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EFFECT OF ANNEALING TEMPERATURE ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ULTRAFINE GRAINED BRASS PRODUCED BY EQUAL CHANNEL ANGULAR PRESSING

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ABSTRACT

The present study investigated the mechanical properties and microstructure of ultrafine grained (UFG) brass processed by four passes of equal channel angular pressing (ECAP) and annealed at elevated temperatures. The mechanical properties of all samples were assessed using tensile and micro-hardness tests. Microstructure analysis was performed using optical microscopy (OM) and scanning electron microscopy (SEM). Ultimate tensile strengths (UTS) and yield strengths (YS) of 878 and 804 MPa, respectively, ductility of 15%, and hardness of 248 HV were obtained for samples processed by four passes of ECAP with equivalent true strain of 4.20. Annealing at 300°C caused UTS and YS to decrease significantly, to 510 and 408 MPa, respectively, ductility to increase to 28%, and hardness to decrease to 165 HV. Fractography analysis of un-annealed samples after four ECAP passes showed small brittle fractures with shallow dimpling. Ductile failures were found on annealed samples. After four ECAP passes, the microstructure of un-annealed samples was UFG and dominated by lamellar grain with shear band. In contrast, after annealing, the microstructure changed due to recrystallization, showing nucleation and grain growth.

Keywords: Annealing; ECAP; Mechanical properties; Microstructure; Ultrafine grained

1. INTRODUCTION

Many studies have been conducted on improving the mechanical properties of metals. One area of investigation has been increasing strength by improving the microstructure using thermal and mechanical treatments. Such improvements refine the metal's grain and microstructure to either ultrafine-grained (UFG) or nano-sized. In accordance with the Hall-Petch theory, refining the grain has been shown to increase strength (Azushima et al., 2008; Valiev & Langdon, 2006; Pasebani & Toroghinejad, 2010).

In the last several decades, efforts to refine metal microstructure have focused on deforming the metal using a great strain, which is called severe plastic deformation (SPD) (Azushima, et al., 2008; Valiev & Langdon, 2006; Valiev et al., 2000; Pasebani & Toroghinejad, 2010). SPD can deform metal microstructure to UFG or nano-sized grains (nanocystallins). Several SPD

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methods have been developed, including high-pressure torsion (HPT) (Zhilyaev et al., 2014; Valiev & Langdon, 2006), accumulative roll bonding (ARB) (Pasebani & Toroghinejad, 2010; Azushima et al., 2008; Valiev et al., 2000), multidirectional forging (MDF) (Gubizca et al., 2011; Azushima et al., 2008), rolling at cryo temperatures (cryorolling, (CR)) (San et al., 2012; Kumar et al., 2015), and equal channel angular pressing (ECAP) (Suryadi et al., 2013; Bahadori et al., 2013; Azushima et al., 2008). ECAP, first introduced by Segal in the 1970s and 1980s (Valiev & Langdon, 2006), has considerable SPD potential, enabling it to produce UFG metals with relatively large dimensions. ECAP can produce UFG metal rods and square bars, while other SPD processes produce UFG metal of only limited shapes and sizes.

UFG metal requires advanced processing to reach a final product. It is important to note that such processing changes the metal's characteristics, including its mechanical properties and microstructure, and deforms the UFG metal Combinations of SPD and advanced processing have been performed. One study produced UFG metal of Cu-18Zn using CR with 90% area reduction, followed by short annealing (Kumar et al., 2015). This combination produced nanostructures of 48 nm and significant changes in mechanical properties after annealing at various temperatures. In another combination, ECAP followed by cold rolling produced a UFG strip of copper with a smooth structure and increased tensile strength (Bahadori et al., 2013).

The objective of the current research was to analyze changes in the microstructure and mechanical properties of UFG brass Cu-Zn 70/30 produced using four ECAP passes and annealed at various temperatures.

