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Grain refinement and mechanical properties of Cu–Al 10%– Fe 4% alloy produced by equal channel angular extrusion

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Equal channel angular extrusion (ECAE) process was carried out for a commercial aliminium bronze alloy (Cu–Al 10%–Fe 4%) produced by hot-rolling at high temperatures. A suitable processing temperature of ECAE for the alloy was determined. The effect of ECAE on microstructural evolution and mechanical properties of the alloy was investigated. Experimental results showed that the extrusion temperature must be higher than the eutectoid reaction temperature of the alloy. Optical electron microscopy and X-ray diffraction (XRD) were used to study the microstructural evolution of the alloy. The results showed that the grains of the alloy were refined after ECAE and gradually reduced with the increase of the pass number; accordingly, the mechanical properties of the alloy were significantly improved after ECAE.

Key words: *fine-grained aliminium bronze alloy; ECAE; microstructural evolution; mechanical properties*

1. Introduction

Equal channel angular extrusion (ECAE) technique is a severe plastic deformation process invented by Segal et al. [1]. An important advantage of ECAE is that it imposes much higher plastic strain during pressing without reducing the cross-sectional area of working billets, resulting in unique combinations of mechanical properties and grain size. Recently, active research efforts have been made and successful applications have been reported for various materials such as pure copper [2–4], Al alloys [5–8], magnesium alloys [9, 10] and Ti alloys [11, 12], etc.

The effect of ECAE on microstructure and mechanical properties of pure copper has been investigated in detail. However, previous ECAE studies have mostly been

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concerned with producing an ultrafine-grained microstructure from single-phase alloys. For single-phase alloys with high ductility, ECAE is usually carried out at room temperature. For two-phase alloys, an increase of the extrusion temperature is generally required to increase their ductility because the two-phase microstructure with a high volume fraction of the second phase is too brittle to be deformed during ECAE at low temperature [13]. Not many attempts have been made so far to process two-phase alloys by ECAE. Therefore it is essential to establish optimum processing conditions of such difficult-to-fabricate materials to study the effects of ECAE on microstructural changes and mechanical properties of two-phase alloys.

In this study, a commercial aliminium bronze alloy (Cu–Al 10%–Fe 4%) was chosen to determine its suitable processing temperature for ECAE, and the effect of ECAE on the microstructure and mechanical properties was also investigated.

2. Experimental

A Cu–Al 10%–Fe 4% alloy rod obtained in the as-rolling conditions was used as experimental material for ECAE. The billets with 9.6×9.6 mm^2 in cross-sections and 100 mm long were cut from the alloy rod. The die used for ECAE consisted of two rectangular channels with the cross-section area of 10×10 mm^2 intersecting at the angle of 90°. The billets were coated with a graphite lubricant to reduce the friction between the die and the billets during ECAE. The ECAE processes were carried out at 550 °C, 600 °C and 650 °C , respectively.

Optical electron microscopy and XRD were used to study the microstructural evolution of the alloys. The specimens for microstructure observation were cut along the extrusion direction. They were ground mechanically using abrasive papers and alumina powders, and their surfaces were etched by immersing in a solution of 8% HF, 22% HNO_3 and 70% H_2O for about 15 s.

Vickers microhardness measurements were taken with loads of 50 g applied for 13 s. The specimens for tensile test were machined from the as-received and the extruded billets with the gauge length of 25 mm and cross-section of 2×2 mm^2. The specimen axis was aligned with the extrusion direction. The tensile test was conducted at room temperature with the strain rate of 10^{-3} s^{-1}.

3. Results and discussion

Figure 1 shows the external appearances of the billets with ECAE after one pass at three different temperatures. It can be seen from Fig. 1 that, at the temperature of 550 °C and 600 °C, the ECAE process has not been carried out successfully. At the extrusion temperature of 550 °C, the billet exhibits failure characterized by the formation of a series of segments, as shown in Fig. 1a. At the extrusion temperature of 600 °C, there are some extensive cracks on the surface of the billet but the billet does not break, as shown in Fig. 1b. When using a higher extrusion temperature (650 °C), the billet is extruded successfully without breaking and any surface cracks, as shown in Fig. 1c. In addition, the extruded material with ECAE after one pass is pressed easily at 650 °C through four passes without surface cracks.

