Annealing effect on microstructure and mechanical properties of Cu-Al alloy subjected to Cryo-ECAP

Kun Xia Wei, Sheng Long Wang, Wei Wei*, Qing Bo Du, Igor V. Alexandrov*, and Jing Hu

Abstract: Cu-7%Al alloy subjected to equal channel angular pressing at cryogenic temperature with liquid nitrogen cooling (Cryo-ECAP) was treated by annealing. The microstructure and mechanical properties of Cu-7%Al alloy before and after annealing were investigated. It shows that a large number of annealing twins formed in Cu-7%Al alloy subjected to Cryo-ECAP. After 300°C and 0.5 h annealing in Cu-7%Al alloy processed by Cryo-ECAP, tensile strength and uniform elongation was increased up to 644 MPa and 76% respectively. The enhanced mechanical properties of Cu-7%Al alloy after annealing is attributed to the high density nanoscale twins.

Keywords: Cu-7%Al alloy; cryo-ECAP; annealing; microstructure; mechanical properties

Introduction

Cu-Al alloy is one of the most common materials used in mechanical manufacturing due to good mechanical properties, high corrosion and wear resistance. With the higher requirements for service properties of the Cu-Al alloy, developing the Cu-Al alloy with high strength and large elongation has become a trend [1, 2]. Compared with the traditional strengthening way, nanotwins can improve the mechanical properties of materials, and maintain a good ductility at the same time. Twin boundary can not only block the dislocation movement to improve the strength of the material, but also can absorb the dislocation to withstand larger plastic deformation [3–5]. Equal channel angular pressing (ECAP) is an effective way to obtaining ultrafine grains or nanotwins [6, 7]. The former Soviet Union scientist Segal put forward the principle of ECAP [8]. ECAP has been widely applied for ultrafine grained material. However, most of the literatures on ECAP were carried out at room temperature [7–10]. Few works of severe plastic deformation (SPD) has been done for nanoscale twins at cryogenic temperature [11–14]. Cryo-rolling or ECAP can increase the tensile strength and micro-hardness, compared with the counterparts in ambient conditions [11, 12]. However, it is opposite in Al-Mg alloy due to the lower strain rate sensitivity and the higher activation volume [15]. The non-oriented electrical steel rolled at cryo-conditions achieves better magnetic properties after annealing due to the optimal grain size and the increase of the cubic texture [16].
Cu-7%Al alloy subjected to ECAP at cryogenic temperature with liquid nitrogen cooling (Cryo-ECAP) was treated by annealing. The microstructure and mechanical properties of Cu-7%Al alloy before and after annealing were investigated.

2 Experimental procedures

Cu-7%Al (wt. %) samples with a size of 12 mm×12 mm×80 mm were subjected to annealing treatment at 800°C for 2 h in NBD-01200 tubular vacuum furnace. Before ECAP samples were dipped into liquid nitrogen (-196 K) and maintained for 5 min. Cu-7%Al alloy specimens were soaked in the liquid nitrogen for 5 min after ECAP deformation. ECAP was conducted in air with a pressing speed of 0.25 mm s⁻¹ using a die with φ=120° and ψ=0°. The lubricant was MoS₂ mixed with machine oil. After the Cryo-ECAP, Cu-Al samples were subjected to annealing treatment at 300°C for 30 min.

Microstructure characterization was conducted on JEOL-2100 transmission electron microscope (TEM). The prepared thin samples were then first mechanically ground to about 50 μm thickness and finally thinned by the DJ 2000 twin-jet electro-polishing device in a solution of 33% nitric acid and 67% methanol at -25°C with a current of 80-90 mA. TEM was done in a conventional manner at 200 kV. X-ray diffraction (XRD) was conducted using a D/Max-2500pc X-ray diffractometer. The parameters of XRD was 40 kV/100 mA and scanning angle was 30°-100°. A load of 0.3 kgf was applied for 15 s using a HXD-1000TM digital hardness tester. Tensile properties were tested using machine-cut samples with a dimension of 1×3 mm² in cross section and 8 mm in gauge length. Strain measurements were confirmed by measuring the elongations in the marked gage length. All tests were carried out at room temperature using a Shimadzu AGS-10kND machine operating at cross-head speed 1 mm min⁻¹.

3 Results and discussion

Figure 1 (a) and (b) shows the TEM images of the Cu-7%Al alloy before annealing treatment. TEM images of the Cu-7%Al alloy after annealing treatment at 300°C for 30 min are shown in Figure 1 (c) and (d). Before annealing treatment, it can be seen that lots of twin lamella with different width with high density of dislocation. Many nanotwins can be observed in Figure 1 (b). After annealing, there are more fine twins. In other words, annealing treatment could enhance the density of twins in Figure 1 (c) and (d). The mechanism responsible for improving the density of twins after annealing treatment is still dark.