2. EXPERIMENTAL PROCEDURE

The present study used samples taken from a 13-mm thickness of commercial brass having the chemical composition shown in Table 1. The plates were cut longitudinally to a width of 12 mm and then machined by lathe into rods 10 mm in diameter and 80 mm long. Before deformation by ECAP, the samples were annealed at 600°C for 90 min to produce a homogenized microstructure with a grain size of ~45 μm.

The ECAP process was conducted at room temperature using a solid die in which the two parts of the channel intersected at an internal angle of 90° (Φ) and an arc of curvature of 20° (Ψ). The values of Φ and Ψ resulted in an equivalent true strain of ~1.05 on each pass through the die (Azushima et al., 2008; Suryadi et al., 2013). The samples were pressed on the dies using a 1000-kN Schenk Trebel universal tensile testing machine with a pressing speed of 5 mm/s. To produce a UFG structure, the samples were processed by ECAP through 4 passes using route Bc, in which the sample is rotated 90° in the same direction between each pass (Azushima et al., 2008; Bahadori et al., 2013). After 4 ECAP passes, the accumulated equivalent true strain was ~4.2. Then, the samples were heated in an electric furnace at temperatures of 200, 300, 400, and 500°C for 15 min.

Table 1 Chemical composition of the brass alloy

Components	Zn	Fe	Al	Ni	Pb	Cu
Wt.%	29.34	0.048	0.026	0.02	0.025	Balance

The microstructures of the samples were observed cross-sectionally and longitudinally using optical microscopy (OM). To observe the microstructures, the samples were cut, mounted, ground, polished, and etched using $K_2Cr_2O_7$ (2 g), H_2O (100 ml), H_2SO_4 (8 ml), and HCl (2 drops). The linear-intercept method was used to measure the grain size at the center length from the line intersections.

The mechanical properties were identified using the Vickers micro-hardness and tensile tests. Vickers micro-hardness tests were conducted on sample surfaces using a load of 3 kg for 15 s. The tensile tests were conducted using a Schenk Trebel universal tensile testing machine with a capacity of 1,000 kN. The samples were prepared for the tensile tests according to the American Society for Testing and Material (ASTM) E-8 standards, with a gauge length of 12.5–25 mm and a diameter of 3 mm. Surface fractures on the tensile test samples were evaluated using scanning electron microscopy (SEM).

3. RESULTS AND DISCUSSION

3.1. Microstructure Investigation

Figure 1 shows the microstructure of the Cu-Zn 70/30 alloy before deformation by ECAP, after four ECAP passes, and following annealing at temperatures of 200, 300, 400, and 500°C.

