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Effect of Equal Channel Angular Pressing and Post Heating on Microstructure and Hardness of Cu-Zn 70/30

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Abstract. Severe plastic deformation (SPD) using various pass number of Equal Channel Angular Pressing (ECAP) experiment and followed heating at 400°C has been done for rod brass Cu-Zn 70/30 to investigate the operation on microstructure and hardness of the alloy. Optical microscopy and SEM are used to examine the microstructure change. Mechanical testing such as macro and micro hardness test is used in order to examine the change of mechanical properties. The grain structure of the alloy was refined from 34 µm to 2 µm after 4 passes ECAP and increased to 4 µm after post heating. The hardness of the alloy significantly increased from 78 Hv to 235 Hv after 4 passes and decreased to 135 Hv after post heating after ECAP. The microstructure and mechanical properties of the alloy was homogenous after 4 passes ECAP because the strain was found more homogenous.

Introduction

The grain size and homogeneity of microstructure influence on formability of sheet metal [1-2]. Refined grain increases tensile strength of metal and decreases weight to strength ratio.

There are many studies on the formation of sub grain and fine grain size to less than 1 µm (ultra fine grain) which explain the formation of fine grain. Some of them are severe plastic deformation method that had been done as accumulative roll bounding (ARB) [2, 3], high pressure torsion (HPT) [2,4], rolling at cryogenic temperature [5], and equal channel angular pressing (ECAP)[2, 5-8]. Other researchers provide combination of several process as HTP and cold rolling (CR) [4, 9], ECAP and post heating [7]. All of the studies focuse on obtaining fine grain and better structure.

ECAP is one of the SPD processes that have considerable potential to produce material with fine structure in a solid condition. In this process the rod metal can be deformed repeatedly without changing the cross-sectional area of rod (cylindrical or rectangular rod) [2]. The rod is deformed using ECAP die with two rounds or rectangular channels intersection with angle ϕ and curvature angle ψ [2, 8]. Effective strain acting on the ECAP process is a function of ϕ and ψ as expressed in Equation (1) [2, 8] is

$$\varepsilon = \frac{n}{\sqrt{3}}\left(2\cot\left(\frac{\phi}{2}+\frac{\psi}{2}\right)+\psi 2\cos ec\left(\frac{\phi}{2}+\frac{\psi}{2}\right)\right) \quad (1)$$

Where ϕ is the angle of intersection of two channels and called as "die corner angle", ψ is the angle subtended by the arc of curvature at the point of intersection or die curvature angle, and n is the number of pass ECAP. According to Equation (1), equivalent or effective strain maximum of 1.15 at $\psi=0^o$ and a minimum of 0,907 at $\psi = 90^o$ while the die corner angle is fixed at $\phi = 90$. The ECAP die with $\psi = 20^o$ and $\phi = 90$ is commonly used by several study [1, 7, 8]. Homogeneity of structure ECAP is determined by route used. There are for basic route used in ECAP, they are A, B_A, B_C and C. It is clearly explained in Reference [2].

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Saviraman and Uday Chakkingal [10] reported their ECAP experiment on pure aluminum. The tensile strength of the annealed aluminum was increased from 58 MPa to 144 MPa after ECAP 1 pass. The grain size of the aluminum was decreased to 0,6 μm after ECAP 1 pass. Pasebani *et. al* [3] reported on ARB experiment of Cu-Zn 70/30 that the hardness of the alloy was increased to three times greater than the received material after 1 pass ARB and the small recrystallized grains with average grain size of below 100 nm appeared after 6 cycles of ARB on the alloy.

The objectives of the research are studying the mechanism of grain refinement of brass Cu-Zn 70/30 by SPD process using ECAP, observe and analyze the effect of process variables on the microstructure and mechanical properties.

Material and Experimental Procedure

The material used in this study is Cu-Zn 70/30 brass plate with chemical composition shown in Table 1.

Table 1. Chemical composition of the brass sheet Cu-Zn 70/30 (%wt.)

