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DEVELOPMENT OF MICROSTRUCTURE, MECHANICAL AND WEAR CHARACTERISTICS OF THE BRASS ALLOY PROCESSED BY ECAP

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ABSTRACT

In the present study, the microstructure, mechanical and wear characteristics of commercial Cu-30Zn brass alloy were developed by an equal channel-angular process (ECAP) using a particular die in constant dimensions. The ECAP process was experimentally conducted at room temperature using (1-4) passes in route C with lubricating conditions. Also, the post-annealing treatment at 350 °C has been done for some brass samples, which were deformed with four passes. Findings revealed that by conducting the ECAP, a significant reduction in the grain size of the deformed brass samples is achieved compared to the as-received alloy. The grain refinement increased with the increasing number of ECAP passes. However, the post-annealing treatment increased the grain size of the deformed brass alloy, but still it was lower than the as-received alloy. Moreover, the mechanical performance, i.e. micro-hardness and strength, was significantly enhanced after the ECAP. The samples processed with three passes presented the highest hardness value (237 HV) and mechanical strength (UTS= 692 MPa, and YS= 542 MPa) due to the homogeneous strain hardening and substantial grain refinement throughout the ECAP process. However, the micro-hardness and mechanical strength of brass alloy decreased after post-annealing treatment compared to those of the ECAP deformed samples. The elongation to failure also decreased greatly with increasing the number of passes of ECAP. Additionally, the wear resistance of the investigated samples increased significantly after increasing the number of ECAP passes compared to the as-received alloy. The highest wear resistance has been achieved for samples deformed by three and four passes of ECAP due to the considerable grain size refinement and higher hardness. However, a slight increase in the wear rate occurred after post-annealing treatment on a brass alloy sample processed with four passes due to the increase in grain size.

KEYWORDS

Brass, ECAP, Mechanical properties, Microstructure, Wear.

1. INTRODUCTION

It is extensively recognized that brass alloys are a vital class of engineering materials utilized for several engineering applications. They can be considered the materials of choice for diverse engineering fields due to their exceptional characteristics like specific strength, elongation to failure, and corrosion behavior (Chawla and Gupta, 1993). They are widely used in producing heat exchangers, engine parts, tanks, and propellers, as well as in petroleum refineries, air-conditioning, refrigeration and marine engineering (Xia and Szklarska-Smialowska, 1990; Selvaraj et al., 2003). Recently, much attention and focus have been paid from researchers, scientists and engineers on the engineering materials with reduced grain size since the discovery of the Hall-Petch equation (Hall, 1951; Petch, 1953). This equation has verified that the strength can be developed by reducing the grain size. Also, grain size reduction can enhance the fracture toughness of the materials and promote superplastic deformation at higher strain rates. Many techniques and methods have been developed to induce a higher grain refinement (in sub-micron or nano-scale) compared to that obtained from conventional processes (above 10 μm). In this regard, severe plastic deformation (SPD) is a modern top-down technique for producing advanced materials in ultrafine-grained (UFG) or nanoscale structures from coarse-grained materials. The microstructure and mechanical properties of deformed alloys can be considerably modified and improved by this deformation approach. During SPD processes, severe strain is applied to the deformed alloy to produce UFG (100-1000 nm) or nano-materials (NS) (< 100 nm) (Valiev et al., 2000). Nowadays, there are lots of techniques and methods used to achieve SPD, such as high-pressure torsion (HPT) (Lugo et al., 2008), accumulate roll bonding (ARB) (Jang et al., 2008), hydrostatic extrusion (HE) (Garbacz et al., 2007), cryorolling (Sarma et al., 2008), constrained groove pressing (CGP) (Ebrahimi et al., 2014), surface mechanical attrition treatment (SMAT) (Wang et al., 2003), friction stir processing (FSP) (Mishra and Ma, 2005) and ECAP (Neishi et al., 2001; Zhang et al., 2011). ECAP is a cost-effective approach with several advantages over other SPD techniques. The main feature that distinguishes this process from traditional metal-working methods is its ability to deform the metallic workpiece repeatedly without changing its cross-sectional area (Azushima et al., 2008). During ECAP, shearing and bending deformations may influence the size and orientation of grain crystals of the deformed samples without a change in their dimensions (Han et al., 2007). This process is influenced by many major parameters, like die angle, route mode, the number of passes, and temperature, which must be regulated to refine the grains to a requisite level. Also, ECAP can be applied to a wide range of pure metals and their alloys for various engineering applications such as Al (Kumar et al., 2012), Mg (Muralidhar and Narendranath, 2014), Ti (Agarwal et al., 2020), Cu (Lugo et al., 2008; Kim et al., 2012; Mousavi et al., 2018), etc. Over the last few decades, the interest in developing ECAP for brass alloys has grown considerably using different parameters and fabrication procedures of ECAP. This is to eliminate various technical problems involved during the experimentation and induce more grain refinement in brass microstructure with higher mechanical properties.