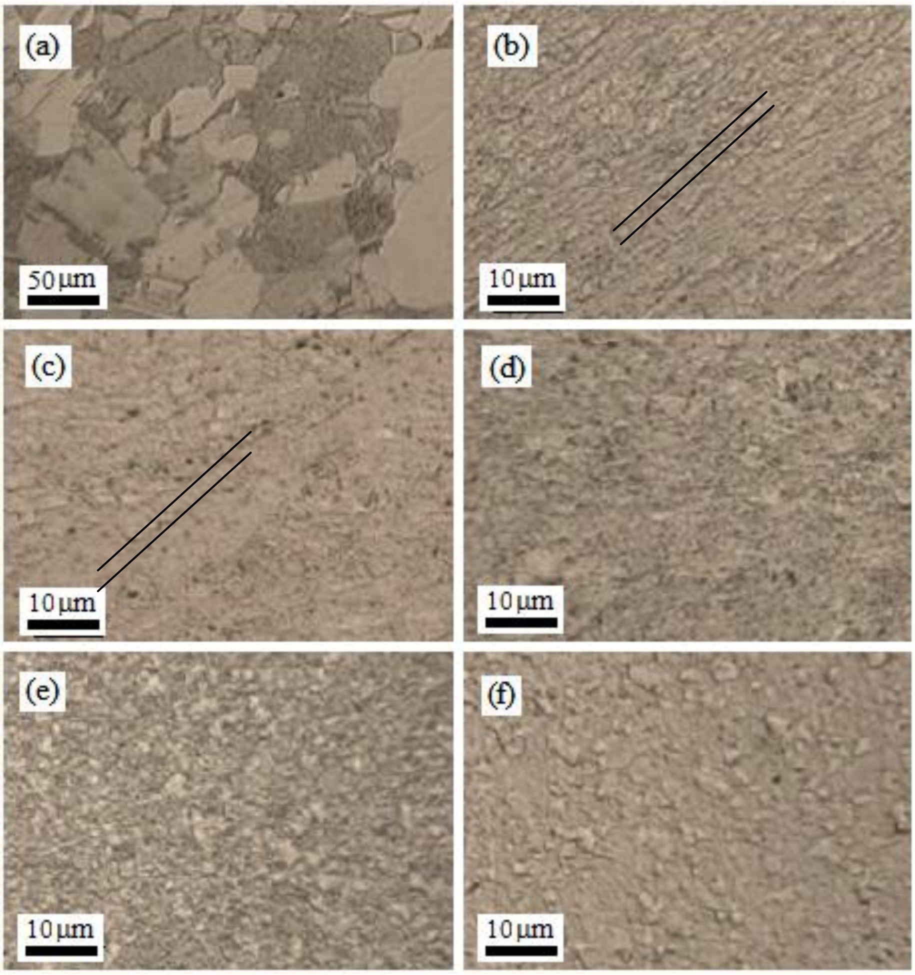

Figure 1 Optical micrograph of CuZn 70/30 alloy: (a) before ECAP; (b) after 4 passes ECAP, and after 4 passes ECAP followed by annealing at elevated temperature of: (c) 200°C; (d) 300°C; (e) 400°C; and (f) 500°C for 15 minutes

As Figure 1a shows, before ECAP, the samples were annealed at 600°C for 90 minutes to achieve more homogeneous structures. The initial microstructure was equi-axe with coarse grains and an average grain size of 45 µm, as estimated from a micrograph using the line-intersection method. This micrograph also showed twin grains in the annealed face center cubic (FCC) alloy. As Figure 1b shows, after four ECAP passes, the microstructure was significantly changed, showing parallel lines in the direction of the shear plane. ECAP with a 90° channel angle resulted in a shear plane with a 45° angle to the sample's cross-section, which is seen as a layered structure.

The parallel lines produced on the microstructures of ECAP samples were shear bands, formed by great strain ($\varepsilon > 0.8$) (Humpreys & Hatherly, 2004; Pasebani & Toroghinejad, 2010). These lines became more apparent with the cumulative strain of sequential ECAP passes. In previous studies, an ECAP pass with a strain equivalent to ε ~1.05 reduced the grain size to 25% (Azushima et al., 2008; Suryadi et al., 2013) and elongated it in the shear-band direction (Humpreys & Hatherly, 2004). In addition, increased strain was found to increase the density of the dislocation, which was directed perpendicular to the plane of shear, indicating the formation of cells or subgrains (Humpreys & Hatherly, 2004; Sakai et al., 2014). In the present study, after four ECAP passes, the strain was equivalent to true strain (ε) of 4.2 and the accumulated strain had formed a lamellar structure, with the thickness of the layers indicating a grain size. In addition, the four ECAP passes using route B_c produced UFG with equiaxed grains, high-angle grain boundaries (HAGb), and a size of ~0.8 µm, as observed using OM. These results are consistent with those of previous studies (Suryadi et al., 2013; Humpreys & Hatherly, 2004).

Pasebani and Toroghinejad (2010) reported that the microstructure of Cu-Zn 70/30 alloy deformed by six passes of ARB with a strain equivalent ~4.8 had a grain size of ~300 nm and a lamellar structure, as observed using TEM. In addition, Kumar et al. (2014) reported that brass alloy deformed by CR with a 90% reduction (ε = 2.4) resulted in a microstructure with a subgrain of 48 nm, as observed using TEM. Furthermore, Suryadi et al. (2013) reported that Cu-Zn 70/30 alloy produced by four ECAP passes using the B_c route had a more homogenous UFG microstructure, as seen in cross section. Finally, Bahadori et al. (2013) reported that copper deformed by four ECAP passes using the B_c route had a microstructure with a grain size of ~0.7 µm, as observed by both OM and TEM.