Cu	Zn	Fe	Sn	Al	Ni	Pb	Si	Mn
70,37	29,48	0,052	-	0,024	0,030	0,023	0,005	0,009

The rectangular samples 13 mm x12 mm x 250 mm dimensions were cut from the plate in the rolling direction. The samples of brass rods with 10 mm diameter and 80 mm of length were machined from the rectangular samples. The samples were subjected to homogenization annealing at 650° C for 90 minutes to get homogenous of microstructure with 35 μm grain sizes. After annealed, multi pass deformation up to 4 passes were done by the ECAP die with two round channels intersection at 90^o and curvature angle of 20^o at room temperature via Bc route [2, 7]. The deformed alloys then heated at 400° C for 15 minutes.

The pressing of ECAP performed by using the Schenk Trebel machine with pressure capacity 1000 kN and speed at 5 mm/minute. The microstructure change was investigated from metallographic specimens that were taken from longitudinal direction and transverse direction using Litz Metalloplan Microscopy and Phillips Scanning Electron Microscopy. The micro hardness was tested on metallographic section, using Micro Vickers Hardness Tester at 300 grams load for 15 sec.

Results and Discussion

The Microstructure of brass after annealing at 600 ° C for 90 minutes can be seen in Fig. 1 (a). The grain structures were uniform with average size of 34 μm. It is also appeared that some grains have parallel lines indicate some twin crystal. This is the effect of heating at long time and at a temperature above the temperature of recrystallization. After first pass ECAP, the microstructure was changed. This can be illustrated in Fig. 1 (b) and (c). The structure shaped is equiaxed before turning. After the turn of ECAP, the structure was elongated unidirectional plane of shear on the ECAP process [2]. In the elongated structures the parallel lines form 45° angle to the shear plane. Parallel lines seen in the direction of the plane of shear was shear band. The shear band are clearer and most dense at higher number of ECAP passed, see in Figure 1 (d) and (e). Reference [3, 4, 9] explain the formation of the lines as shear band on the brass 70/30 that treated by ARB and HPT. Figure 2 displays the results of SEM photograph of brass before ECAP, after ECAP and after post heating. The grain boundaries are evident in the microstructure of brass before ECAP. Grain boundary indicated by the change in direction of the crystal orientation [1, 2], as shown in Fig. 2 (d). Meanwhile, on brass that has been done ECAP 3 passes, the grain boundaries are difficult to observe, but the structure was seen with disconnected orientation. According to reference [1, 2] this phenomenon is due to which dislocations which is increased with increasing number of pass (Fig. 2 (b) and (e)). On the brass heated up, it was seen more uniform structure and spread evenly (Figure 2 (c) and (f)).