In this work, ECAP was experimentally carried out on Cu-30Zn brass alloy using a new die at specific parameters. The microstructure, mechanical properties and wear resistance of deformed brass samples were evaluated. The major objective is to accomplish greater grain size reduction with exceptional mechanical and tribological characteristics.

2. MATERIALS AND METHODS

In this study, rod samples of Cu-30Zn (C260) brass alloy with a diameter of 1.2 cm and length of 100 cm were used for the experimental work. These samples were then cut into cylindrical billets with a diameter of 10 mm and length of 54 mm to be used later for different ECAP processes. This short length is due to very high friction force resulting from a long input channel, which could cause punch buckling or fracture under these critical conditions. Afterwards, annealing treatment for brass samples was made at 600 ^{o}C for 60 min to obtain homogeneous microstructure with recrystallized grains. Finally, the billets were cut to almost 0.3 mm in diameter as losing dimension for fitting the samples precisely in the strongly closed ECAP die. It is important to mention here that a particular die, made of high carbon-chromium tool steel (D4), was designed and manufactured to be applicable for processing several brass samples by ECAP. More details about the ECAP processing and its die used in this study were mentioned elsewhere (Radhi et al., 2021). The ECAP experiments in this study were carried out at room temperature using one to four passes of route C. It was pointed out that C route is an effective way for obtaining the better exploitation of material to around 83.3% (Xu et al., 2007). The die parameters, die angle (Φ) and curvature angle (Ψ), were precisely adjusted to be 90° and 28°, respectively. It was found that homogeneous deformation may be obtained after manufacturing the ECAP die with these parameters (Barber et al., 2004). Moreover, to enhance the microstructure and mechanical properties of investigated brass alloy, brass alloy samples deformed with four ECAP passes experienced annealing heat treatment at 350 °C for 20 min.

The microstructural characterization of initial and processed brass alloy samples was studied using an optical microscope (OPTIKA Company, XDS-3MET, ITALY). For this purpose, cylindrical samples (diameter: 10 mm, and thickness: 2 mm) were cut perpendicularly to the pressing direction using a wire cut machine. Afterwards, the samples were mechanically grinded to 1200 grit using SiC papers and then polished in an electrolyte of 0.5 µm diamond. Finally, the samples were etched within a few seconds using a solution of 90 mL distilled water and 10 mL NOH_3, dipped in warm water, and then dried. Indentation micro-hardness test of initial and ECAP deformed brass samples was carried out with an MHV-2000S Vickers tester using 9.8 N load and 15 seconds dwelling time. The micro-hardness value was statistically averaged from the results of five indentations on the cross-section of each tested sample. A tensile test was conducted on standard smaller-size tensile samples (ASTME8- E8M) for initial and ECAP deformed brass samples. The test was performed at room temperature by a GUNT HAMBURG WP300 machine using a 20 kN load cell to attain different mechanical properties, i.e. yield strength (Ys), ultimate tensile strength (UTS) and elongation to failure. The wear resistance of investigated brass samples was also evaluated by a pin-on-disk sliding wear tester (MICRTEST, S.A., Spain) at room temperature using 20 N applied force, 20 rpm sliding velocity and 6 mm pin diameter.

3. RESULTS AND DISCUSSION

Fig. 1 shows the EDS results of the initial brass alloy used in this study. The figure reveals the major elements of investigated alloy, i.e. Cu, Zn, and O.

