



Effect of PTCAP Passes on the Mechanical Properties of Copper Tubes

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ABSTRACT

Commercially pure copper samples were successfully deformed by parallel tubular channel angular pressing (PTCAP) up to different passes at room temperature. The effects of the PTCAP number of passes on the microstructure, mechanical properties, microhardness and wear resistance of the processed samples were fully investigated. The microstructure of processed samples were observed by SEM and showed notable decrease in the grain size with increase number of passes. The mechanical properties of the pure copper in each pass were studied by tensile testing and microhardness method at room temperature. In this respect, UTS, $\sigma_{0.2\%}$ and microhardness have been markedly improved from 199.03, 102.17 MPa and 67.83 HV as the annealed condition to 331.40, 293.51 MPa and 144.03 HV after the fourth pass, respectively. Besides, the elongation percent were decreased while, the wear resistance improved with increased number of PTCAP passes. Ductile fracture with extensive necking zone and many big dimples occurs in annealed samples, while fine dimples were decreased with the deformation final passes of PTCAP processed samples.

Key words: Parallel Tubular Channel Angular Pressing (PTCAP), Copper Tubes, Mechanical Properties, Microhardness and Wear Resistance

1. INTRODUCTION

Copper has been used for thousands of years and its mechanical properties are well known. Its used in many branches of industry intensively and steadily increasing in recent decades. The major applications are in wires, industrial machinery, copper-based solar power collectors, integrated circuits and generally intensively used in electronics, in recent decades. Copper can be also recycled very effectively and represents a relevant engineering material also for the future [1-6].

The effort to increase mechanical properties of engineering materials led in the last two decades to application of severe plastic deformation (SPD), resulting in fine-grained structures, producing improved mechanical and physical properties in ultrafine-grain (UFG) and nanostructured (NS) materials. Naturally, copper is a suitable material for basic research and, simultaneously an improvement of its mechanical tensile strength is a permanent research challenge [7, 8]. This can be obtained by using severe plastic deformation (SPD) techniques.

They are known as an effective way of grain size refinement below 1 micron and the production of ultrafine-grained (UFGed) materials. Such materials feature extremely high strength due to grain boundaries strengthening as described quantitatively by the Hall-Petch relationship [9, 10]. Nowadays, the most popular SPD techniques include equal channel angular pressing (ECAP) is specially processing technique for many reasons [11]. ECAP was previously applied to bar and rod form parts [12], along with application to plate and sheet parts [13]. Despite the need for high strength tubes in a wide range of industrial applications. Toth *et al.* [14] proposed an SPD method based on high pressure torsion (HPT) for producing UFG tubes. It seems that the method by Toth *et al.* may have some limitations such as low homogeneity. Mohebbi and Akbarzadeh [15] developed an accumulative spin-bonding (ASB) method based on accumulative roll-bonding (ARB) to produce UFG tubes. Recently, the other two novel SPD methods are based on the conception of ECAP technique. The tubular channel angular pressing (TCAP) [16, 17] and the parallel tubular channel angular pressing (PTCAP), have been proposed by Faraji *et al.* for fabrication of (UFG) and (NS) tubes. Of these, PTCAP process has some advantage such as needing lower process load and a better strain homogeneity compared to TCAP process [18, 19]. TEM analysis of specimen showed that increase in the number of passes changes the elongated grains to equiaxed grains with ~150 nm sizes from the initial of average grain size about ~59 μm . Microhardness value of the processed tubes significantly increases to ~117% after first pass from the initial value of ~62 HV. Yield (0.2%) and (UTS) were increased 2.5 and 2.28 times as compared to initial specimen.

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Wear properties of engineering materials have significant effects on the serviceability and durability [20]. It is generally known that, the wear resistance of metals and alloys is proportional to their hardness. Enhancement of the wear resistance of materials can be attained by various methods as heat treatment and surface coating.

The PTCAP process consists of two half cycles shown schematically in Fig. (1). In the first half cycle, the first punch presses the tube material into the gap between mandrel and die including two shear zones to increase the tube diameter to its maximum value, as shown in Fig. (1 a). Then, the tube is pressed back using the second punch in the second half cycle, decreasing the tube diameter to its initial value, as shown in Fig. (1 b). The equivalent strain achieved from the N passes of PTCAP process can be estimated via the following equation [19]:

$$\varepsilon_{TN} = 2N \left\{ \sum_{i=1}^2 \left[\frac{2 \cot(\varphi_i/2 + \psi_i/2) + \psi_i \operatorname{cosec}(\varphi_i/2 + \psi_i/2)}{\sqrt{3}} \right] + \frac{2}{\sqrt{3}} \ln \frac{R_2}{R_1} \right\} \quad (1)$$

From equation (1), the total equivalent plastic strain after first pass PTCAP with the parameters used in this work is ~ 2.5 .