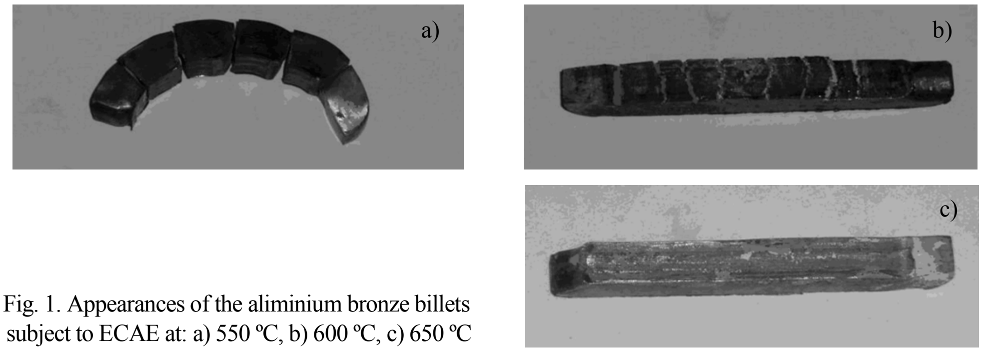

Fig. 1. Appearances of the aliminium bronze billets subject to ECAE at: a) 550 ºC, b) 600 ºC, c) 650 ºC

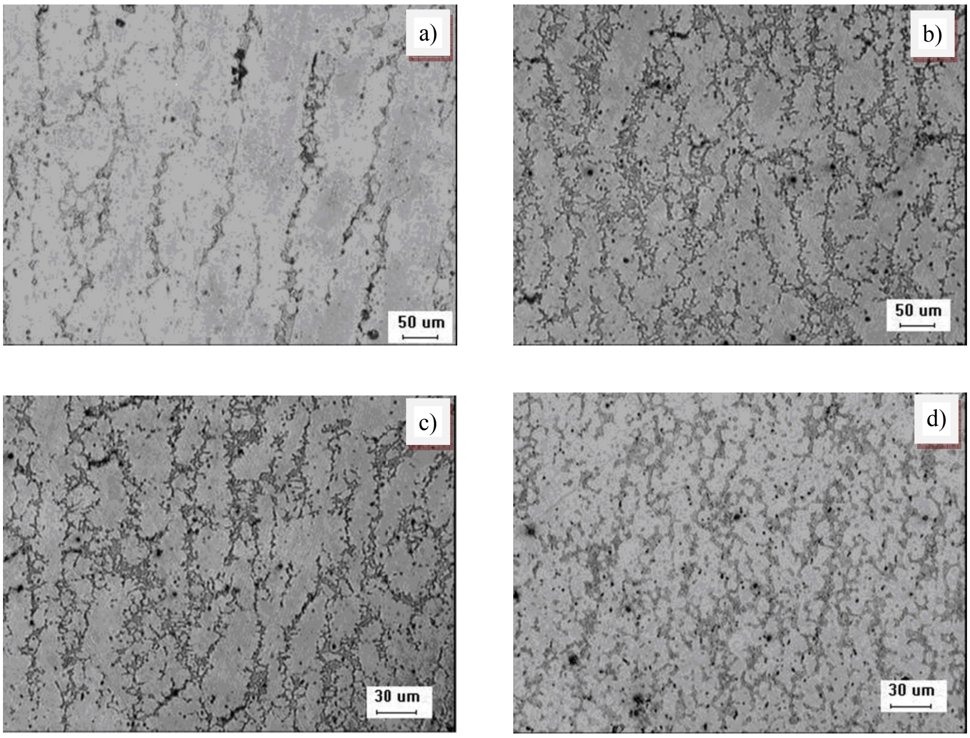

Fig. 2. Microstructures of aliminium bronze specimens: a) without ECAE, b) after ECAE with one pass, c) after ECAE with two passes, d) after ECAE with four passes

Microstructure of the as-received aluminium bronze alloy consists of two phases, α phase and $(\alpha + \gamma_2)$ eutectoid structure, as shown in Fig. 2. The γ_2 phase is brittle and difficult to deform during ECAE. At the extrusion temperature of 550 °C, ECAE is carried out below the eutectoid reaction temperature and γ_2 phase still exists in the microstructure of the alloy. Thus, ECAE process failed because of the brittle nature of γ_2 phase. At 600 °C, although the extrusion temperature is higher than the actual eutectoid reaction temperature, extensive cracks are still present on the surfaces of the specimens. The main reason is due to heat losses when the billet and the die are taken out from furnace during ECAE. When the extrusion temperature increases, for example to 650 °C, ECAE process can be performed successfully. It is reasonably explained that γ_2 phase disappears when the extrusion temperature is higher than the eutectoid reaction temperature of the alloy.

