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Influence of ECAP Parameters on Electrical Conductivity and Hardness of Pure Cu

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ABSTRACT

In this study, the ECAP pprocess was conducted using two different dies of channel angles (Φ) 120° and 90° to extrude pure Cu for 2 and 6 passes of route Bc at room temperature. Optical Microscopy (OM) was used to study the microstructure of Cu before and after ECAP processing. Vickers's microhardness was measured along the transvers section of the Cu billets. The electrical conductivity of the Cu billets was measured at room temperature and expressed as a relative percentage of the international annealed copper standard. 2-passes using the two dies revealed an elongated ultrafine-grained structure that aligned parallel to the extrusion direction. 6 passes using the 90°-die resulted in more ultrafine-grained equiaxed structure compared to the Cu billets processed through the 120°-die. Processing through 6-passes revealed a significant increase in the Cu Vickers's microhardness by 56% and 72% through processing using the using the 120°-die and 90°-die, respectively when it put in comparison with the as-annealed samples. The electrical conductivity finding revealed that ECAP processing up to 6-passes resulted in insignificant decrease of 6.6% compared to the as-annealed counterpart which indicated that ECAP processing can strengthen the Cu billets without losing its electrical conductivity

Keywords: ECAP-Die 90-Die120

1. INTRODUCTION

One of the famous and widely non-ferrous metals which have been closely associated to human beings is copper. Not only for there have opulent resources in environment, but copper also possesses excellent electrical and thermal properties. Therefore, copper is widely used in the fields of electrical, electronics, and energy. Copper is used in its pure form or alloyed with other metals as the need for increased levels of strength and other mechanical properties as hardness and wear [1, 2]. As a corrosion resistant and durable metal, copper is capable of withstanding up to 1,000 psi of pressure [3] it is also lightweight, which reduces the manufacturing time and cost, and also easier to extend over long stretches without supports [4]. Although copper costs less than steel, it costs more than plastic as plastic pipes can bend to fit into almost any space necessary [1, 4]. The main disadvantage of using copper is the low strength and wear resistance compared to other metals as it oxidizes, episodically at high temperatures [4], and encounter problems from water acidity [3]. For example, applications depending on shock hazards, fibre optical cables have a lower shock hazard than copper wire [5]. Copper is susceptible to a great degree of electrical interference, leading to a less clear signal than fiber optics, as it is vulnerable to electromagnetic interference, potentially resulting in some devices working improperly [5]. Enhanced electrical conductivity and mechanical properties for copper has been reported using alloying elements [2, 6]. Various reinforcing materials are added to copper matrix to enhance the strength and wear resistant of the copper alloys [2, 8]. Copper alloys properties can be also enhanced using heat treatment on precipitate alloy composition [8, 9]. As gradual incorporation of copper in the precipitates during the heat treatment is essentially related to the slower diffusivity resulting in better strength of the copper alloys [9].

Subsequently, experimental severe plastic deformation (SPD) techniques could lead to improvements in mechanical and electrical properties [10,11]. The microstructure of the annealed copper alloys subjected to equal-channel angular pressing (ECAP) producing nano-crystalline (NC) structure. The results show that the heat treatment and the hard cyclic deformation in the viscoplastic regime influence the properties of nano-crystalline copper alloys profoundly [10] in case of strength and hardness measurements. Similarly, copper alloys ECAPed deformed at room temperature and at 200°C for up to 4-passes improved the crystallographic alloy texture and hardness homogeneity [11]. Also, multi-channel spiral twist extrusion (MCSTE) as one of the severe plastic deformations (SPD) techniques was used for producing superior mechanical properties associated with grained microstructure in bulk metals and alloys [12].

The strength and hardness of the metallic alloys increased with the increase of the number of MCSTE passes at a relatively low twisting angle of 30 degrees coupled with retention of ductility [12] as well as structural uniformity increased of the metallic alloys. Similarly, significant improvements of the corrosion behavior and mechanical properties of biodegradable alloys using ECAP was reported [13].