The present work was performed in order to achieve two mean objectives. First to investigate the effect of grain-

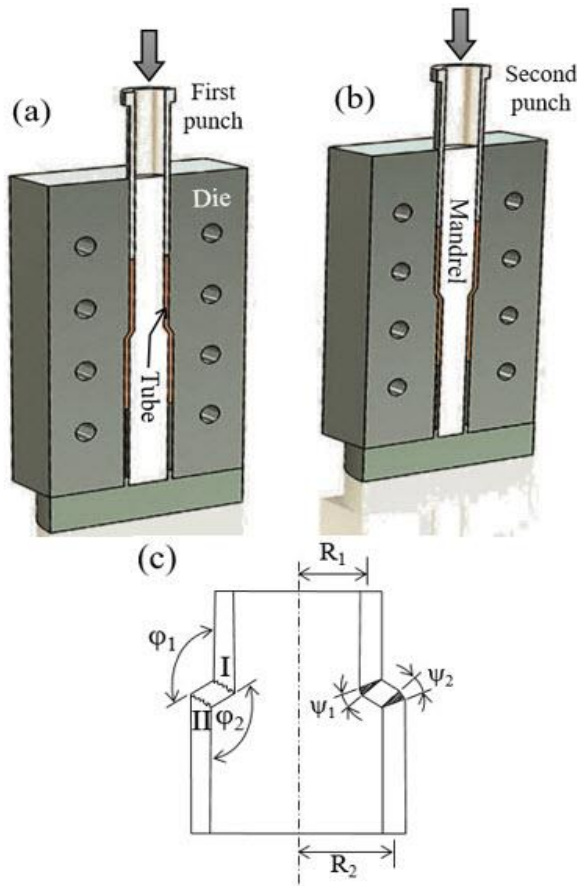


Figure (1), Schematic of the PTCAP process in the a) first, b) second half cycles and c) die parameters.

refinement through each pass of PTCAP with variations in the mechanical property including tensile properties, microhardness and fracture surface. Second to study the effect of number of PTCAP passes on the wear properties (resistance).

2. EXPERIMENTAL WORK

The material used in the present study was commercially pure copper that was received in the form of the extruded tube. Its chemical composition was 0.0005 wt.% Sn, 0.0035 wt.% Pb, 0.0001 wt.% Fe, 0.0016 wt.% Ni, 0.0001 wt.% Mn, 0.0005 wt.% Si, 0.0031 wt.% P, 0.0016 wt.% Be, 0.0075 wt.% Mg, 0.0025 wt.% As, 0.0001 wt.% Cr, 0.0037 wt.% S, 0.0003 wt.% Co, 0.0001 wt.% Al, 0.0317 wt.% Zn and balanced copper. Original tube of 2.5 mm in thickness, 20 mm in diameter and 80 mm in length were machined from the copper tube. Prior to the PTCAP process, a recrystallized homogeneous microstructure was obtained by heating the samples to 700 °C for 1 hour.

The PTCAP die with parameters including the channel angle $\varphi_1 = \varphi_2 = 135^\circ$, the angle of curvature $\psi_1 = \psi_2 = 0^\circ$ and $K = R_2 - R_1$, was equal to the tube thickness (2.5 mm) are shown in Fig. (1 c) was manufactured from the tool steel and hardened to ~ 55 HRC. The samples were lubricated with zinc stearate [21]. During the PTCAP process, the tube samples were pressed into the tubular angular gap between the die and the mandrel. Pressing were performed with 300 KN Universal METRO COM model (10334) press under a pressing speed of 2 mm/min at room temperature (RT).

The microstructure was carried out using optical microscopy (OM) model (BX41M-LED), has been employed to confirm the grain refinement of PTCAPed tube alloys. The microstructure of samples sectioned along their axial direction after PTCAP. The metallographic samples were polished using $0.05 \mu\text{m}$ Al_2O_3 and then etched using a solution of (5g FeCl_3 , 100 mL ethanol, and 5 mL HCL) for 10-40 sec [22]. The average grain size was calculated using linear intercept method [23].