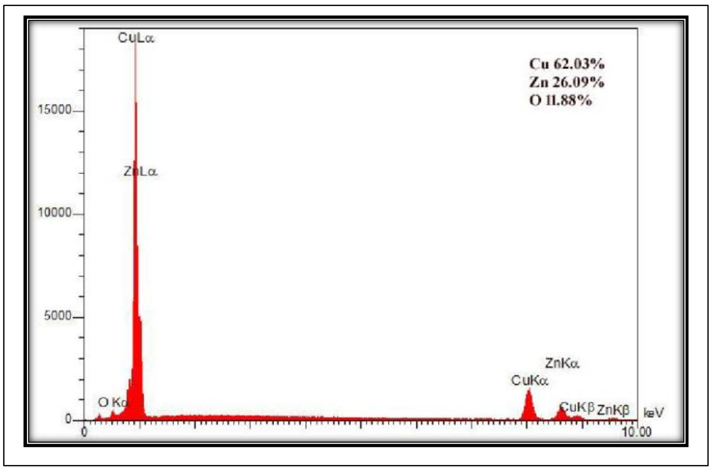

Fig. 1. EDS analysis of Cu-30Zn alloy used in this study.

Fig. 2 demonstrates the microstructural characteristics of initial and deformed brass samples at different passes. The microstructure of the initial annealed alloy (Fig. 2a) consists of wide major regions of equiaxed α phase (lighter) and minor amount of β phase distributed on the grain boundaries (darker) (Dutkiewicz et al., 2009). The microstructural percentage of these two phases has been determined to be almost 70% for α phase and 30% for β phase. Also, the average grain sizes of α and β phases in the initial alloy were estimated to be 22.5 and 5.3 µm, respectively. It is important to note that the grain boundaries in initial samples are more clear and can be distinguished, unlike in deformed samples where it is difficult to distinguish them. Previous studies have shown that it is very difficult to distinguish the grain boundaries of the brass samples after they are deformed by several ECAP passes (Iwahashi et al., 1997; Oh-Ishi et al., 1998). This result may be closely related to the increase in the density of dislocations after the multi-pass ECAP process. The microstructure of brass samples deformed by ECAP at different passes is shown in Fig. 2 (b-f). It can be observed that grain refinement occurs after one pass of ECAP, where grains elongated and flattened in the direction of alloy flow and across a unidirectional plane of shear during ECAP processing (Yoon et al., 2007). It was reported that parallel lines, called shear bands, in the elongated grains of ECAP deformed brass alloy may be formed in the direction of the shear plane from the die angle to the shear plane. Increasing the number of ECAP passes is an effective way to make these shear bands clearer and denser. However, in this study, the shear bands were not visible in the microstructure of the brass alloy after the ECAP, regardless of the number of passes. Furthermore, after subsequent passes, the grain size decreased significantly, as noted in the microstructures of the samples deformed for 2, 3 and 4 passes (see Fig. 2 (c-e)). This is due to the slowing of the strain hardening, which in turn may cause continued and accumulative deformation energy within the shear bands (Suryadi et al., 2013). It should be noticed that Cu alloys in general have a lesser stacking fault energy compared to that of Al alloys (Humphreys and Hatherly, 2012) and higher density of dislocations (Kim et al., 2012). This greatly prohibits the recovery, which may cause a significant refinement in the microstructure of the brass samples deformed by ECAP processing. Moreover, it is important to know that the kind of processing route is an effective parameter in refining the microstructure of the ECAP processed samples. Thus, in this study, route C was chosen since it is more efficient in achieving high-strain energy with more microstructural homogeneity and refinement than other processing routes (Iwahashi et al., 1997; Oh-Ishi et al., 1998). Post-annealing after ECAP led to producing a uniform structure with new small grains. Also, the orientation of grains is almost returned to its original orientation, and the grains become equiaxed with an increase in their grain size due to the phenomena of recrystallization and grain growth (see Fig. 2 (f)). Interestingly, the grain size of the post-annealed brass sample is still significantly smaller than that of the initial brass. Moreover, it is expected that the brass alloy sample processed by ECAP followed by annealing treatment has a lower stacking fault energy, which promotes obtaining a fine microstructure by discontinuous dynamic recrystallization (DDR) during annealing treatment (Humphreys and Hatherly, 2012). The higher strain energy induced by route C can effectively develop the microstructure due to the large number of DDR's recrystallization nuclei at high-strain energy sites, along with the development of annealing twins. In this work, ECAP processing for 4 passes before annealing treatment induces higher deformation and heat energy into the processed brass alloy, which activates the recrystallization mechanism (Azushima et al., 2008).