Figures 1c through 1f show the microstructure of brass after four ECAP passes and annealing at temperatures of 200, 300, 400, and 500°C, respectively, for 15 minutes. As Figure 1c shows, after annealing at 200°C, the microstructure did not differ significantly from that before annealing. It had a clear shear band and layered structure. As Figure 1d shows, after annealing at 300°C, the microstructure changed significantly. No shear band or layered structures were visible. Instead, new grains were seen in the microstructure, which had very fine-grained boundaries. The microstructure had recrystallized, which was marked by the emergence of new grains and the beginning of growth. The average grain size was 1.2 µm. As Figure 1e shows, after annealing at 400°C, the microstructure showed more growth, with grains of larger sizes. After annealing at 500°C, the microstructure consisted of even larger grains, of about 1.7 µm, and more obvious grain boundaries. It also showed the presence of twin grains. In this condition, the grain structure consisted of stacked subgrains with dislocations at the grain boundaries (Valiev et al., 2000; Humpreys & Hatherly, 2004).

These results showed that the microstructure changes began at the annealing temperature of 300°C and increased significantly at higher annealing temperatures. They also showed that the recrystallization temperature (T_R) of the UFG brass was below 0.5 of the melting temperature (T_M), which was in accordance with the results of Humpreys and Hatherly (2004), Kumar et al. (2015), and Ozgowicz et al. (2010). Kumar et al. (2015) reported that deformation using CR with 90% area reduction and annealing at 200°C to 300°C produced Cu-18Zn brass with a microstructure in which subgrain size had increased from 48 to 106 nm. This indicates that the microstructure changed significantly after annealing at temperatures from 200°C to 300°C. In addition, Ozgowicz et al. (2010) reported cold rolling with various reductions of thickness up to 70% on Cu-30Zn brass followed by recrystallization annealing at various temperatures up to

650°C, indicating that the microstructure of the alloy rolled by greater reduction of thickness can be changed significantly at lower annealing temperature.

3.2. Mechanical Properties

Shows the hardness-evaluation results of samples deformed by four ECAP passes and annealed at various temperatures. The hardness of the alloy increased significantly after four ECAP passes, from 74 HV to 248 HV. This means that the hardness of the alloy deformed by four ECAP passes was more than three times its initial hardness. The average hardnesses of the brass samples after annealing at 200, 300, 400, and 500°C were 248, 165, 135, and 122 HV, respectively. Sample hardness did not decrease significantly when annealed at 200°C, possibly because recrystallization occurred but was not followed by grain growth. In this condition, shear bands were still clearly seen. At an annealing temperature of 300°C, hardness decreased significantly but was still well above the initial hardness before ECAP. Figures 1d and 1e show recrystallization nucleation at the grain boundaries followed by grain growth. In these conditions, the shear-band structure was no longer seen, due to the movement caused by dislocations to the grainboundaries. At higher annealing temperatures, the hardness decreased less rapidly, reaching 122 HV at the annealing temperature of 500°C. These results are consistent with those of Kumar et al. (2015), who cryorolled Cu-18Zn with a 90% area reduction, which is a large strain. After CR, the alloy's hardness was 221 HV. After annealing at 300°C, the hardness decreased significantly, to 129 HV, but remained above its initial hardness of 54 HV. The present study's results are also in accordance with those of Zhao et al. (2014), who processed aluminum by multi-axial compression (MAC).