Microhardness testing was carried out on ground and polished surfaces. Each value was the average of at least 36 readings. All measurements are at a load of 1000 g, and dwell time of 15 s, before and after PTCAP using microhardness tester Model HV-1000.

Tensile specimens 5 mm wide and of 23 mm gauge length were cut from the tubes as shown in Fig. (2). The axis of tensile test specimen was parallel to PTCAP pressing direction. Tensile testing was conducted at room temperature (RT), until failure according to BS 6746 (L). Tensile tests were carried out using Instron Universal Testing Machine model 4208-002, at a strain rate of $3 \times 10^{-3} \text{ s}^{-1}$, and each test was repeated three times. Also, fracture surfaces of 99.95% Cu tube samples were investigated

using Scanning Electron Microscope (SEM) model FEI INSPECT-S50 at 20 KV.

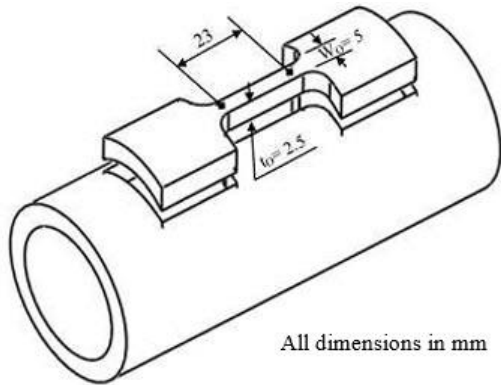


Figure (2), Schematic of dimension of tensile test specimen.

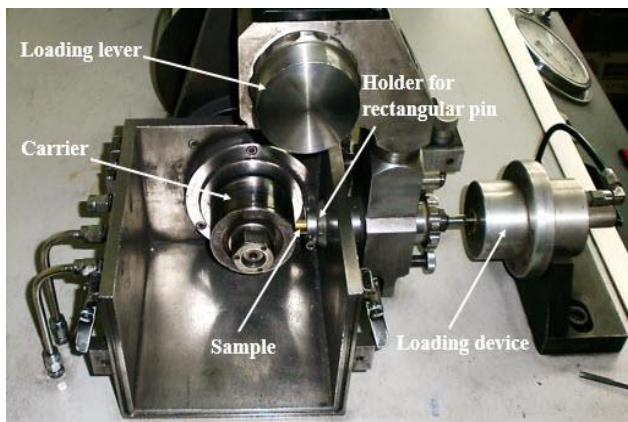


Figure (3), Wear machine block-on-ring test rig (ASTM) model TNO TRIBOMETER.

Finally, dry sliding wear tests were conducted using TNO-Tribometer block-on-ring wear test machine as shown in Fig. (3). Specimen of size 2.5 mm x 8 mm x 14 mm, with its axis parallel to the pressing direction, and were machined from the center zone of specimen before and after PTCAP using CNC machine for carrying out wear test. The wear test was performed at room temperature ($20 \pm 5^\circ\text{C}$) and $50 \pm 5\%$ humidity conditions. The rotating sliding ring (carrier), made of tool steel, was 73 mm diameter, with hardness of $\sim 63\text{Rc}$. The test was performed at a sliding speed of 1.146 m/s with total sliding distances 2062.8 m. Two different loads of 40 N and 50 N were applied in order to investigate the effect of the load on the wear rate. The samples were cleaned before and after the test with acetone in an ultrasonic bath for 12 min and were subsequently dried with hot air. The wear rate was determined according to the difference in the weight of the samples before and after the wear test using a digital balance device with a sensitivity of 10^{-6} kg.

3. RESULTS AND DISCUSSION

3.1. Microstructure Characterizations

Figure (4 a) shows the recrystallized microstructure of annealed 99.95% Cu tube sample, in which the average grain size is $\sim 36 \mu\text{m}$. The microstructure of 99.95% Cu tube after PTCAP are shown in Fig. (4 b-e). The processed microstructure exhibits significant grain refinement. However, differences in microstructure characteristics are less distinct with increasing the accumulated strain. The average grain size in the unprocessed tube is relatively coarser than that of the processed one and the grain size at various regions of processed tubes are clearly refined. The microstructure reveals shear deformation, and the lamellar boundaries. Also, the grain size of copper is strongly affected by the accumulated strain values [24]. It is obvious that a single PTCAP pass of the tubular materials can lead to a decrease grain size with the number of PTCAP passes. Faraji *et al.* [18] conducted PTCAP of Cu at strain per pass equal (~ 3), which is higher than applied in the present work (~ 2.5). They reported such microstructure grains in range of 150-300 nm and evolved equivalent strain ~ 12 after four passes, such range of grain size is expected in this work as shown in Fig. (4 b-e) and equivalent strain ~ 10 after four PTCAP passes.