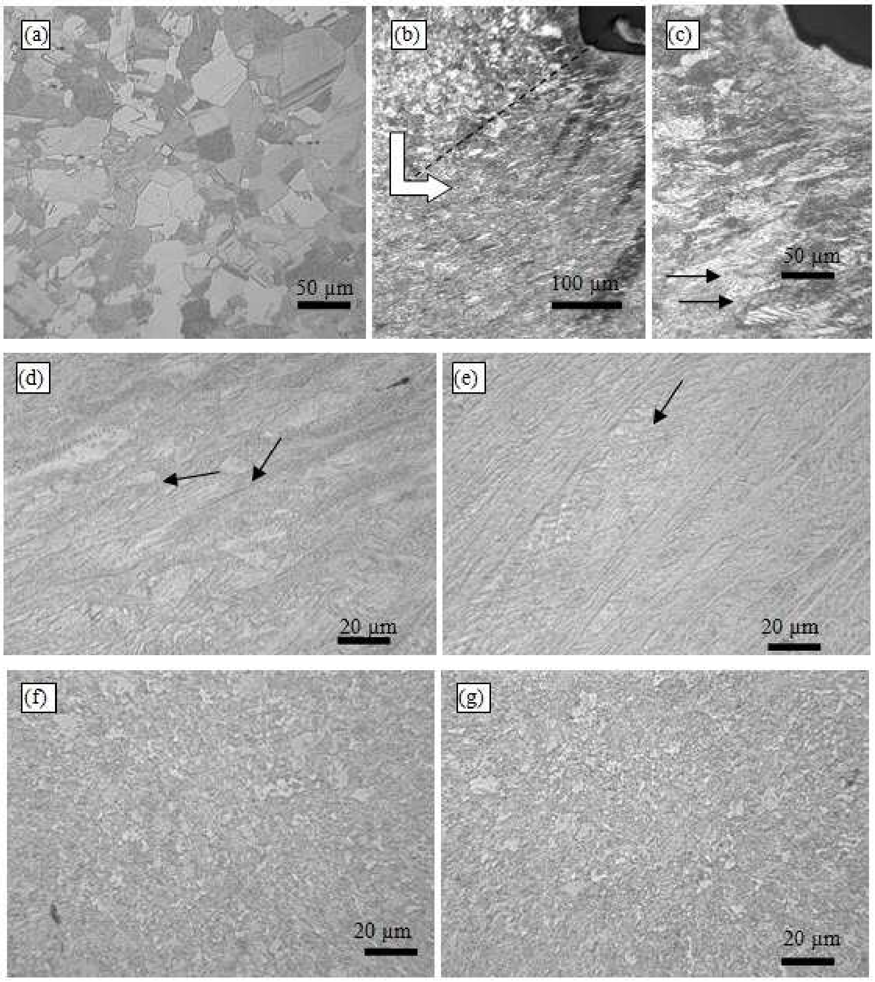

Fig. 1 Optical micrographs of Cu-Zn 70/30 (a) before ECAP (after annealing at 600^{o} C for 90 minutes), after ECAP (b) 1 pass at longitudinal section area with 100x magnification and (c) with 200x magnification, (d) 2 passes, (e) 4 passes, (f) 2 passes + post heating at 400 ^{o} C for 15 minutes, and (g) 3 passes + post heating at 400^{o} C for 15 minutes. White arrows indicate the direction of ECAP. Black arrows indicate microstructure changes in shear plane.

The results of measurement of average grain size of brass 70/30 after ECAP and post heating was shown in Figure 3. Grain size was decreased from 34 µm to 8 µm after 1 pass ECAP and to 2 µm after 4 passes ECAP. Substantial grain refinement occurs after 1 pass ECAP, where grains elongated and flattened. After subsequent pass the grain size decreases slowly. This is due to the slowdown of strain hardening. Deformation increasingly pressed within shear bands. Further heating causes an increase in the grain size of the grain size after ECAP. After post heating is seen the new small grain through recrystallization with shear bands that are no longer visible.

Post heating after ECAP put up the microstructure of alloy was more uniform (Figure. 1 (g) and (h)). It can be seen that the post heating up after ECAP returning the orientation of grain structure becomes equiaxed, with larger grain size and shear bands was disappeared. Post heating causes recrystallization reached the stage of grain growth. W. Ozgowicz et. al. in [11] reported that the brass 70/30 after cold rolling with a reduction of 56.2% (ε = 0.83) was heated up to 400^{o} C recrystallization occurs that produces fine grain with grain size up to 7 µm. Heat up temperature take the same visible difference in the growth of the grain size after ECAP with different number of pass. For a larger number of pass, the deformation energy received by metal increase so the heat energy will produce more recrystallization [1, 2].

