each sample. The XRD patterns were recorded with a scan rate of 4 s in the scanning range $2\theta = 20$–$90°$.A smaller angular steps of $2\theta = 0.01°$ was taken to measure the intensity of each Bragg reflexions.

Then, tensile tests of annealed and as-MACed samples were conducted at room temperature using a MTS810 electric servo-hydraulic testing machine. Tensile specimens were sliced from samples. The gauge section of tensile specimen was 10 mm × 5 mm × 2 mm with the long axis parallel to the final press direction according to the ASTM: E8M-11.

Table 1
Chemical composition of commercially-pure Al (in wt.%).

Al	Fe	Si	Mg	Zn	Other
99.8	0.11	0.055	0.021	0.01	0.004

3. Results and discussions

3.1. Microstructures

The microstructures of as-MACed and annealed Al are shown in Fig. 1. The as-MACed microstructure is mainly composed of severely elongated grains in the final press direction, and each elongated grain consists of the parallel bands of elongated substructures, 300 nm in width and 800 nm in length, containing high density of dislocations (seen in Fig. 1(a)). This appearance could be attributed to their low angle misorientation. This microstructure and the corresponding selected area electron diffraction (SAED) pattern also demonstrate that the microstructure consists of arrays of boundaries with low angle misorientations. After annealing at 60 °C for 30 min, the rearrangement of dislocations results in the disappearance of elongated grains. Formation of transverse boundaries and slight increase in the width of the sub-grains are responsible for reduction of the sub-grain aspect ratio, 600 nm in width and 900 nm in length. Most of the grain boundaries are curved, thick and fuzzy (see Fig. 1(b)). The spreading of the diffraction spots in the associated SAED patterns confirms the presence of boundaries with high angle misorientations. When the as-MACed Al undergo 130 °C and 200 °C annealing for 30 min, the GBs appear thinner and sharper and most of the elongated grains are substituted by equiaxed grains, whose average size further increase to 0.9 × 1.0 μm and 1.0 × 1.2 μm, respectively. The spreading of the diffraction spots in the associated SAED patterns confirms the presence of high angle grain boundaries in the microstructure (shown in Fig. 1(c) and (d)).

As is shown in Fig. 2, the distributions of the low-angle boundaries and high-angle one in MACed and annealed samples are exhibited in the EBSD maps. It is noted that in the EBSD maps black line mark the location of high-angle boundaries (i.e. the boundaries having a relative misorientation greater than 15°) and the red lines the location of low-angle boundaries (i.e. those having a relative misorientation between 2° and 15°).The EBSD maps for samples in different conditions display a substantial variation in grains and subgrains sizes.

The initial structure which consists of elongated grains changes completely into approximately equiaxed structure, obviously, as illustrated in Fig. 2. For the MACed sample presented in Fig. 2(a), structure predominantly consisted from mostly elongated grains, and the interior of the elongated grains include high density of low-angle boundaries in red lines, which is attributable to the high residual stress and lattice microstrain [15], so the sub-grain boundaries cannot be distinguished. Fig. 2(b) displays the EBSD map after annealing at 60 °C for 30 min. Compared with the microstructure after MAC, a significant change of grain morphology is observed. Some of the elongated grains are subdivided into subgrains by low angle grain boundaries, approximately equiaxed structure existed alongside within elongated grains suggesting the occurrence of a recovery process by rearrangement of dislocations in these boundaries. While some low angle grain boundaries turn into the high angle misorientation, fine submicron-sized grains (surrounded by high angle boundaries) are observed and the average boundary spacing is 0.9 × 1.1 μm. As is shown in Fig. 2(c), presenting the EBSD map after annealing at 130 °C for 30 min, fine grains induced by the initial deformation have grown into a coarse-grained structure in some part of the sample. And it is likely that recrystallization occurs at a temperature slightly below 130 °C. The microstructure mainly contains of high angle grain boundaries and becomes more homogeneous within the observed area, where the average boundary spacing is 1.5 × 1.7 μm, but in partial region some low-angle boundaries are still visible. After annealing at 200 °C for 30 min, with dramatic increase of grain size, equiaxed-