

# ENHANCEMENT OF MECHANICAL PROPERTIES AND GRAIN REFINEMENT IN ECAP 6/4 BRASS

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**Abstract.** This study evaluated the grain refinement and mechanical properties of 6/4 brass processed by equal channel angular pressing (ECAP). Increased passes in ECAP led to notable grain refinement, from 13  $\mu\text{m}$  in the initial sample to 300 nm after 4 passes. Grain refinement enhances mechanical properties, such as Vickers microhardness and tensile strength.

## 1. INTRODUCTION

Brass is widely used in many industrial products, such as lead frames, connectors, valves, pipes and so on, because of its excellent mechanical properties, including outstanding corrosion resistance, good formability, and suitable strength [1-3]. However, to improve the pressure tightness, strength, and machinability, lead has been used as an additive due to its low cost [2,4]. Recently, the use of lead in alloys has been strictly regulated due to its health effects; thus, lead-free brass is required to prevent health hazards. In addition, industry has been anticipating new applications for brass, specifically, electronic parts such as lead frames and connectors. Therefore, lead-free brass with optimized strength and conductivity, as well as minimized environmental and health concerns, is desirable.

ECAP is an effective method for enhanced grain refinement and mechanical properties [5-7]. Also, ECAP provides a versatility in the strain path, such as route A (refers to pressing the sample repetitively without any rotation),  $B_A$  (refers to rotation of  $90^\circ$  in the opposite sense),  $B_C$  (refers to rotation of  $90^\circ$  in the same sense) and C (refers to rotating by  $180^\circ$  between each pass), which allows a design of various interesting microstructural characteristics.

Routes  $B_C$  and C are more effective for refining grain size than routes A and  $B_A$  [8-10]. Route C is the most effective in complex-phase materials [11]. Therefore, in this study, ECAP via route C was used to produce lead-free 6/4 brass, and microstructural and mechanical properties were evaluated in terms of GBCDs.

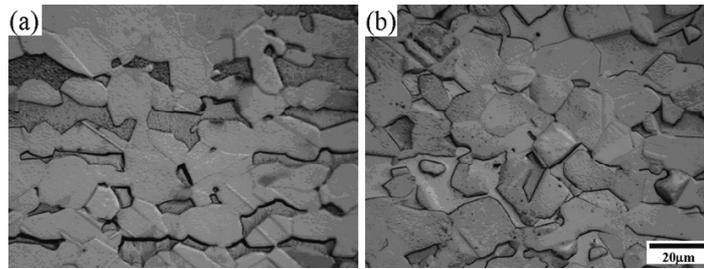
## 2. EXPERIMENTAL PROCEDURES

The material used in this work was commercial-grade 6/4 brass. For ECAP, 6/4 brass was prepared (10 mm in diameter and 100 mm in length), heat-treated at  $350^\circ\text{C}$  for 180 min, and used as an initial material. ECAP was carried out at  $250^\circ\text{C}$  via route C, which rotates the sample to  $180^\circ$  clockwise along its longitudinal axis and repeatedly processes it up to 4 passes. A closed die made of SKD61 with a cross-channel angle ( $\Phi$ ) of  $90^\circ$  between the vertical and horizontal channels and an outer corner angle ( $\Psi$ ) of  $20^\circ$  was used for ECAP. Samples were subsequently annealed at  $350^\circ\text{C}$  for 20 min to obtain recrystallized microstructures.

To investigate the microstructures, ECAP samples were machined to  $10\times 10$  mm, and a solution of  $\text{K}_2\text{Cr}_2\text{O}_7$  (2 g),  $\text{H}_2\text{O}$  (100 mL), and  $\text{H}_2\text{SO}_4$  (8 mL) was used to mechanically polish and etch the

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**Fig. 1.** Microstructures of (a) commercial 6/4 brass and (b) the initial material annealed at 350 °C for 180 min.

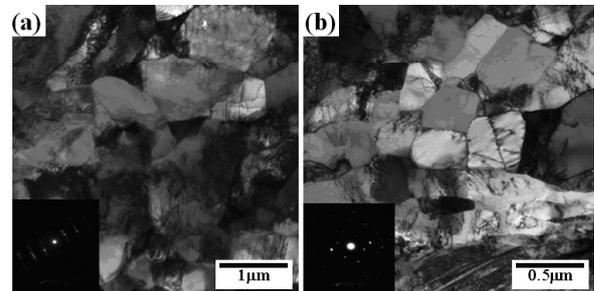
sample surfaces. EBSD analysis was used to investigate GBCDs on annealed materials, and samples were further polished by vibromet. EBSD data were acquired using a Hitachi 4300SE field-emission gun scanning electron microscope (FEG-SEM) operated at 20 kV. To investigate deformed microstructures, such as those resulting from 1 pass and 4 passes via route C, transmission electron microscopy (TEM) analysis was used. For TEM analysis, discs of 3 mm in diameter were mechanically polished to 80  $\mu\text{m}$  and then twin-jet polished using a solution of nitric acid (20 mL) and methanol (80 mL) under 8 V at -40 °C.