Figure 2 shows the optical images of the specimens without ECAE, after one pass, after two passes and after four passes of ECAE. It can be seen form Fig. 2a that the microstructure of the specimens without ECAE exhibits strong wire texture and the measured average width of the texture is about 70 μm. The α phase parallels the second phase and both of them are elongated along the rolling direction. After one pass of ECAE, the grain structure is refined and the microstructure of the specimen evolves into a structure with a considerable fraction of low-angle boundaries, as seen in Fig. 2b. Most of the low-angle grain boundaries still align along the extrusion direction, but some low-angle boundaries deviate from the extrusion direction. Therefore, it can be seen that in some areas, α phase intersects the second phase and the grain boundaries are no longer straight, but more curved than the boundaries without ECAE. After two passes of ECAE, the spacing of the boundaries is about 30 μm and some equiaxed grains occur in some areas, although the proportion of low angle grains is large, as shown in Fig. 2c. After four passes of ECAE, the grain size is further refined and the spacing of the boundaries is about 21 μm, as shown in Fig. 2d.

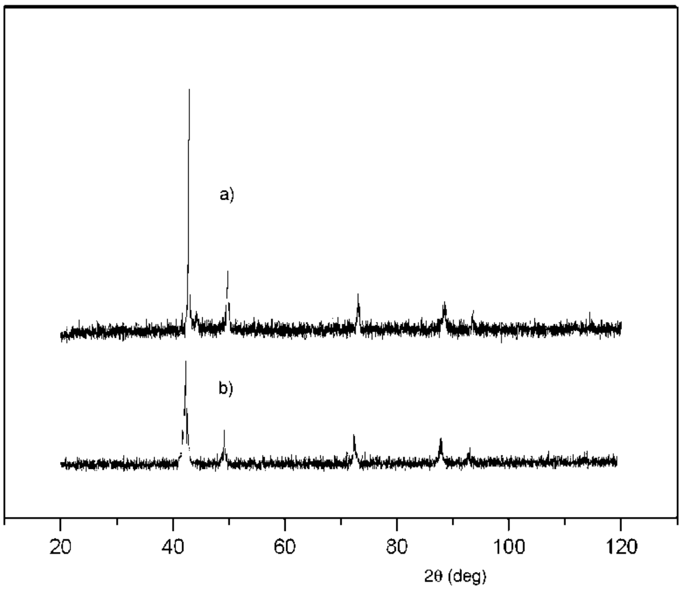

Fig. 3. XRD patterns of aliminium bronze alloy specimens without ECAE (a) and after ECAE (b) with two passes

The occurrence of some equiaxed grain structure in a few areas of the specimen indicates localized deformation and new slip systems operate during ECAE [14]. Prior low-angle boundaries are truncated by different shear planes, forming a microstructure with large angle boundaries. As a result, the grain size reduces progressively with the increase of pass number of ECAE.

Figure 3 shows the XRD patterns of specimens without ECAE and after ECAE with two passes. It can be seen that the XRD pattern of a specimen with ECAE after two passes exhibits significant broadening of all peaks, which is associated with the grain refinement, defect density increase and higher lattice distortion [15]. It also can be seen that the volume fraction of eutectoid structure of the specimens after ECAE is higher than that of the specimen without ECAE. This is attributed to the use of a high extrusion temperature which leads to an increase of the second phase. ECAE is carried out at a temperature of 650 °C, which is higher than the eutectoid reaction temperature of the alloy. After ECAE process, the temperature of the billet drops quickly because of larger difference in temperature between the billet and the atmosphere. The phase transformation ($\beta \rightarrow \alpha$) cannot be carried out completely and the β phase is still partially preserved. As a result, more β phase is available to the subsequent eutectoid reaction ($\beta \rightarrow \alpha + \gamma_2$) and the volume fraction of the second phase increases after ECAE.

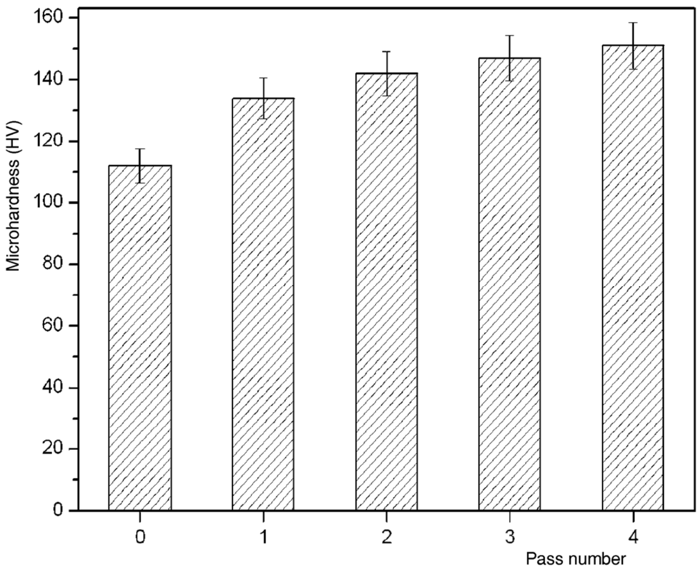

Fig. 4. Vickers microhardness of the aliminium bronze specimens with various pass numbers

