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Mechanical and electrical responses of nanostructured Cu–3 wt%Ag alloy fabricated by ECAP and cold rolling

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ABSTRACT

This paper investigated the mechanical and electrical responses of nanostructured Cu–3 wt%Ag alloy as a function of the strain imposed by a combination of equal channel angular pressing and subsequent cold rolling. The influence of cold rolling on the microstructural change was observed to be pronounced, resulting in the occurrence of lamellar band structure. Room-temperature tension tests results revealed that, as the amount of strain increased, the tensile strength of the nanostructured Cu–3 wt%Ag alloy increased while losing its ductility. The electrical conductivity slightly decreased with increasing amount of strain imposed. This phenomenon was mainly attributed to the nano-grained structure together with the non-equilibrium state of the grain boundaries.

1. Introduction

With the rapid growth in the electronic industries over the past several decades, attention has focused on the development of copper-based alloys having ultrahigh mechanical strength and high electrical conductivity as well. To this end, most of studies have attempted to enhance the required properties of copper alloys by experimenting with alloy design using silver, niobium, etc. [1–5]. Since several elements in copper-based alloys inevitably act as an impurity center for the scattering of electron motions, the electrical conductivity of such materials is inherently lower than that of low-alloyed materials in spite of their reasonably high strength due to solid solution hardening [1]. Thus, the use of solid solution hardening is not always desirable in this respect. To alleviate the problem, equal channel angular pressing (ECAP) method, in which significant structural refinement can be achieved without any change in chemical composition, has been applied with the aim of fabricating nanostructured materials combining high strength and fairly good electrical conductivity [6–8]. Also, Stolyarov et al. [9] recently reported that cold rolling (CR) was used as a secondary processing step following ECAP to further improve the strength of pure-titanium to a level comparable to that of titanium alloys.

To date, little information is available in literature on the microstructural evolution and mechanical properties of the nanostructured copper-based alloys processed by the combination of ECAP and CR. Indeed, Wang et al. [10,11] reported high performance of pure-copper with a bimodal grain size distribution controlled via ECAP and severe rolling. It is, however, noted that two significant differences were found in terms of processing variables such as rolling temperature and subsequent annealing treatment, leading to the microstructural difference. In addition, it remains doubtful whether, with a reduction in grain size, the electrical conductivity of copper-based alloys significantly drops to the extent of alloys deformed by conventional forming methods. In this study, we investigate the mechanical and electrical behavior of the nanostructured Cu–3 wt%Ag alloy prepared by ECAP and CR.

2. Experimental

The programmed material, Cu–3 wt%Ag alloy, was homogenized at 1023 K for 1 h and aged at 658 K for 8 h, resulting in a nearly equiaxed microstructure with an average grain size of ~50 μm, as shown in Fig. 1. After the sample was machined into a cylindrical specimen of 10 mm in diameter and 130 mm in length, it was deformed first by ECAP using a die with an effective strain of ~1. For ECAP, route B_C, in which the workpieces were rotated 90° clockwise along their longitudinal axes between consecutive passes, was selected since it generated better surface quality and more equiaxed grains than the other routes [6]. The samples were processed with either 4- or 8-passes of ECAP. After ECAP, the deformed samples were cold rolled at a speed of ~0.1 m s^{-1} with a height reduction of 1 mm per pass in order to avoid surface cracking until a height reduction of 80% was achieved giving an effective strain of ~1.6.

The room-temperature tension tests were performed at a strain rate of 1.0×10^{-3} s^{-1} on plate-type specimens (gage length: 10 mm, gage width: 4 mm, and gage thickness: 2 mm) machined along the ECAP direction. Three samples were tested for each processing condition to ensure reliable mechanical data. The deformed microstructure of the samples was observed by transmission electron microscope (TEM, JEOL-2010) after the samples were prepared by a twin-jet polishing technique. The electrical conductivity of the specimens was measured using an

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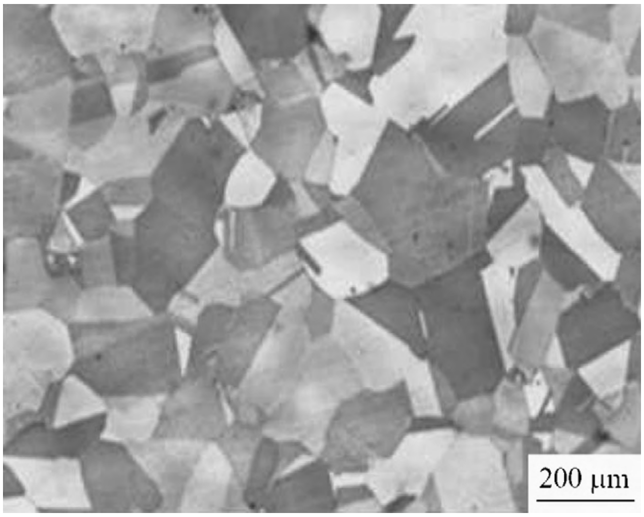

Fig. 1. Initial microstructure used in this study.