ECAP is a promising candidate for further development of different industrial applications [14-16]. ECAP die consists of two channels with the same cross-section intersecting at channel angle Φ, and with curvature angle Ψ [17]. In addition, the ECAP processing route type also has a significant effect on both the microstructural evolution and mechanical properties of the ECAPed billets [18]. The common ECAP route types are A, Bc, and C. The processed sample is not rotated at all between the subsequent passes, whereas in routes Bc and C the sample is rotated 90° and 180°, respectively between subsequent passes [19]. The equivalent strain

$$\varepsilon_{eq} = \frac{N}{\sqrt{3}}\left[2\cot\left(\frac{\varphi+\psi}{2}\right) + \psi\,cosec\left(\frac{\varphi+\psi}{2}\right)\right] \qquad (1)$$

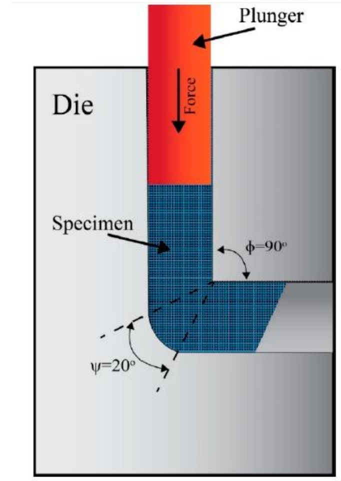
(ε_{eq}) during ECAP processing in terms of the number of passes (N) can be calculated from the following Eq. 1 [19].

Zhao et al. [20] studied the deformation behaviour and dislocation's formation mechanism of nanostructure in metallic alloys subjected to ECAP as the influences of different routes on generated strain and microstructural changes. As it was concluded that one-pass of ECAP is not enough the create a uniform microstructural alloy, and as increasing the number of routes the strain homogeneity increase. studied grain size refining after each pass and its influence on the strength of samples. It was reported that SPD refines grain size which leads to the enhancement of ductility in both hard and brittle materials [13, 20]. Also, the channel angle (φ) and the number of passes through the ECAP process plays a significant rule in enhancing the metallic alloys hardness [11] especially in brittle metallic alloys. Significant decrease in grain size was observed in pure copper subjected to severe plastic deformation using ECAP, high pressure torsion (HPT), and a combination of both [20]. Also, micro tensile and microhardness measurements of the three processing materials have been reported to noteworthy improve [21]. Higuera-Cobos et. al. [22] reported rises in the stored energy for pure copper with the increasing of ECAP passes deformation (increasing number of passes up to 16 times), as the recrystallization of pure cupper at room temperature decreases. Also, microstructural and mechanical tensile measurements display stable microstructure after four passes, whereas, electrical conductivity decreases with increasing ECAP passes [22, 23]. Due to being the most significant parameter that influence the characteristics of the ECAP processed materials, further investigation on the effect of φ on both the mechanical and electrical behaviour of commercial pure Cu billets. Accordingly, this study aims to examine and describe the connection between processing parameters such as the ECAP die's channel angle and the number of processing passes, and pure Cu properties such as the microstructural evolution, Vickers' microhardness and electrical conductivity.

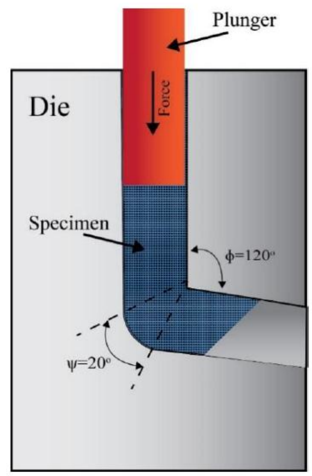
2. Materials and Experimental Procedure

Billets of a commercial pure Cu with a chemical composition of (0.5% Zn, 0.46% Si, traces elements of Sn, Mn, Al, Fe, and the balance is Cu, wt%) were received as rolled rods 20 mm in diameter and 50 cm length. The as-received (AA) rods were machined to form the ECAP billets with 20 mm diameter and 60 mm length. The Cu specimen was annealed at 400 °C for 2 h and then cooled in the furnace. The as-annealed (AA) Cu billets were processed through ECAP die for 2 and 6-passes of route Bc using two different channel angles (Φ) of 120° and 90° and an outer corner angle of ψ = 20° (shown in Figure 1). From Eq. (1), the previous processing parameters generated equivalent strains of 1.054 and 0.634 per pass, for the 90° and 120° channel angles, respectively. ECAP processing was carried out at room temperature (RT) with a crosshead speed of 0.05 mm/s. Finally, to reduce the friction graphite lubricant was used during processing. Microstructural examination of the Cu samples was carried out using OM. Cu billets were mounted, ground using 600, 800,1000, and 1200 grit silicon carbide sandpaper, polished to a mirror-like finish using alumina solution, and finally etched using a mixture of HCl and HNO_3 solutions with a volume ratio of 3:1 as a final preparation step. Vickers's microhardness values (HV) were measured after a load of 1 kg was impressed on the specimen for 15 s. 5-equispaced indentations were measured and the displayed results were the average of the recorded values.