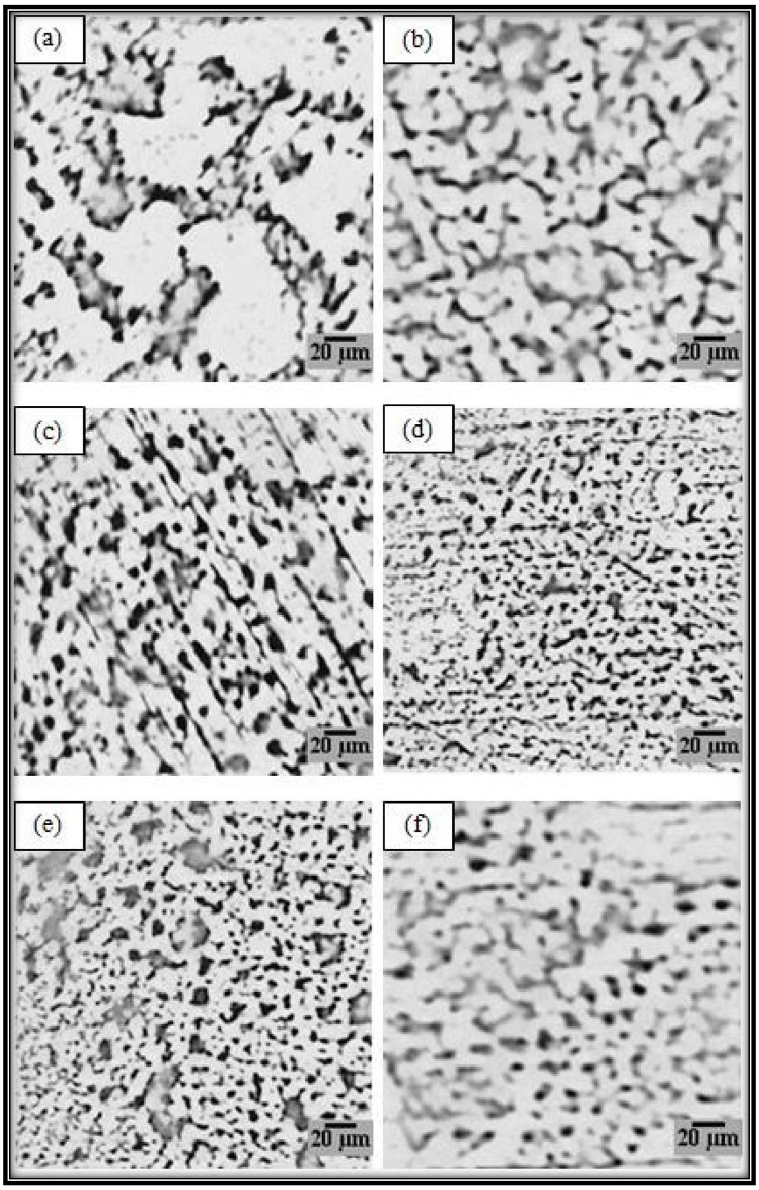

Fig. 2. Optical microstructures (1000 X) of brass alloy samples: (a) as-received, (b) 1 pass, (c) 2 passes, (d) 3 passes, (e) 4 passes and (f) 4 passes followed by annealing.

Fig. 3 shows the average grain size of α phase and β phase in the microstructure of initial brass alloy and after different passes of ECAP. Among all deformed brass samples, the sample processed with 3 passes of ECAP has the smallest average grain size (3.7 μm for α phase and 2 μm for β phase). It is important to conclude here that the results of these gain size measurements are more significant than those of the microstructural observations seen in Fig. 2.