To evaluate the mechanical properties, Vickers microhardness and tensile tests were used. Vickers microhardness was carried out on cross sections of the materials with a load of 9.8 N and a dwell time of 15 s. Tensile test samples were used to evaluate the longitudinal tensile strength of the ECAP and recrystallized materials.

### 3. RESULTS AND DISCUSSION

The microstructures of commercial 6/4 brass and the ECAP initial material are shown in Fig. 1. Commercial 6/4 brass had a small amount of deformation, exhibiting elongated grains with an average grain size of 13  $\mu\text{m}$  in the  $\alpha$  phase and 10  $\mu\text{m}$  in the  $\beta'$  phase, as shown in Fig. 1a. The ECAP initial material was annealed at 350 °C for 180 min, removing elongated grains in the microstructure, as shown in Fig. 1b. The average grain size of the initial material was 13  $\mu\text{m}$  in the  $\alpha$  phase and 17  $\mu\text{m}$  in the  $\beta'$  phase, which was slightly smaller than the grain size in the  $\beta_2$  phase of commercial 6/4 brass.

TEM was used to identify the microstructures of ECAP materials. The sample that was pressed for 1 pass was composed of grains ranging from 500 nm to 1  $\mu\text{m}$ , including a large number of dislocations, as shown in Fig. 2a. The sample processed for 4 passes using route C had grains ranging from



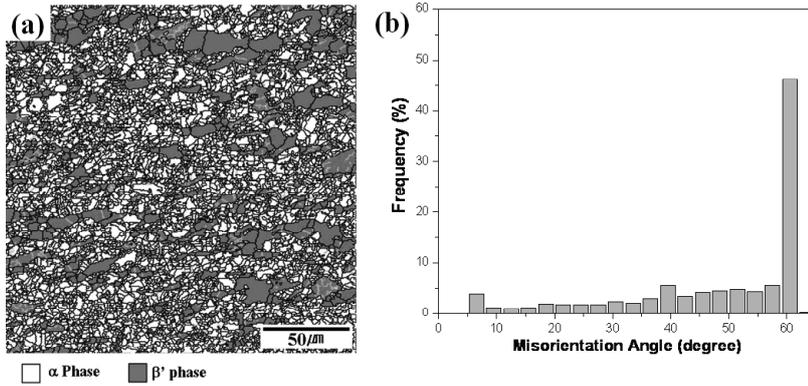
**Fig. 2.** TEM images of (a) 1- and (b) 4-pass materials.

100 nm to 500 nm in size; thus, it was more refined than the 1-pass sample, as shown in Fig. 2b.

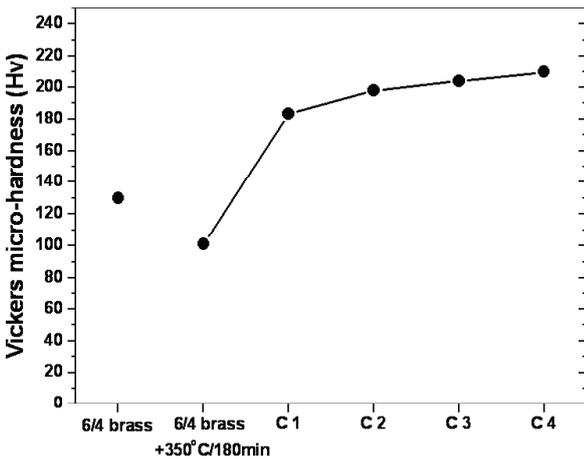
Fig. 3 shows phase maps and misorientation angle distributions, obtained by EBSD, for 4-pass ECAP samples that were subsequently recrystallized. The recrystallized material showed the equiaxed grain structure, consisting of grains with an average size of 2.4  $\mu\text{m}$  in the  $\alpha$  phase and 4  $\mu\text{m}$  in the  $\beta'$  phase, as shown in Fig. 3a. Also, the material was perfectly recrystallized, with the high-angle grain boundaries accounting for more than 92% of the grain structure, as shown in Fig. 3b.

The distribution of Vickers microhardness is shown in Fig. 4. Commercial 6/4 brass and the initial material had microhardnesses of 130 Hv and 101 Hv, respectively. However, Vickers microhardness was significantly increased by ECAP, which was accelerated with increased passes of ECAP. As a result, Vickers microhardness increased from 184 Hv for 1 pass to 210 Hv for 4 passes (Fig. 4).

Tensile properties are shown in Fig. 5. The tensile strength of the ECAP samples markedly increased compared to that of the initial material and was accelerated with increased passes of ECAP, similar to Vickers microhardness, as shown in Fig. 5a. The yield strength of the material processed by ECAP increased from 469 MPa for 1 pass to 544 MPa for 4 passes, which was an increase of nearly 340% compared to the initial material. In contrast, elongation decreased with increased passes of



**Fig. 3.** (a) EBSD phase map and (b) misorientation angle distribution of the ECAP and recrystallization heat-treated material. The green and black lines in the EBSD phase map correspond to low-angle grain boundaries and high-angle grain boundaries, respectively.