The electrical conductivity of the Cu billets was measured at room temperature on AG 4311B RLC-meter (Ando, Japan) at zero frequency. A thin circular disk of 20 mm diameter and 1 mm thickness was prepared for the Cu billets. Four Cu wires were stretched across the opening of a Teflon (or other non-conductive plastic block), glued in place, parallel, and with precisely-known separation (gage length) between the two inner wires. The Cu billets surfaces were polished before testing to clean the surface. The electrical conductivity was expressed as a relative percentage of the international annealed copper standard (%IACS).



(a)



(b)

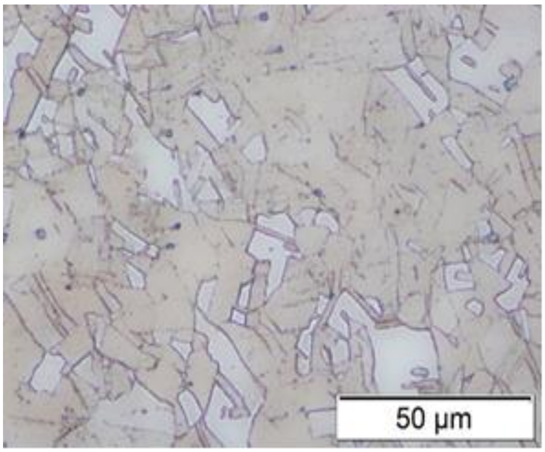
Figure 1: ECAP die drawing. a) 90º channel angle, b) 120º channel angle.

3. Results and Discussion

3.1 Microstructural Evolution

Figure 2 shows the OM micrographs of AA and ECAPed Cu billets processed through different processing parameters of ECAP. The AA billets revealed relatively coarse grains which had an almost irregular shape and different grain sizes ranging from relatively fine grains to significantly coarse grains as shown in Figure 2a. In addtion, from Figure 2a it was clear that the annealing process resulted in disappearing the great majority of the grain boundaries. Processing through 2-Bc using the 90°- die revealed a relatively ultrafine-grained structure (UFG) which aligned parallel to the extrusion direction (ED) as shown in Figure 2b.

Accumulation of the plastic strain up to 6-Bc revealed a significant grain refining of the Cu samples where the UFG equiaxed structure and completely recrystallized fine grains dominated the Cu structure as shown in Figure 2c. To characterize the pure Cu microstructure and its relatedness to the channel angle used, the billets were processed through 2-Bc and 6-Bc using the 120°- die at RT and compared with the OM micrographs of the 90°-die counterparts.



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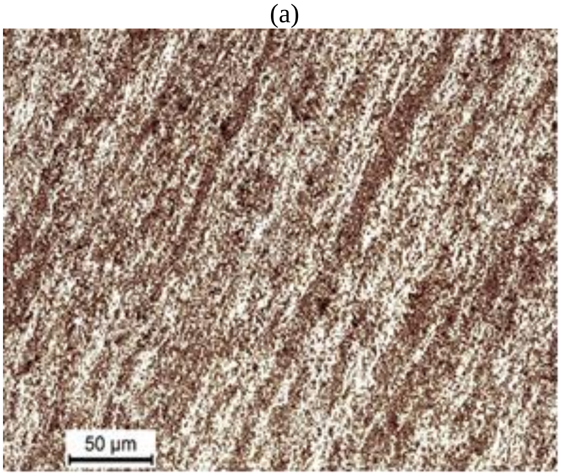

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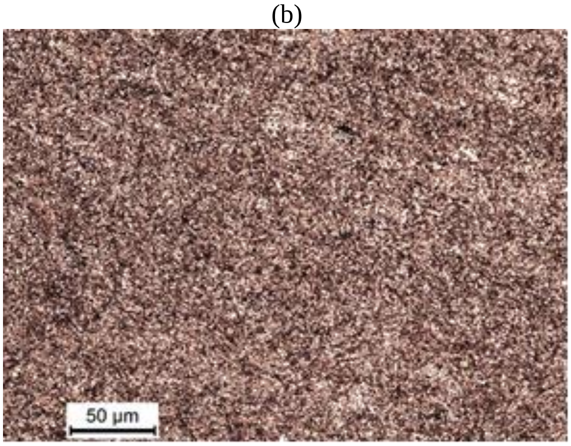