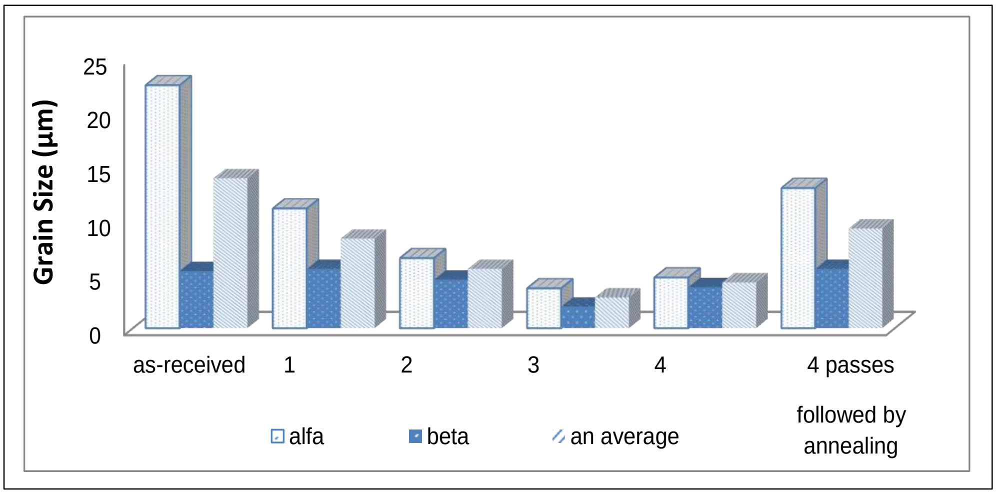

Fig. 3. Grain size distribution of brass alloy deformed by ECAP in different conditions.

The average micro-hardness results of the investigated brass samples are presented in Fig. 4. Here, the lowest hardness was observed in the initial brass alloy (75 HV). However, the micro-hardness of ECAP processed samples increased considerably, indicating the ECAP process's greater influence on the mechanical performance of the brass alloy. After the first pass of ECAP, a sharp increase in micro-hardness is made to 195 HV. Among ECAP processed samples, the sample deformed with 3 passes of the ECAP has the highest value (237 HV). This noticeable increase in the hardness is due to the dynamic effect of the strain hardening throughout the plastic deformation accompanying the ECAP process. It was pointed out that the refinement in grain size at severe strain could induce a vital development in hardness (Azushima et al., 2008; Pasebani and Toroghinejad, 2010). Hence, the brass sample that was deformed by ECAP for 3 passes has the highest value of micro-hardness owing to the significant reduction in its grain size (see Fig. 2 (d) and Fig. 3), along with the homogeneous strain that occurred during the 3 passes of ECAP. However, a slight decrease in brass alloy micro-hardness occurred after ECAP for 4 passes. This may be due to the saturation phenomena that induced throughout SPD at large strains (Sarma et al., 2008; Azushima et al., 2008; Pasebani and Toroghinejad, 2010). For the post annealed brass sample, the micro-hardness was decreased to160 HV, which is still greater compared to the initial hardness. This hardness reduction is due to the recrystallization process that occurred during the annealing treatment and a considerable increase in grain size (see Fig. 2(f)). It should be noted that the recrystallization process can result larger strain easily; hence, a greater reduction in hardness is anticipated (Zhao et al., 2008).

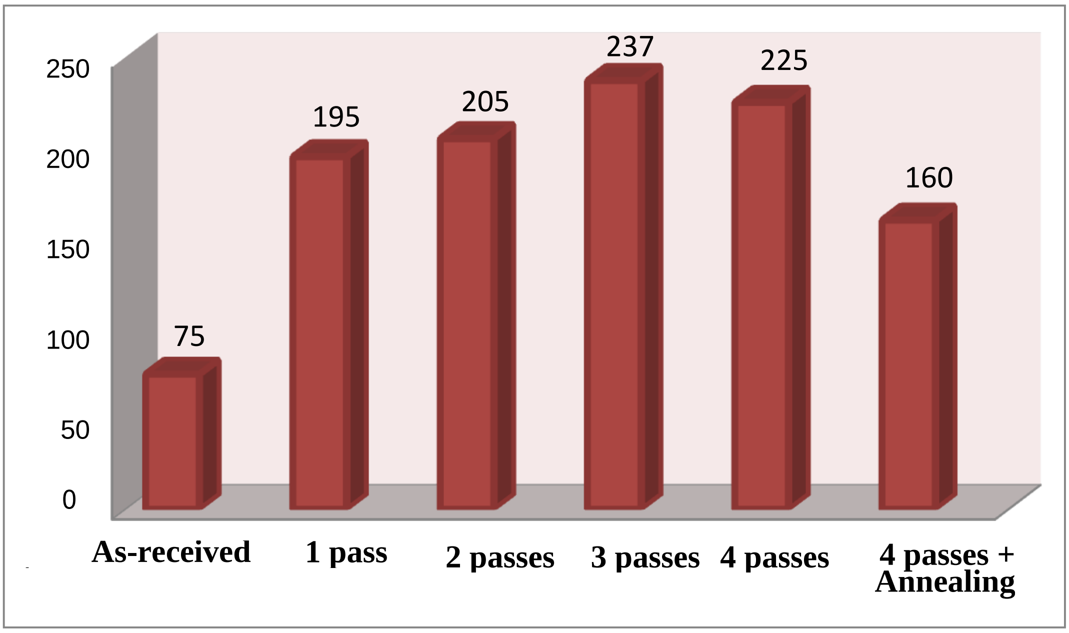

Fig. 4. Variations of micro-hardness for brass alloy deformed by ECAP in different conditions.