High strain rates, low temperatures and SFEs facilitate twinning in the face-centered cubic (FCC) metals [16]. In fact, the effect of the strain rate, ε, and the temperature, T on the deformation microstructure can be expressed through the Zener-Hollomon parameter [17]:

$$\ln Z = \ln \dot{\varepsilon} + \frac{Q}{RT}$$  \hspace{1cm} (1)

where Q is the activation energy for deformation, taking into account the grain boundary self-diffusion. R is the gas constant. For the low SFE alloys, the highly regular boundaries are available readily for enhancing the tendency to raise the activation energy. Therefore, the lower are the SFE and the deformation temperature, the higher is ln Z, inducing an increase in twinning capability and the twins propagation in Figure 1. Deformation twinning occurs and the number of twins increases with increasing ln Z [18].

Figure 2 shows the XRD patterns for Cu-7%Al alloy before and after annealing treatment. The internal residual stress of the material led to a shift in the XRD peaks. There is no obvious shift in the peaks and changes in the peak shape. Through the XRD, it is possible to calculate the crystallite sizes and the microstrain of the Cu-7%Al alloy after cryo-ECAP. After annealing at 300°C for 30 min, the average grain size of the alloy increased from 213 nm to 221 nm, and the microstrain of material reduced from 0.330% to 0.298% as shown in Table 1. The dislocation density ρ, may be calculated from crystallite size d, and microstrain, \(\langle\varepsilon^2\rangle^{1/2}\), measured by XRD using the relationship [19]:

$$\rho = \frac{2\sqrt{3}\langle\varepsilon^2\rangle^{1/2}}{d \cdot b}$$  \hspace{1cm} (2)

Where b is the absolute value of the Burgers vector. After annealing treatment, the dislocation density of Cu-7%Al alloy decreased from 2.1×10¹⁴ to 1.8×10¹⁴. These measurements show that the Cu-7%Al alloy with the lower SFE (5 mJ/m²) effectively blocked the dislocation slip because of the wide stacking fault when the alloy subjected to annealing treatment.

Figure 3 shows the relationship of microhardness and annealing temperature when Cu-7%Al alloy was annealed for 15 min. Microhardness was increased with the annealing temperature and up to the maximum at 300°C. As the annealing temperature increased, microhardness was rapidly decreased due to the recrystallization in the alloy. Along with the increase of annealing temperature, the effect of annealing hardening began to shift to the recrystallization in the alloy. When the alloy was subjected to annealing treatment at 300°C, the relationship between the
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Figure 1: TEM images of the Cu-7%Al alloy before (a) and (b) and after annealing (c) and (d).

Table 1: Dislocation density and effective grain size of Cu–7%Al alloys after Cryo-ECAP and annealing.

<table>
<thead>
<tr>
<th>States</th>
<th>$b$  / nm</th>
<th>$(\varepsilon^2)^{1/2}$ /%</th>
<th>$d$ / nm</th>
<th>$\rho/10^{14}$ m$^{-2}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cryo-ECAP</td>
<td>0.2593</td>
<td>0.330</td>
<td>213</td>
<td>2.1</td>
</tr>
<tr>
<td>Cryo-ECAP+Annealing</td>
<td>0.2593</td>
<td>0.298</td>
<td>221</td>
<td>1.8</td>
</tr>
</tbody>
</table>

With an increase of annealing time, the hardness of the Cu-7%Al alloy increased slowly and decreased when the annealing time was prolonged.

Figure 5 shows the tensile engineering stress-strain curves of Cu-7%Al alloy at room temperature before and after annealing at 300°C for 30 min. Before annealing treatment, the tensile strength and the uniform elongation of the alloy are 585 MPa and 6.9%, respectively. After annealing treatment at 300°C for 30 min, the tensile strength and the uniform elongation of the alloy was 644 MPa and 7.6%, respectively. Therefore tensile strength and uniform elongation were enhanced after annealing treatment.

The results of XRD show that after annealing treatment at 300°C for 30 min, the average crystallite sizes of the Cu-7%Al alloy increased slightly, the corresponding alloy internal microstrain and dislocation density are reduced slightly. In Figure 1, before annealing treatment, a high density of dislocation was accumulated in the alloy, the lower SFE and wide stacking fault in the alloy, the finer the formation of annealing twin [11, 14]. There is a large number of nanoscale annealing twins after annealing treatment at 300°C. Due to the effect of the interaction of dislocations and twins, the twin boundaries not only can block the dislocation motion, but also can absorb the dislocations. Thus it can enhance the strength and ductility simultaneously.
Figure 2: XRD patterns for Cu-7%Al alloy before and after annealing treatment.

Figure 3: Microhardness of Cu-7%Al alloy as a function of the annealing temperature.

Figure 4: Microhardness of Cu-7%Al alloy as a function of the annealing time.

Figure 5: Tensile engineering stress-strain curves of Cu-7%Al alloy before and after annealing at 300°C for 30 min.

4 Conclusions

1. A large number of annealing twins formed in Cu-7%Al alloy subjected to Cryo-ECAP.
2. After the annealing treatment, the average crystallite sizes of Cu-7%Al subjected to Cryo-ECAP increased slightly, while the microstrain and dislocation density of the alloy decreased slightly.
3. In Cu-7%Al alloy processed by Cryo-ECAP and subsequent annealing, tensile strength and uniform elongation was increased up to 644 MPa and 7.6% simultaneously.

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