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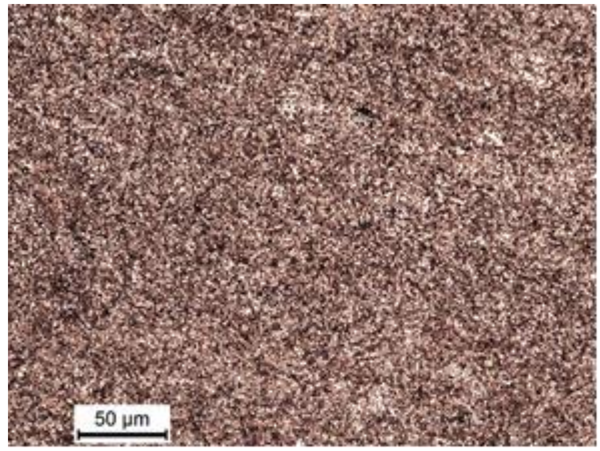

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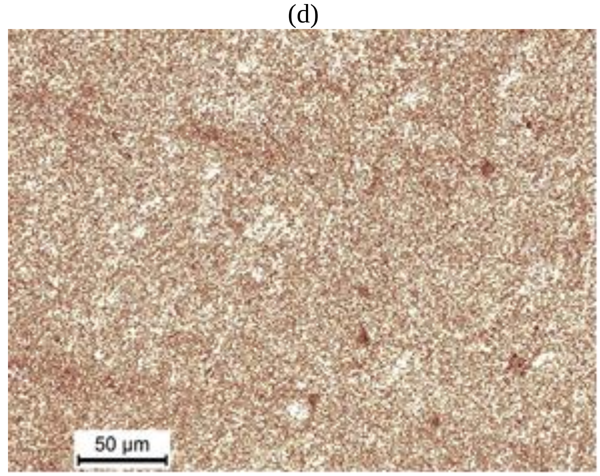

(e)

Figure 2: OM micrographs of the AA (a) Cu billets and ECAPEed billets processed through 2-Bc (b), 6-Bc (c) using the 90°- die, 2-Bc (d), 6-Bc (e) using the 120°- die.

Similar to the 90°- die, processing through 2-Bc using the 120°- die at RT exhibited UFG structure which aligned parallel to the ED as shown in Figure 2d. In addition, the aligned grains (Figure 2d) exhibited a lower orientation angle on the shear direction compared to 2-Bc counterparts processed using the 90°- die which due to the lower imposed shear strain resulted from the ECAP die of 120°- die compared to the ECAP die of 90° according to equation (1). Furthermore, 6-Bc processing using the 120°- die exhibited an UFG structure despite of being relatively coarser compared to the microstructure of 6-Bc using the 90°- die as shown in Figure 2e. From Figures 2b, and 2d it was clear that 2-Bc through both ECAP dies with internal channel angle of 90° and 120° displayed non homogenous distribution of the grain size where fairly elongated fine grains co-exist with dynamic recrystallized (DRX) ultrafine grains. This findings indicated that ECAP processing through multiple passess will improve the homogeneity of the material's grain size distribution [24]]. Furthermore, Processing through 6-Bc through both 90°-die and 120°-die exhibited a fully DRXed UFG as shown in Figures 2c and 2e. During the early ECAP processing passes, a huge number of dislocation was generated and multiplicated then it tangled with each other and then arranged to form low-angle grain boundaries (LAGBs) [25].

Furthermore, ECAP processing through multiple passes leads to occurring the recrystallization process and the LAGBs transformed into high-angle grain boundaries (HAGBs) with UFG structure (as shown in Figure 2c, and 2e) which indicted that the completion of the DRX process [26]. Increasing the fraction of HAGBs after multiple passes leads to strengthening the ECAPed billets due to hindering and blocking the dislocation movement [27-28].

3.2 Vicker's Hardness

Figure 3 shows Vicker's microhardness of the AA and ECAPed Cu billets processed using both the 90°-die and the 120°-die. The AA billets exhibited an almost constant Vicker's micrhardness (HV) value of 100 along the billet's transverse section from the central area to the peripheral regions. The ECAP die with internal channel angle of 90° displayed improved hardness than Cu billets processed using the ECAP die with internal channel angle of 120° due to the higher imposed shear strain of the 90°-die. From Eq. (1), the used processing parameters resulted in equivalent strains of 1.054 and 0.634 per pass for the 90° and 120° channel angles, respectively. Processing through 2-Bc through the ECAP die with internal channel angle of 90° led to improving the HV by 38% compared to the AA counterpart. Further increasing the plastic strain up to 6-Bc revealed addition increase in the HV by 72% compared to the AA condition (Figure3). In addition, ECAP processing through 2-Bc and 6-Bc using the 120°-die exhibited increasing the HV values of the Cu billets by 28% and 56%, respectively when it put in comparison with the AA samples. Increasing the plastic strain through increasing the ECAP passes resulted in the formation and multiplication of dislocations which resulted in transferring the LAGBs into HAGBs and hence, UFG structure was attained (as shown in Fig. 2) which agreed with earlier studies [11, 15, 19, 24]. Furthermore, the UFG structure obtained after ECAP processing through multiple passes plays a vital role in blocking the dislocation motion and strengthening the Cu billets [17-19]. Accordingly, the grain refining mechanism is the most effective strengthening mechanism which led to increasing the Cu hardness [11,15].