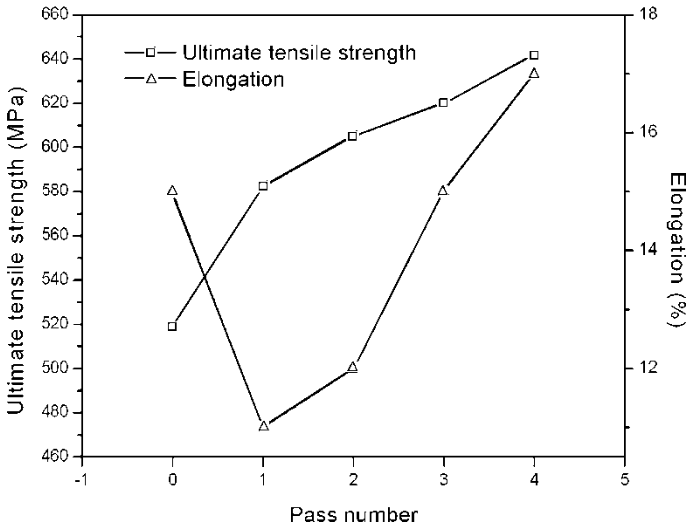

Fig. 5. Ultimate tensile strengths and tensile elongations of the aliminium bronze specimens with various pass numbers

Vickers hardness, ultimate tensile strength and tensile elongation of specimens with various pass numbers are shown in Figs. 4 and 5, respectively. It is seen that the hardness and the ultimate tensile strength of the specimens after ECAE are higher than that of the specimen without ECAE. The hardness and the ultimate tensile strength increases remarkably with a significant decrease in elongation after one pass of ECAE. However, the hardness, ultimate tensile strength and elongation increase with the increase of the pass number after two passes of ECAE.

The improvement of the mechanical properties is attributed to the difference in microstructures between as-received and extruded aliminium bronze specimens. Grain size decreased and grain was refined during the progress of ECAE. Grain refinement can affect mechanical properties of polycrystalline materials [16]. The classical effect of grain size on hardness can be explained by the Hall–Petch model [17]:

$$H = H_0 + kd^{-0.5} \tag{1}$$

where H_0 is the hardness constant, k the constant and d is the diameter of the grain. The Hall–Petch effect in grain refinement materials is attributed to the grain boundaries acting as efficient obstacles to dislocations. Consequently, a dislocation pile-up can be formed against a grain boundary inside a grain. In addition, it can be seen from Figs. 2a, b that the volume fraction of the second phase increases remarkably after one pass of ECAE. However, the volume fraction of the second phase does not increase after two passes of ECAE, as shown in Figs. 2b, c. It is well known that hardness and ultimate tensile strength are directly proportional to the volume fraction of the second phase [18]. Thus, the increase in hardness and ultimate tensile strength after the first pass is larger than that of the specimen with ECAE after two passes. And the decrease in elongation after one pass is also mainly due to a high volume of the second phase produced at high temperature. After two passes of ECAE, the distribution of the second phase is rearranged and some equiaxed grains occur in the microstructure of the alloy, which leads to a stronger material with more dispersed distribution of the second phase. In addition, the grain size of the alloy is further refined after the second pass, and this grain refinement can result in an increase in strength and elongation of the alloy [19]. Therefore, the improvement in mechanical properties of the billet after two passes of ECAE is due to rearrangement of the second phase and grain refinement.

4. Conclusions

It is demonstrated that the extrusion temperature of ECAE for aliminium bronze alloy must be higher than the eutectoid reaction temperature of the alloy. After ECAE, the alloy has more homogeneous fine-grained structure than the as-received one and some equiaxed grains occur in some areas. The fraction volume of the second phase increases after one pass while it does not increase during the subsequent pass. With the increase of the pass number of ECAE, the grain size of the alloy progressively decreases, and the hardness and strength gradually increase. After one pass of ECAE, the increase of hardness and strength of the specimen is due to grain refinement and the increase of the second phase, while the improvement in mechanical properties of the alloy after two passes of ECAE is a consequence of grain refinement and rearrangement of the second phase.

Acknowledgements

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