Fig. 5 presents the tensile mechanical properties (strength and elongation) of initial and brass samples deformed via ECAP in different passes. It can be seen that there is a noticeable increase in the strength values after the ECAP process, as the initial sample has the least strength. This development of the strength had an analogous tendency to the previous magnitudes of micro-hardness (Fig. 4). Also, the increase in the number of ECAP passes played an effective role in increasing the YS and UTS strengths of processed brass samples. By the beginning of deformation in the first pass, mechanical strengths had increased up to the third pass and then slightly decreased at the fourth pass. This decrease in strength after the third pass means that the deformation reaches a steady state. Among ECAP processed brass samples, 3 passes deformed samples presented the best strengths (UTS= 692 MPa and YS= 542 MPa). This is closely related to the considerable decrease in grain size during the ECAP process (see Fig. 1 (d)). It is important to refer here that the decrease in grain size as a result of high strain during the ECAP process may lead to an increase in both the density of the dislocations and the area of the grain boundaries, which in turn led to an expansion in the size of the blocking areas for these dislocations. Consequently, under these sever conditions the mechanical strength is considerably improved (Mousavi et al., 2017). However, there is an evident decrease in ductility associated with this improvement in strength. The elongation reduced with increasing the number of passes of ECAP. The lowest elongation values, 5.5% and 5.6% were obtained after 4 and 3 passes of ECAP, respectively. The loss of elongation in UFG or NC alloys can be attributed to the development of the shear fracture without apparent necking features. This means that the grain size reduction may lead to the existence of microstructural shear bands that cause a decrease in the angle of shear fracture and the degree of necking (Zhang et al., 2014). The shear bands are the most important mechanism for plastic deformation through the SPD materials, which could ultimately cause a rapid fracture of these materials (Ivanisenko et al., 2010). Moreover, for brass alloy treated by post-thermal treatment after the ECAP, the YS and UTS decreased considerably comparing with that of ECAP deformed samples; but they are still significantly higher compared to the initial alloy. The post-thermal treatment is an efficient way to enhance mechanical strength without a greater decrease in elongation. The development in mechanical properties, i.e. strength and elongation, can be clarified in terms of grain reduction and recrystallization phenomena induced by ECAP and thermal treatment, respectively. The post-annealed brass sample had a considerable grain refinement compared to the initial alloy (see Fig. 1 and Fig. 2), which increased its mechanical tensile strength. On the other hand, the elongation of this sample increased greatly to 12.5%, which is higher than that of all ECAP processed samples. Similar results have been reported for Cu-40Zn alloy (Kim et al., 2012). Interestingly, compared to the initial alloy, ECAP and post-thermal treatment can induce higher numbers of refined grains related to α and β phases, which meet at the phase interface. These interfaces between α and β consist of high angle boundaries, which promote more boundary slipping compared to those of interfaces between the same α regions or same β regions (Humphreys and Hatherly, 2012).

