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Effect of annealing on microhardness and electrical resistivity of nanostructured SPD aluminium

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The influence of microstructure evolution on microhardness and electrical resistivity of ultrafine grained (UFG) commercial purity Al under annealing at different temperatures within a range of 363–673 K was studied. The initially coarse grained Al was processed by high pressure torsion (HPT) technique for the formation of UFG structure. The microstructure was characterized by electron backscattering diffraction and X-Ray diffraction. It was shown that annealing of UFG Al at temperatures within a range of 363–473 K leads to simultaneous increase of microhardness (by 6–13%) and electrical conductivity (by 4–8% at 300 K). The correlation between microstructural features and the resulting properties were analyzed. The average width *s* of potential barriers at grain boundaries (GBs) in HPT-processed Al was estimated in the frame of a tunnel model. The obtained large value of *s* compared with the GB crystallographic width is associated with elastically distorted lattice near GBs. The obtained results suggest a new way to increase simultaneously strength and electrical conductivity of UFG Al alloys by an appropriate annealing.

1. Introduction

In recent years Al alloys in nanostructured and ultrafine grained (UFG) states are of great interest due to their unique mechanical and physical properties [1–4]. Electrical conductivity of pure Al is 62% IACS (International Annealed Copper Standard), but due to lower density, Al has about twice higher conductivity per weight unit than Cu [5]. Considering the lower density and lower cost of Al compared with Cu, Al may be regarded as a very promising material to develop high-strength conductors for electrical power lines.

However, the main disadvantage of Al is its low mechanical strength. Conventional strengthening methods, including alloying and ageing, lead to dramatic decrease in electrical conductivity due to its strong dependence on the induced microstructural changes [6]. Achievement of high strength and good electrical conductivity for Al and Al-based alloys is a challenge and finding a solution for this problem will contribute to the increase of energy transmission efficiency and decrease of the power lines cost. Recently, a good combination of high strength and electrical conductivity has been demonstrated for Al-Mg-Si alloys processed by severe plastic deformation (SPD) in two stages: at room temperature (RT) and at elevated temperatures [7–9]. Refinement of grains down to ultrafine size by various SPD techniques at a temperature $<0.4T_m$ (T_m is the melting temperature) makes it possible to achieve high strength [1,3,4]. SPD processing at higher (elevated) temperatures increases electrical conductivity in the UFG alloy through dynamic ageing, which results in clearing the Al matrix from dissolved impurity atoms by the formation of nanosized secondary phase precipitates [7,8]. However, the ways to further increase strength while keeping or even increasing the high level of electrical conductivity are still of current interest. For example, introducing an additional dislocation density in the UFG structure with preserving other microstructural parameters leads to enhanced mechanical strength while keeping high electrical conductivity of the material almost unaffected [10]. As was shown recently, strength of UFG Al can be increased by appropriate annealing. In Refs. [11,12] the influence of

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annealing on microstructure and strength of commercial purity (CP) Al and high-purity Al processed by accumulative roll bonding (ARB) was studied. It was demonstrated that annealing at a temperature of the range 423–523 K during 0.5–1 h leads to the anomalous increase in strength. However, there are controversies on published issues relating to the effect of annealing on the strength of UFG Al. For example, in Ref. [13] it was shown that annealing of UFG Al processed by rotary swaging does not lead to the increase in microhardness throughout the annealing temperature range from 373 to 723 K.

The aim of this study is to investigate the possibility of simultaneous increase in strength and electrical conductivity of HPT-processed UFG CP Al by annealing and to identify the key microstructural parameters responsible for improvement of these functional properties.

2. Material and experimental procedures

Commercial purity Al (99.5 wt%) was chosen as the material of interest for this study because the effects of its solute atoms and precipitates are assumed to be negligible. Initially coarse grained (CG) disks of the diameter 20 mm and thickness 2 mm were subjected to SPD processing by high pressure torsion (HPT) [3] under a hydrostatic pressure of 6 GPa–10 revolutions at RT for the formation of UFG structure. After HPT processing the true strain at the distance of 5 mm from disk centre was γ~6.6 [3]. The HPT-processed samples were annealed at various temperatures in the range 363–673 K for 1 h to obtain a variety of microstructural parameters (grain size, grain boundary misorientation angle, dislocation density and others). Hereafter, the HPT-processed samples without annealing are referred to as Al_RT, the samples with subsequent annealing at T_{an} are referred to as Al_T_{an} (for example Al_363, Al_403 and so on).

Microstructural characterization was performed by electron backscattering diffraction (EBSD) analysis using the scanning electron microscope Zeiss Merlin. The samples for EBSD studies were prepared by conventional metallographic techniques consisting of polishing with the diamond and colloidal-silica suspensions. EBSD mapping was performed on a scan area of 32.6x24.4 μm^2 with a scan step of 0.2 μm and over 1000 grains were analyzed for each UFG sample. Seven Kikuchi bands were used for indexing the diffraction patterns. Distributions of grains on size and grain boundaries (GBs) between the adjacent grains on their misorientation angle θ were determined from the EBSD maps. GBs with $\theta \leq 15°$ were referred to the low-angle grain boundaries (LAGBs) and GBs with $\theta > 15°$ were considered as the high-angle grain boundaries (HAGBs). The grain-reconstruction method was applied to determine the average grain size [14].