3.2. Mechanical Properties

The pictures of the annealed and PTCAP processed 99.95% Cu tube specimens were shown in Fig. (5). While, figure (6 a) exhibits the room temperature tensile engineering stress-strain curves of commercial 99.95% Cu specimens processed from zero to fourth pass of PTCAP process. As expected, the strength of the PTCAP specimens increases significantly over the initial specimen.

The stress-strain curves, including ultimate, proof strength and reduction at the elongation percent of the 99.95% Cu tubes annealed and after different number of PTCAP passes are summarized in Fig. (6 a and b) and Table 1. The results show that the magnitudes of (UTS and $\sigma_{0.2\%}$) have been enhanced from (199.03 and 102.17 MPa) at the annealed situation to (257.82 and 135.94 MPa), and (331.40 and 293.51 MPa) after the first and the four passes of PTCAP process, respectively while the elongation to failure percent is reduced from 24.85% to 5.12% and 4.48% after first and four passes of PTCAP process. These indicate that about 30% and 33% improvements have been obtained after the first pass comparing with the annealed state while only 29% and 116% increases have been attained after the four passes comparing with the first pass for the ultimate and proof strengths, respectively. In addition, the elongation to failure at the first and the four passes are decreased close to the $\sim 79\%$ and $\sim 13\%$ as compared to the annealed and first pass of PTCAP process. Figure 7 shows that, fracture behavior is controlled by a classical ductile mechanism of initiation, growth, and cavities coalescence (dimples). Initiation of cavities is the most important. There are two categories of dimples of trans-crystalline ductile fracture, large dimples formed by decohesive mechanism in the inclusion matrix inter-phase, and small dimples which are initiated probably by the

dislocation mechanism [25, 26]. Assuming that the dislocations move towards the nano-grain boundaries, the dislocation density increases with deformation from the first to the fourth pass. Then, the dislocations transform into unstable crack, which is the nucleus of a cavity initiation. Next, growth and cavity coalescence follow the usual mechanism [25]. After several PTCAP passes, the number of dimples increases, and the size of dimples

becomes finer and more diversified in local regions, as shown in Fig. (7 b and c). It is clear that, the sample with 3 PTCAP passes in longitudinal section displays higher ductility than that with 4 passes in transversal section, because the size of its dimples is larger, as shown in Fig. (7 d and e).