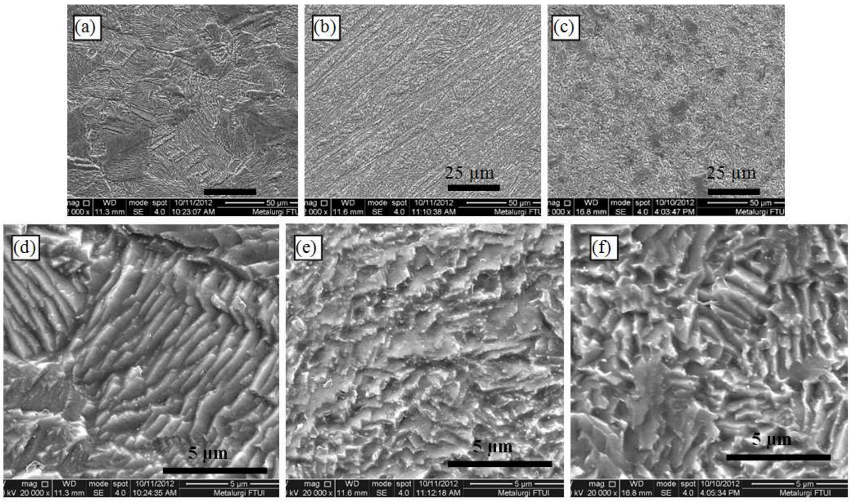

Fig. 2 SEM Micrographs of Cu-Zn 70/30 (a) before ECAP, after ECAP (b) 3 passes, (c) passes +post heating at 400° C for 15 minutes with a 2.000x magnification, and (d) before ECAP, after ECAP (e) 3 passes, (f) 3 passes +post heating at 400° C for 15 minutes with 20.000 x magnification.

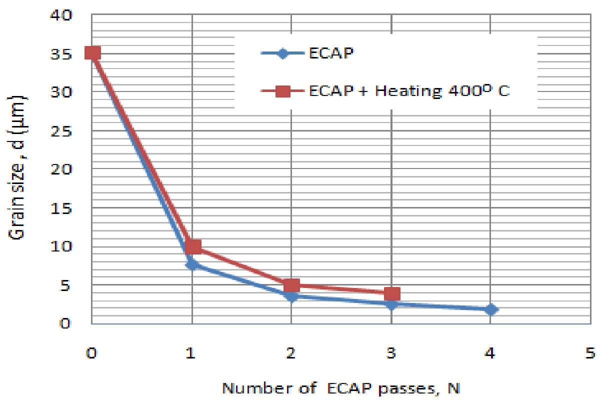

Fig. 3 The grain size of Cu-Zn 70/30 after deformed by ECAP with several passes and post heating.

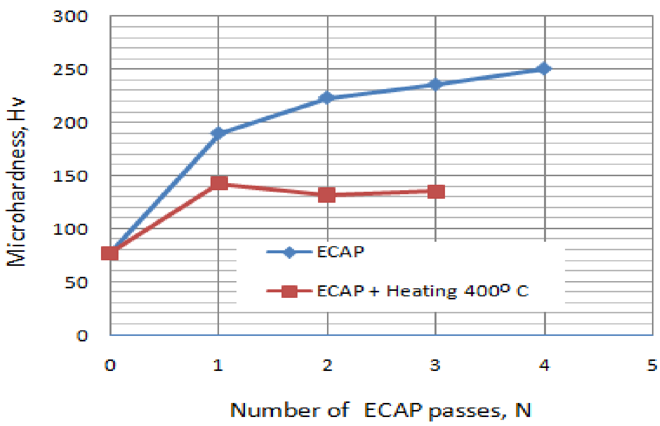

Fig. 4 Microhardness Vickers of Cu-Zn 70/30 after deformed by ECAP with several passes and post heating.

The ECAP samples hardness was measured at longitudinal and transverse section area for unheated up and heated up samples, and the average measurement results are shown in Figure 4. For samples that were unheated after ECAP, the hardness increases incredible after ECAP 4 passes, three times greater than the initial value. The hardness increased sharply after the first pass and then saturated at the next pas. Hardness of brass early 78 Hv to 235 Hv after ECAP 4 passes. The largest enhancement of hardness occurred after the effective strain that worked at first pass was 1,05. The increased of hardness rapidly at first strain appears to be associated with the same strain hardening with increasing strength. Grain refinement on a larger strain does not cause increase hardness significantly. Several studies on UFG materials produced by SPD exhibit the hardness behavior showed saturation at large strains [2,3,5]. The strain hardening during plastic deformation continues to decline according to the Hall-Petch relationship with grain size reduction at higher cycles, the hardness will be increased [1, 2, 3].