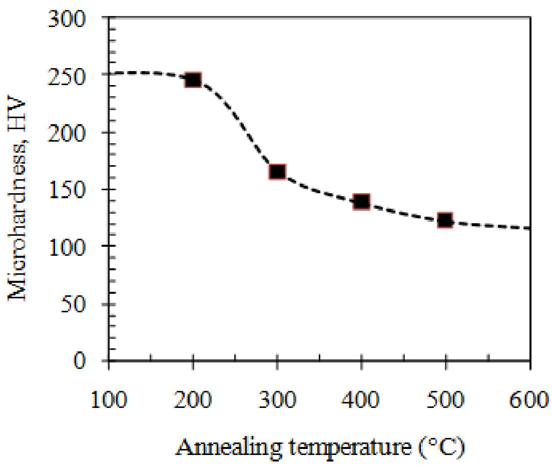

Figure 2 Average Vickers micro-hardness value of Cu-30%Zn alloy deformed by four ECAP passes and annealed at elevated temperatures

Figure 3 shows the results of tensile tests on samples of the brass alloy after four ECAP passes and annealing at various temperatures. The alloy's tensile strength (UTS) and yield strength (YS) increased significantly, from 316 and 212 MPa to 878 and 804 MPa, respectively, after four ECAP passes. The alloy's ductility (% E) decreased from 65% to 15% after four ECAP passes. The alloy's strength increased significantly due to grain refinement, a result that is in accordance with the Hall-Patch theory (Azushima et al., 2008; Valiev & Langdon, 2006). After annealing at 200°C, the UTS and YS decreased to 824 and 720 MPa, respectively. After annealing at 300°C, UTS and YS decreased to 510 MPa and 408 MPa, respectively. After annealing at 400°C, UTS and YS decreased to 450 and 325 MPa, respectively. And after annealing at 500°C, UTS and YS decreased to 435 and 320 MPa respectively. In addition, the elongation increased from 15% to 28% after annealing at 300°C. UTS decreased significantly at an annealing temperature of 300°C, but the YS was more than twice as great as the initial YS. Furthermore, ductility increased significantly at 300°C. These results are in accordance with those of several previous studies (Kumar et al., 2015; Humpreys & Hatherly, 2004; Sakai et al., 2014; Ozgowicz et al., 2010; Zhao et al., 2014), indicating that annealing after great deformation decreases strain hardening due to recrystallization and grain growth occurred at lower annealing temperature, so that great deformation reduced recrystallization temperature of metal.

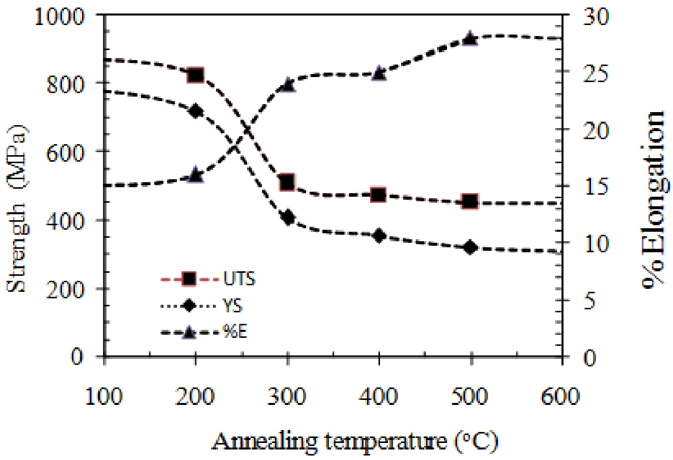

Figure 3 Tensile properties of Cu-30%Zn alloy deformed by four ECAP passes and annealed at elevated temperatures

After tensile testing, the fractured sample surfaces were examined using SEM, and the fractographs are shown in Figure 4.