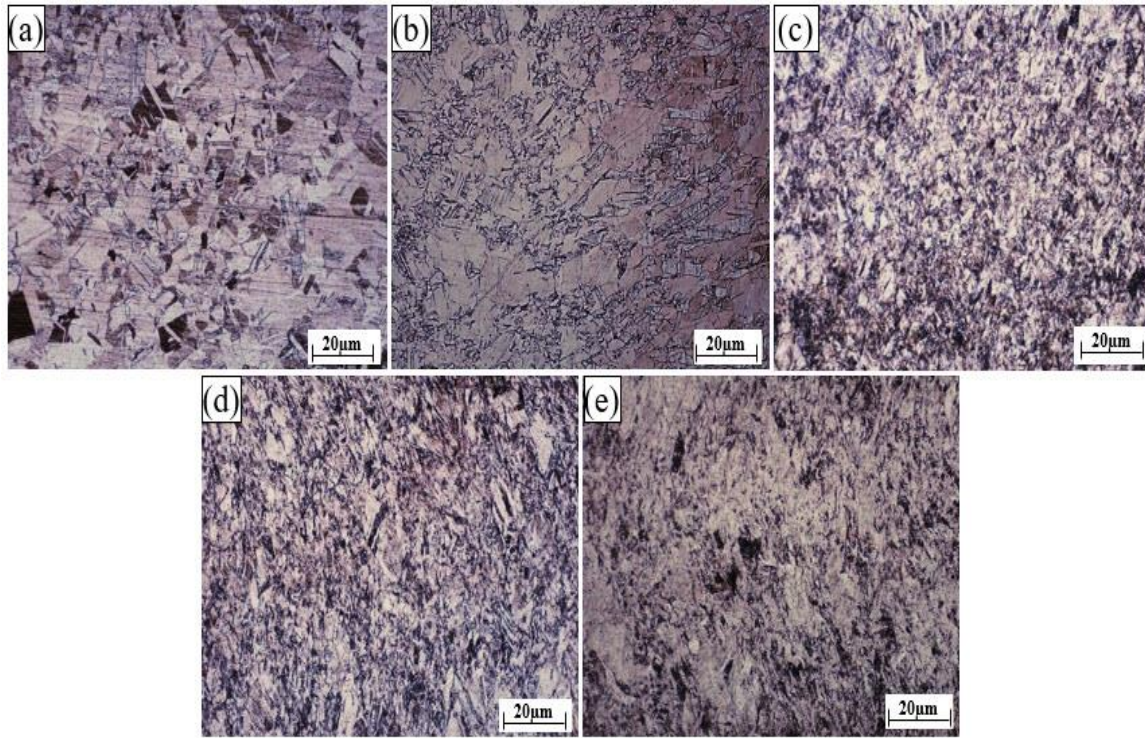


Figure (4), Microstructure of 99.95%Cu tube; a) as-annealed, and after b) pass 1, c) pass 2, d) pass 3, and e) pass 4 of PTCAP processing.

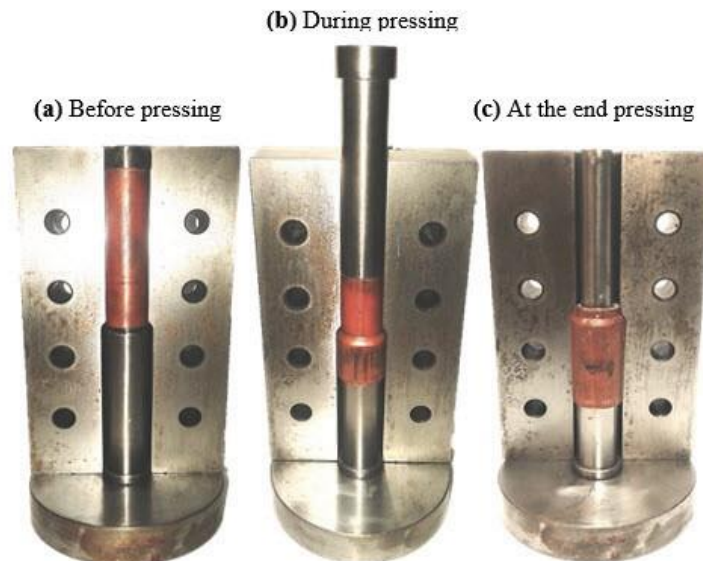


Figure (5), Pictures of the annealed and PTCAP processed 99.95% Cu tube; a) before, b) during, and c) at the end of PTCAP process.

Table 1. Tensile properties of 99.95% Cu tube before and after PTCAP processing

Commercially tube	Mechanical property	PTCAP pass number				
		0	1	2	3	4
99.95% Cu	$\sigma_{0.2\%}$, MPa	102.17	135.94	205.81	259.38	293.51
	UTS, MPa	199.03	257.82	297.25	322.19	331.41
	Elongation,%	24.85	5.18	4.96	4.73	4.48
	Microhardness (VH)	67.83	123.52	135.38	138.60	144.03