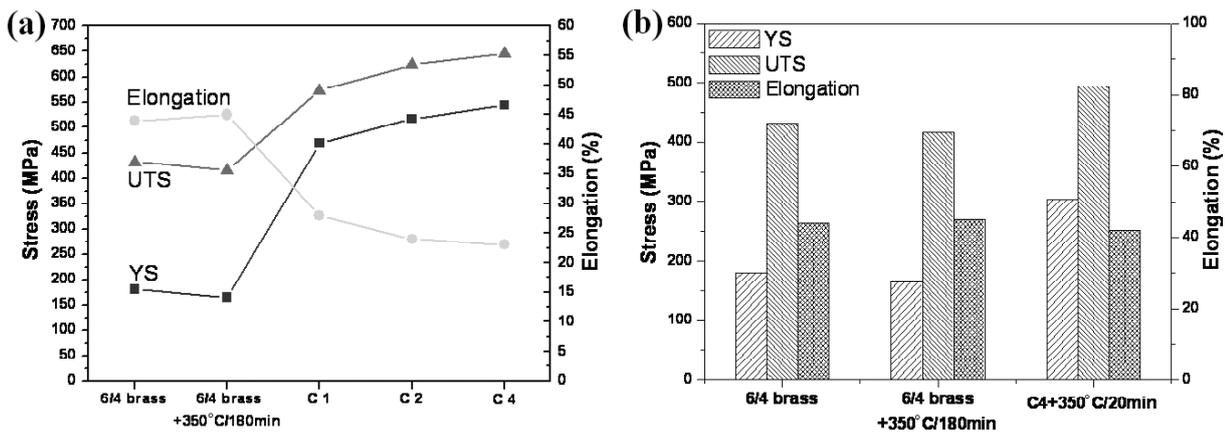
**Fig. 4.** Vickers microhardness profile during 4 passes of ECAP via route C.

ECAP. In addition, the material that was recrystallization heat-treated after ECAP showed increased tensile and yield strength compared to the initial material, with similar elongation, as shown in Fig. 5b. The yield strength significantly increased from

165 MPa for the initial material to 304 MPa for ECAP and annealed material.

ECAP at 250 °C on 6/4 brass effectively refined the grain size, which was accelerated with increased passes of ECAP. Initially, the grain size was 13  $\mu\text{m}$  in the  $\alpha$  phase (Fig. 1b); however, it was markedly refined from 1  $\mu\text{m}$  for 1 pass to 300 nm for 4 passes, as shown in Fig. 2. Generally, ECAP at low temperatures ( $\sim 0.5 T_m$ ) is more effective for achieving submicron grains due to an absence of dynamic recovery and recrystallization [12]. In this study, ECAP was carried out below  $0.5 T_m$ , forming submicron grains (300 nm) with no dynamic recovery or recrystallization due to an absence of annealing twins in the microstructures, and the deformed microstructure shown in Fig. 2 was maintained.

Grain refinement by ECAP directly depends on the pressing route, such as A, C, B<sub>A</sub>, and B<sub>C</sub>. In particular, routes B<sub>C</sub> and C are more effective for achieving refined and equiaxed grains than routes A and B<sub>A</sub> [8-10]. In this work, route C repeatedly elon-



**Fig. 5.** Tensile properties of ECAP and recrystallization heat-treated materials.

gated and restored the microstructure due to its deformation process that rotates the sample to 180° clockwise along its longitudinal axis, increasing the high-strain energy. The material (6/4 brass) used in this work has a lower stacking fault energy, which can achieve refined microstructures by discontinuous dynamic recrystallization (DDRX) during heat-treatment [13,14]. Consequently, higher strain energy by route C can effectively develop the microstructure, which resulted from the large number of recrystallization nuclei at high-strain energy sites by DDRX, along with formation of annealing twins. The material that was annealed at 350 °C after ECAP showed the microstructure developed by DDRX, as shown in Fig. 3.

ECAP effectively achieved sufficient mechanical properties, such as Vickers microhardness and tensile strength. As a result, Vickers microhardness, yield strength, and tensile strength in the 4-pass material increased by more than 100, 50, and 340%, respectively, compared to the initial material. Also, heat-treatment after ECAP effectively achieved sufficient yield and tensile strength with no decrease in elongation compared to the initial material. The yield strength increased 1.8 times compared to that of the initial material. This can be explained in terms of grain refinement. In other words, more refined grains due to ECAP led to increased hardness and strength. Therefore, grain refinement by ECAP and recrystallization heat-treatment can achieve desirable mechanical properties.

#### 4. CONCLUSIONS

ECAP was successfully applied to 6/4 brass, increasing the grain refinement and mechanical properties. As a result, the grain size was significantly refined to 300 nm during 4 passes, which affected the mechanical properties, such as Vickers microhardness, yield strength, and tensile strength. The yield strength of the 4-pass material increased 340% compared to that of the initial material. Furthermore, the material that was recrystallization heat-treated after ECAP also showed increased yield strength compared to that of the initial material, with

nearly no decrease in elongation. Therefore, ECAP applied to 6/4 brass can effectively increase the grain refinement and mechanical properties.

#### ACKNOWLEDGEMENT

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