As for the brass 70/30 which is heated up to a temperature of 400 ° C for 15 minutes after ECAP showed that the hardness was decreased after ECAP 1 pass but it still well above the initial hardness. The hardness after heating up after the first ECAP pass is 153 Hv. For the samples heated up after second pass ECAP the hardness down to 135 Hv, and slightly up for the next pass. This was due to the process of recrystallization and grain size increased. Recrystallzation would be more easily occur for larger strain so the decline of hardness will be greater. The hardness difference between the non heating and after heating up the ECAP process gets worse with the growing strain ECAP. It is also shown in various experiments with large strain [6, 9].

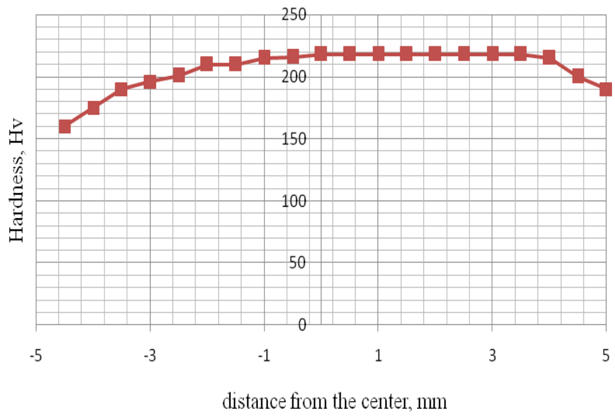

Figure 5: The hardness distribution of the rod Cu-Zn 70/30 at cross section area after 1 pass ECAP.

The Measurement of hardness in cross-sectional and longitudinal after 1 pas ECAP shows the hardness at the center of specimen higher than the edges, as shown in Figure 5. It can be explained that the difference hardness in the region due to differences strain received by the area. Reference [6, 8] conducted finite element simulation to see the strain distribution of ECAP specimen. From the simulation explained that the region was the center of the large strain 1, while at the bottom is reduced to 0,55. By using route Bc, the cumulative strain is expected to be equally at a distance of up to 4 mm from the x-axis, as explained in Figure 6 below. The maximum strain after 4 will pass evenly in a centralized area in the middle section ECAP samples. With a more uniform strain distribution in the middle will result in violence and a more homogeneous structure on the trunk ECAP using B_C route.

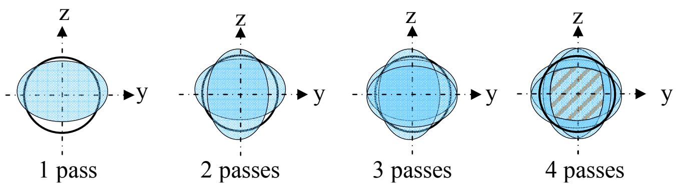

Fig. 6. The maximum strain distribution at each pass ECAP up to 4 passes using route B_C. The maximum strain fields marked with ellipse. At 4 passes ECAP the strain is more centrally distributed into the center of rod (the shaded portion).

Conclusion

ECAP process up to four passes on the Cu-Zn 70/30 brass significantly reduces the grain size and increases the hardness. The grain size of 34 µm decreases to about 2 µm after 4 passes. Grain refinement is caused by the sliding mechanism that occurs by the formation of shear bands followed by increasingly dislocation density. Hardness of the alloy significantly increases after 1 pass from 78 Hv to become 190 Hv and 235 Hv after 4 passes ECAP.

Heating after ECAP on CuZn 70/30 at 400 ° C for 15 minutes slightly increases the grain size and produces a grain which is more uniform with equiaxe shape and the hardness decreases become 153 Hv after 1 pass ECAP and 135 Hv after 3 passes ECAP.

The accumulation of a homogeneous strain produces homogeneity microstructure and mechanical properties. The strain is more homogeneous using 4 passes ECAP via B_C route or every four passes.

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