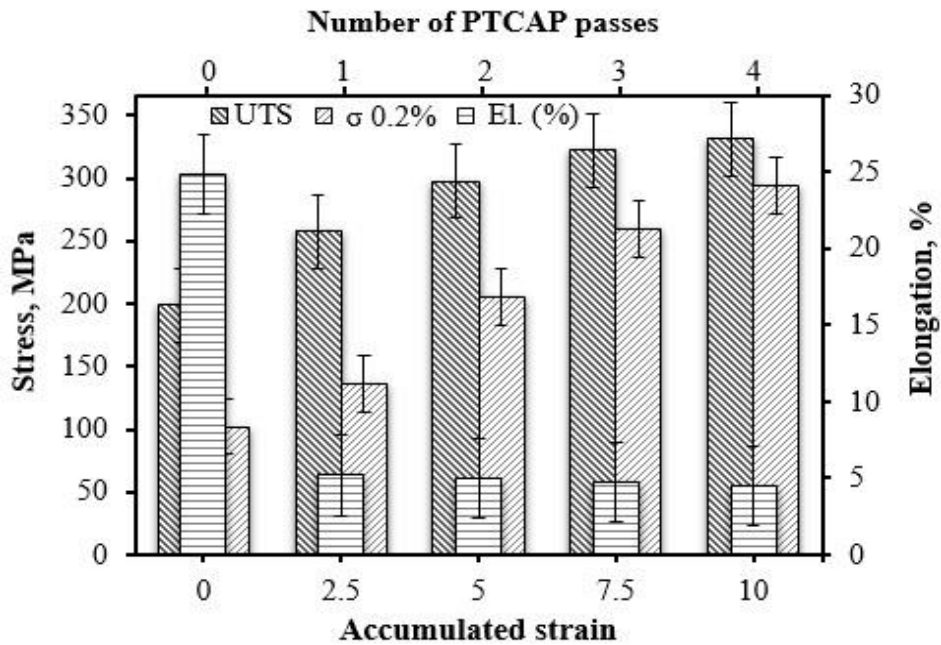
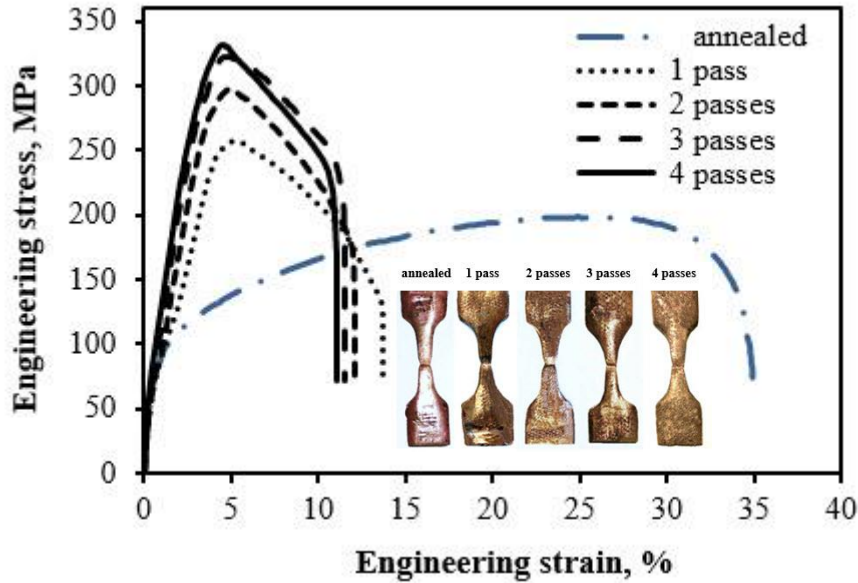


Figure (6), a) Engineering stress-strain curves of annealed and processed 99.95% Cu tubes through 1st-4th pass PTCAP resulted from tensile tests, and b) UTS, $\sigma_{0.2\%}$ and elongation percent of the number PTCAP passes.