3.3 Electrical Conductivity

Figure 3 shows the electrical conductivity (EC) of the AA and ECAPed Cu samples for the two channel angles used as a function of the number of passes. The AA billets revealed an EC of 99.4% IACS whereas all the ECAPed Cu billets revealed an insignificant reduction in the EC as the processed samples exhibited a value of EC ranging from 92.8% up to 96.6% IACS as shown in Figure 3. Furthermore, the ECAP die of 120° channel angle exhibited higher EC when compared to the ECAP die with 90° internal channel angle for all processing condition. Processing through 2-Bc and 6-Bc using the 90°-die resulted in decreasing the EC by 3.1% and 6.6%, respectively when put in comparison with the AA condition. Similarly, ECAP die with internal channel angle of 120° revealed a decrease in the EC by 2.8% and 4.7% when it put in comparison with the AA counterpart.

From the aforementioned findings, it can conclude that the different ECAP processing parameters were not affected significantly in reducing the EC as the reduction percentage was not more than 6.6% compared to the AA counterpart which indicated that ECAP processing can strengthen the Cu billets without losing its EC which agreed with [29].

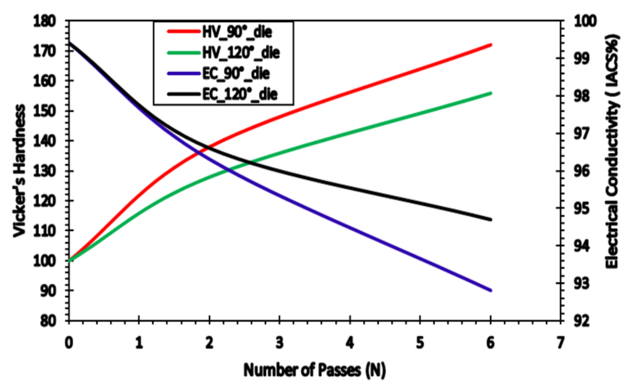

Figure 3: Vicker's hardness and electrical conductivity of the Cu billets as a function of ECAP passes.

On the other hand, it was clear that the EC decreased with increasing the number of passes. The scattering of electrons results from dislocations, grain boundaries, or other defects that are generated and increased during the ECAP processing which leads to the scattering of electrons [30].

Similar findings were reported by Dalan et al. [31, 32] for Cu‑ 0.81Cr‑ 0.07Zr Alloy. They reported that the number of ECAP passes displayed an insignificant change in the EC. Zhu et al. [33] reported the decreasing of the EC of Cu-Mg alloy by refining the grains as well as increasing the dislocation density and point defect through processing via multiple passes. In addition, the effect of ECAP processing on decreasing the EC of Cu-Cr alloy, was reported by Kommel et al. [34]. Kumar et al. [35] explained the reduction of EC of the oxygen-free high conductivity Cu through cyclic channel die compression by increasing the density of deformation-induced defects such as point defect, dislocation density, and grain boundaries. A similar finding of decreasing the EC by increasing the number of passes was reported by Ko et al. [36] for Cu–3 wt%Ag alloy and Wei et al. [37] for pure Cu. On of contrary, Ciemiorek et al. [38] reported the reduction of the Ec of Cu during the first pass while it revealed a subsequent increase as it processed through 8-passes.

4. CONCLUTIONS

Commercial Cu rods were ECAP extruded at RT using two separate dies with channel angles 120° and 90° for two and six passes of route Bc, respectively. The following conclusions are possible:

1. 2-Bc processing resulted in an extended ultrafine-grained structure oriented parallel to the extrusion direction.
2. The 90°-die had more UFG equiaxed structure than the 120°-die
3. The maximum hardness values were achieved from 6-Bc using an ECAP die with a 90° channel angle.
4. When compared to the AA condition, ECAP processing through 6-Bc utilizing the die with a channel angle of 90° enhanced the HV of Cu by 72%.
5. 6-Bc ECAPed via the 90°-die and 120°-die resulted in a 6.6% and 4.7% decrease in electrical conductivity, respectively, compared to the AA.
6. ECAP treatment can reinforce copper rods without significantly affecting their electrical conductivity.

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