AutoSigma 3000-D calibrated as a percentage of the international annealed copper standard (%IACS). Although the SI unit for electrical conductivity is Siemens/meter, conductivity values are often reported as %IACS. The conductivity values in Siemens/meter were converted to %IACS by multiplying 1.72×10^{-6}.

3. Results and discussion

3.1. Microstructural evolution

Fig. 2 displays TEM micrographs of the Cu–3 wt%Ag alloy deformed either with or without CR following 4-pass ECAP. Fig. 2(a) shows the longitudinal section (Y-plane) of the sample subjected to ECAP prior to CR and Fig. 2(b) shows the normal section of the final, cold rolled sample. In Fig. 2, most of the (sub)grains were markedly elongated to the shear direction imposed by ECAP and/or CR, resulting in pronounced lamellar band structures. The deformed structures contained an ill-defined contrast and a high dislocation density inside lamellar bands, which is typical of several metals fabricated through single-pass of ECAP [6,12].

Fig. 3 shows TEM micrographs of the Cu–3 wt%Ag alloy deformed either with or without CR following 8-pass ECAP. After imposing an effective strain of ~8, most of the equiaxed grains were refined to ~350 nm in diameter. With increasing amount of strain imposed by CR, however, most equiaxed grains turned into band structure again, which was attributed to the formation of a preferred orientation. This finding revealed the pronounced influence of CR on the microstructural evolution, irrespective of the number of ECAP operations, resulting in the formation of the lamellar band structure. From the selected area diffraction pattern related to Fig. 3, the azimuth spreading of the diffraction spots indicated the existence of high internal stress, as discussed earlier [6,9,13]. Interestingly, a comparison of Figs. 2(a) and 3(a) revealed that an effective strain of ~4 was not sufficient to induce the formation of equiaxed grains in the Cu–3 wt%Ag alloy. This result differed from previous reports that 4-pass ECAP yielded equiaxed grains in several aluminum alloys [6,14]. When ECAP was applied to aluminum-based alloys, the band structures were clearly formed after single-pass, and the (sub)grained structure developed into the nanostructured grains divided by high-angle grain boundaries with increasing number of operations [15,16]. Although this process also took place in copper-based alloys, the evolution of a uniform, equiaxed structure was expected to be sluggish even under a high plastic strain of ~4. This phenomenon was closely associated with the difference in the value of stacking fault energy (SFE). Due to the lower SFE of the copper-based alloy (~40 mJ m^{-2}) [17] compared to the aluminum-based alloys (~200 mJ m^{-2}) [18], a weaker recovery process occurred, and the cross-slip of dislocations became more difficult during deformation [19,20]. Thus, a strain required for achieving a homogeneous, equiaxed structure would increase if all other processing variables are held constant.