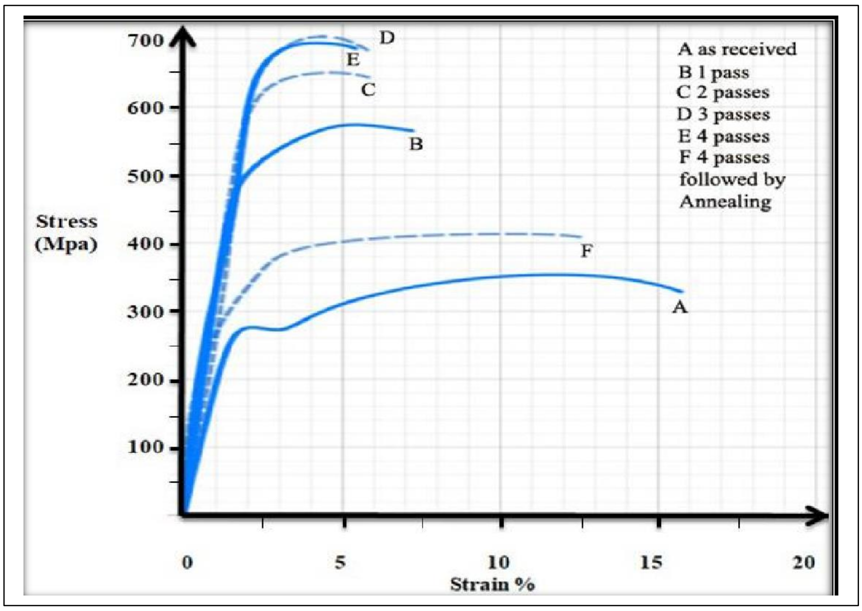

Fig. 5. Stress-strain curve of unprocessed and ECAPed brass alloy samples.

Table 1 presents the wear characteristics of initial and ECAP-deformed brass samples using different passes. It can be seen from Table 1, the ECAP deformed brass samples have much lower values of both wear mass loss and volume loss than the initial alloy. This means that the ECAP process has a significant effect on improving wear resistance of the investigated samples. In this work, a substantial decrease in the mass loss was attained after increasing the number of ECAP passes. This is very clear as the mass loss decreased by 50% from 0.8 mg for the initial sample to 0.4 mg for the one-pass deformed samples. The brass alloy samples deformed by 3 passes and 4 passes of ECAP had the lowest mass loss (0.2 mg) among all processed samples. It should be noted that the wear rate was reduced by about four times after the ECAP process compared to the initial alloy. This result can be interpreted as being closely related to the reduction of the grain size and the increase in the area of the grain boundary after the ECAP process (see Fig. 2 & Fig. 3), which in turn led to an increase in the surface hardness and wear resistance. However, a slight increase in the wear rate was achieved for processed samples at 4 passes followed by annealing treatment owing to increased grain size compared to other processed brass samples.

Table 1. Results of wear test for initial and ECAP deformed brass samples.

Condition	Mass loss (g)	Volume loss (mm^3)	Wear rate (mm^3/Nm)	Wear resistance Improvement (%)
Initial	0.0008	0.0937	$6.24*10^{-4}$	-
1 Pass	0.0004	0.0468	$3.12*10^{-4}$	50
2 passes	0.0003	0.0351	$2.34*10^{-4}$	62.5
3 passes	0.0002	0.0234	$1.56*10^{-4}$	75
4 passes	0.0002	0.0234	$1.56*10^{-4}$	75
4 passes followed by annealing	0.0003	0.0351	$2.34*10^{-4}$	62.5

4. CONCLUSIONS

In this study, the ECAP process was carried out on the Cu-30Zn alloy at room temperature under different operating conditions. The most important conclusions of this work can be summarized as follows:

- ECAP deformed brass samples characterize substantial homogeneity and grain reduction in comparison to the initial alloy. Among all deformed brass samples, the sample processed with 3 passes of ECAP has the smallest average grain size. However, the grain size of post-annealed brass samples increased, but it is still significantly lower than that of the initial alloy.

- A substantial improvement in the micro-hardness of brass samples was achieved after ECAP processing. The brass samples processed with 3 passes of ECAP present the highest value of 237 HV with a percentage improvement of 216%. However, the hardness of ECAP processed brass alloy decreases after post-annealing, which is still higher than its initial hardness.

- A distinctive increase in strength was achieved after the ECAP process. Among ECAP processed brass samples, the three-pass deformed sample offers the highest mechanical strength with percentage improvements of 91.68% and 88.85% for UTS and YS, respectively, However, the elongation was reduced after increasing the number of passes. Also, the mechanical strength of the brass alloy, treated by post-thermal treatment after ECAP, reduces substantially but is still significantly better than that of the initial sample.

- The ECAP process is a beneficial technique to increase the wear resistance of brass alloy. The wear mass loss or volume loss of deformed brass samples decreases extensively after increasing the number of ECAP passes compared to the initial brass alloy. The brass alloy samples deformed by three and four passes of ECAP present the highest wear resistance among all processed samples. However, a slight increase in the wear rate of processed brass alloy samples occurs after carrying out post-annealing treatment.

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