Vickers microhardness H_V was measured using a Shimadzu HMV-G microindentation tester with a load of 1 N for 15 s. For reliable results, 3–4 samples were measured for each T_{an} and each sample was measured 15 times.

For electrical resistivity measurements the samples were cut from HPT disks according to the scheme in Fig. 1. Electrical resistivity ρ was measured by standard four-probe technique. Electrical resistivity was measured at RT, at 77 K in steady-state liquid nitrogen and at an intermediate temperature T_{in} around 200 K, which was achieved by natural evaporation of liquid nitrogen. The temperature of a sample was controlled by a silicon diode placed near the sample. The accuracy of temperature measurements was ±0.03 K. The obtained values of ρ_{77}, ρ_{Tin} and ρ_{RT} fitted well a linear dependence. To compare the resistivity of all studied samples at 300 K, the value ρ_{300} for each sample was determined from this linear approximation. Electrical resistivity measurements are described in more details in Ref. [15].

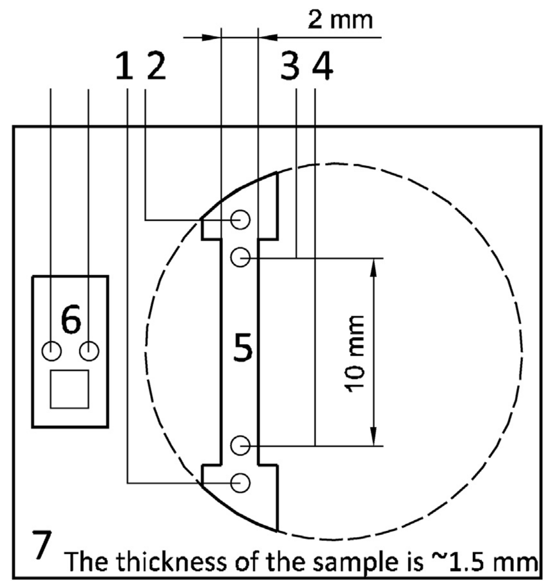

Fig. 1. Position of sample cut from the HPT disks (annealed and non-annealed) for the electrical resistivity measurements. Arrangement of current (1, 2) and potential (3, 4) contacts on the sample (5) and position of silicon diode (6) on the holder (7).

3. Results and discussion

3.1. Microstructure evolution

Fig. 2 presents the typical EBSD maps of HPT-processed samples before and after annealing at different temperatures. The distributions of grains on size and GBs on the misorientation angle are shown in Figs. 3 and 4, respectively. Microstructural parameters obtained from the EBSD maps (Fig. 2) are shown in Table 1. The average grain size (d_{av}) and the standard deviation (Δd) that characterizes the width of size distribution of grains are given for each sample. Amount of grains (in %) having the size below 1 μm, between 1 and 2 μm and above 2 μm (further denoted as $f_{<1}$, f_{1-2} and $f_{>2}$) and amount of GBs (in %) with the misorientation angle ≤15° and >15° (further denoted as $\theta_{\leq 15}$ and $\theta_{>15}$) are also shown in Table 1.

HPT processing transforms the initial CG structure into the UFG structure with equiaxed grains (Fig. 2a) having an average grain size d_{av} ≈ 810 nm, about 73% of grains having the size below 1 μm ($f_{<1}$ ≈ 73%). Most of GBs (77%) are HAGBs (Table 1). Annealing at 363 K does not lead to significant changes in the average grain size, it is equal to ~850 nm, but the value of $f_{<1}$ slightly decreases ($f_{<1}$ = 64%) and f_{1-2} increases from 26 to 34% compared to the non-annealed sample (Table 1). The portion of HAGBs does not change. The values of d_{av} and Δd increase gradually with the T_{an} growth from 403 to 473 (Table 1), the most of GBs remaining the high angle ones. Annealing at 673 K leads already to the formation of CG structure characterized by high value of d_{av} = 7 μm and very wide distribution of grains on size (Fig. 3f and Table 1). In the sample Al_673 the most of GBs are low angle.

Detailed XRD analysis of the studied samples was conducted in our previous work [15] and the estimated values of dislocation density L_{dis} on the basis of this analysis are presented in Table 1. As was shown, the annealing at 363 K of HPT-processed samples leads to the decrease in dislocation density by ~3 times from $2 \cdot 10^{12}$ to $7 \cdot 10^{11}$ m^{-2}, which indicates the beginning of structure recovery process (Table 1). Annealing at a higher temperature of 403K only slightly reduces the dislocation density compared to that in the Al_363 sample.