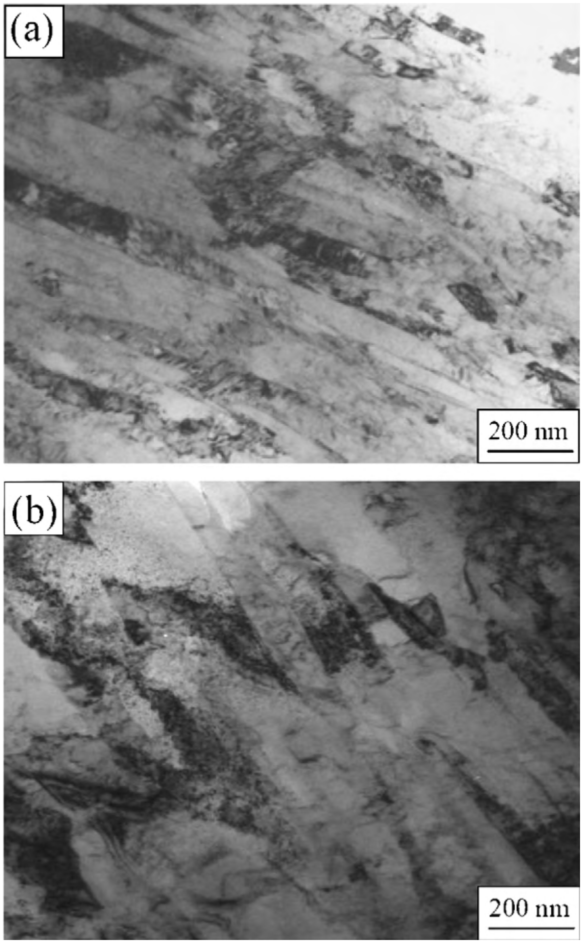

Fig. 2. TEM micrographs of the samples deformed by (a) 4-pass ECAP and (b) 4-pass ECAP followed by CR with a height reduction of 80%.

3.2. Mechanical properties

The nominal stress–strain curves of the initial and deformed Cu–3 wt%Ag alloys at ambient temperature are plotted in Fig. 4. The deformation of the alloy considerably altered its mechanical behavior. As the amount of strain increased, the yield and ultimate tensile stresses of the nanostructured Cu–3 wt%Ag alloys increased, approaching the maximum values of 617 and 647 MPa, respectively, whereas their ductility decreased. In general, materials deformed by ECAP typically exhibited increasing strength as the plastic strain imposed increased up to ~4, beyond which their strength became insensitive to further deformation. In the present study, however, the tensile properties continued to increase gradually with increasing ECAP passes up to an effective strain of ~8. This implied that microstructural development of the boundary characteristics from low-angle to high-angle grain boundaries was still in progress, which was also consistent with microstructural change experimentally observed in the preceding section. Furthermore, the nanostructured specimens exhibited poor strain hardenability at the onset of plastic deformation, causing a continuous decrement in the stress associated with a localized neck formation, which was geometrical softening in stress–strain curves. With subsequent CR

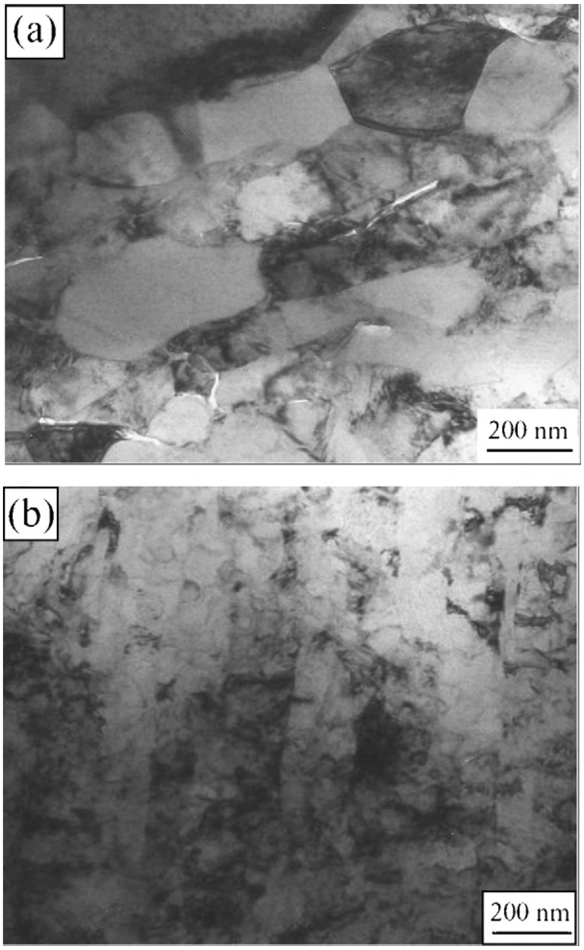

Fig. 3. TEM micrographs of the samples deformed by (a) 8-pass ECAP and (b) 8-pass ECAP followed by CR with a height reduction of 80%.

deformation following 4- and 8-pass ECAP, although the ductility slightly decreased, the ultimate tensile strength increased to 635 and 741 MPa, respectively, which were approximately three times higher than that of the initial sample. As clearly shown in Fig. 4, the combination of 8-pass ECAP and 80% CR afforded a good combination of high strength (765 MPa) and moderate ductility (11%), which were excellent mechanical properties for structural applications.