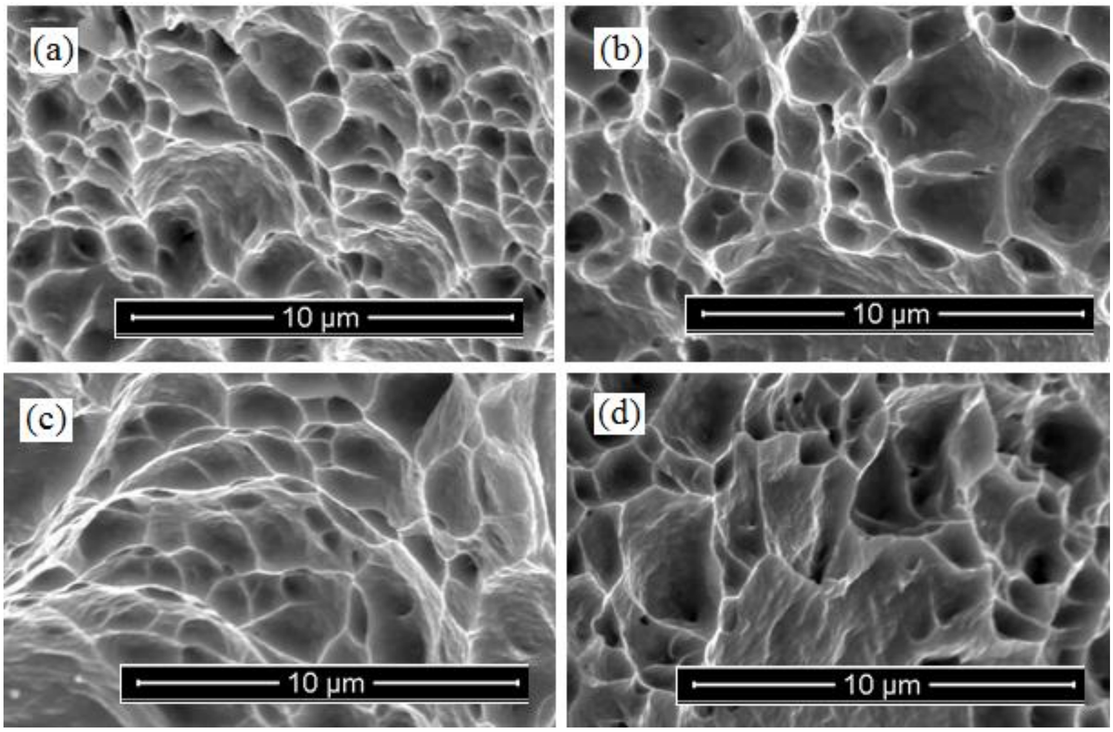

Figure 4 SEM fractographs of tensile samples of Cu-Zn 70/30: (a) deformed by four ECAP passes and annealed at: (b) 200°C; (c) 300°C; and (d) 400°C

Figure 4a shows the fractograph after ECAP. The structure showed very fine dimpling with dimple size reached 1 μm. The dimple size indicates grain size of the alloy. The shallow dimpling indicated that after four ECAP passes, the brass rod was more brittle but could still be categorized as ductile. After annealing at elevated temperatures, the dimpling grew larger, as shown in Figures 4b–4d. Increased annealing temperatures improved the alloy's ductility. The annealing temperature affected the UTS and YS of the UFG brass. The samples' fractured surfaces were in accordance with results reported by Ozgowicz et al. (2010), who deformed Cu-28Zn by CR with a 70% reduction in thickness and annealed it at elevated temperatures. The fracture results were also in accordance with brass deformed by CR and annealed for short times (Kumar et al., 2015).

4. CONCLUSION

UFG brass was prepared from commercial Cu-30Zn alloy processed by four ECAP passes using the Bc route and annealed at temperatures from 200–500°C for 15 minutes. Annealing at elevated temperatures influenced the mechanical properties and microstructure of the UFG brass. The results showed that alloy annealed at 300°C decreased in UTS and YS from 878 and 804 MPa, respectively, to 510 and 408 MPa, respectively. In addition, ductility increased to 28%, and hardness decreased to 165 HV. Furthermore, although the mechanical properties and microstructure did not significantly change at annealing temperatures of less than 300°C, the UFG brass annealed at 300°C still exhibited good performance, with a YS of 408 MPa, about twice that of un-annealed brass. Finally, the annealed UFG brass showed more and larger dimple ruptures, indicating increased ductility. These results indicated that the UFG brass produced by four ECAP passes was recrystallized at temperatures of less than 300°C.

5. ACKNOWLEDGEMENT

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