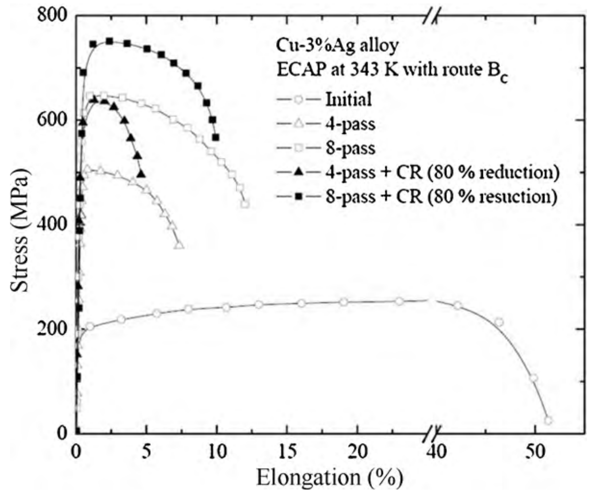

Fig. 4. The nominal stress–strain curves of the nanostructured Cu–3 wt%Ag alloys.

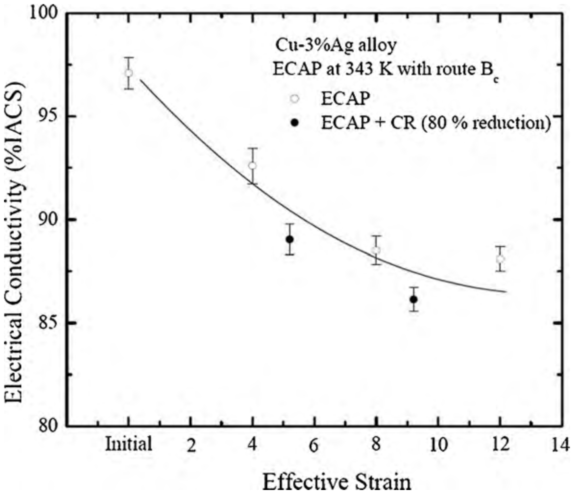

Fig. 5. Variation of the electrical conductivity with respect to accumulative strain.

3.3. Electrical conductivity

Fig. 5 shows the variation of the electrical conductivity of the Cu–3 wt%Ag alloy with respect to accumulative strain. It is certain that solute atoms/impurities of copper alloys would be dissolved during deformation, affecting the change in electrical conductivity. Here, more emphasis was placed on the influence of microstructure rather than that of compositional change [21,22]. The electrical conductivity of the samples decreased with decreasing grain size, as has been previously reported by several investigations [23]. Such a relationship was observed up to an effective strain of ∼10, but the sample deformed via 12-pass ECAP showed somewhat similar electrical conductivity to that deformed by 8-pass ECAP. It was thought that, although the amount of strain was large, this trend of the electrical conductivity was attributed to the rearrangement of point defects due to the enhanced diffusion triggered by heat generation during deformation and/or by non-equilibrium high-angle grain boundaries with high internal stress. Regarding the deformation heating, the samples were hardened with increasing amount of strain as shown in Fig. 4. Considering the fact that the more mechanical work was accumulated in material, the higher the heat was generated during next deformation step [24], the contribution of thermal energy to the diffusion rate during deformation would be significant. On the other hand, Kolobov et al. [25] found that the activation energy for grain boundary diffusion of nanostructured nickel was much lower than that of microcrystalline nickel. This was close to that of free surface diffusion due to the non-equilibrium state of the grain boundaries, which was caused by the intense plastic straining. It was confirmed from the present TEM observation that the non-equilibrium state of grain boundaries appeared. As a result, the dynamic recovery was likely to be activated by the microstructural characteristics, affecting the electrical conductivity.

4. Conclusions

Microstructural evolution, mechanical and electrical responses of the nanostructured Cu–3 wt%Ag alloy severely deformed by the combination of ECAP and CR were established in this study. TEM observation revealed that CR introduced pronounced lamellar bands with a high dislocation density inside them. Then, the ultimate tensile strength and electrical conductivity of the nanostructured Cu–3 wt%Ag alloy were estimated to be 765 MPa and 86%IACS at ambient temperature. Consequently, the combination of ECAP and CR was beneficial for producing nanostructured materials, which exhibited ultrahigh strength in tension with a small amount of drop in the electrical conductivity of the Cu–3 wt%Ag alloy.

Acknowledgements

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