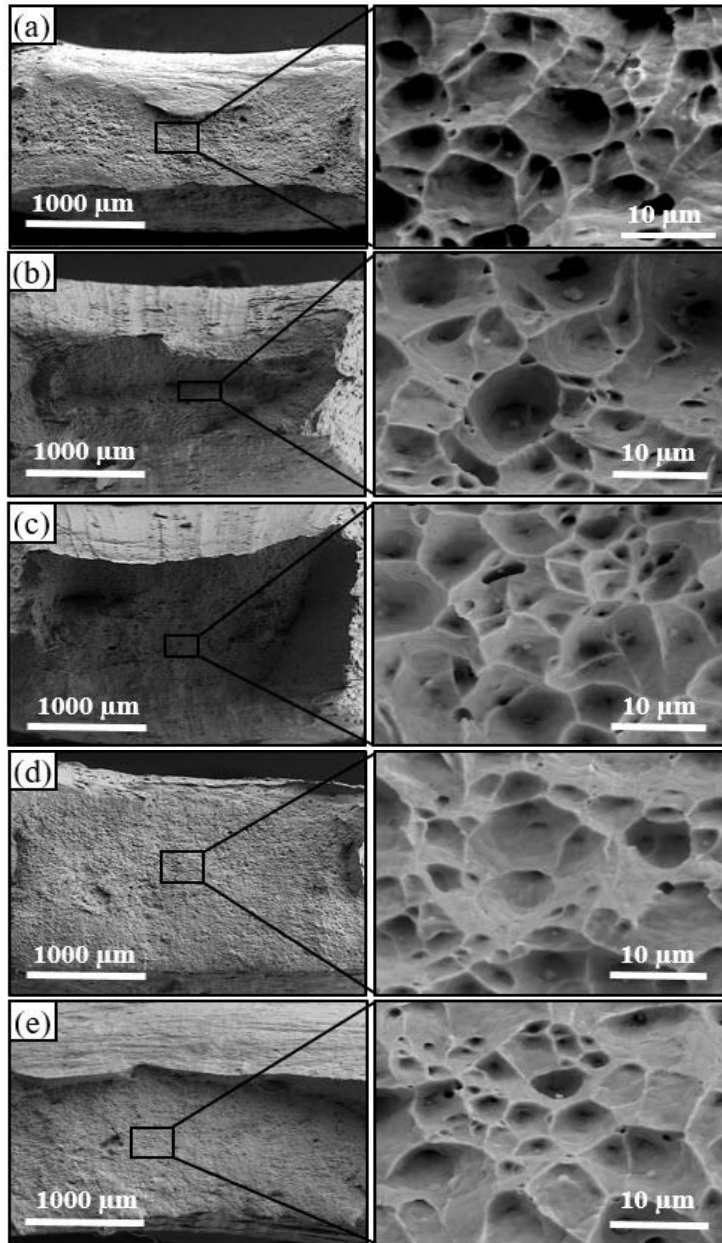


Figure (7), SEM images showing the fracture surfaces of a) annealed tube, b) first pass, c) second passes, d) third passes, and e) fourth passes PTCAP processed tube.

figure (8) shows microhardness values through the thickness of 99.95% Cu tube specimens before and after 1, 2, 3, and 4 PTCAP passes. The microhardness increases notably after first pass of PTCAP and then increases gradually. After the first and four passes, the microhardness is increased about to 123.52 and 144.03 HV from as-received value of only 67.83 HV. This is also verified by the works done by other researchers shown in the chart [27-30].

Moreover, the wear rate values of present 99.95% Cu tubes is determined using a block-on-ring test rig. The obtained results at a speed of 1.146 m/s and under loads of 40 and 50 N as shown in Fig. (9), which shows the effect

number of PTCAP passes on the wear rate. It could be observed that the wear rate decreases with increasing the number of PTCAP passes, specially the first passes.

Furthermore, the applied load strongly affects the wear rate, specially with number of passes, and as expected, higher loads led to higher wear rates. Before PTCAP processing, under 40 N load, the wear rate is ~47% of that under 50 N load. After the fourth PTCAP pass the wear rate under 40 N is ~33% of that under 50 N.

Grain size refining by PTCAP processing leads to the increase of hardness, which in turn increases the wear resistance [31, 32].

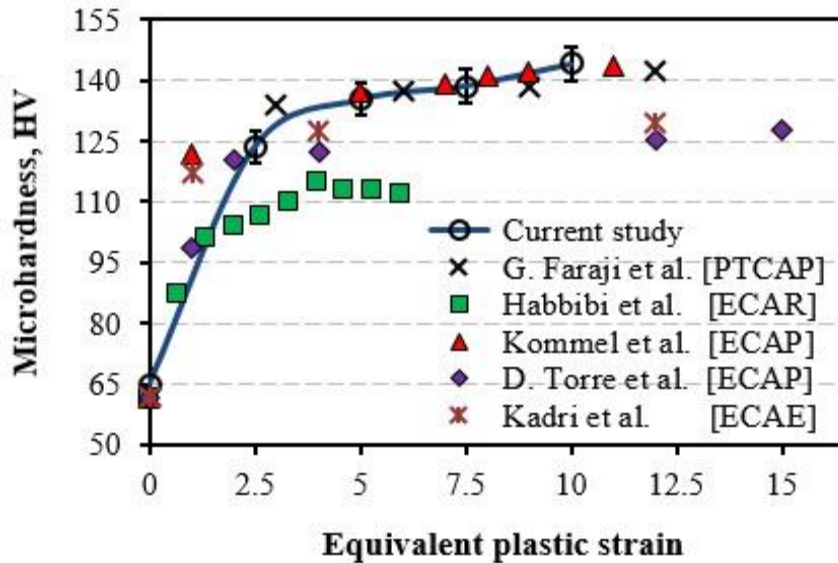


Figure (8), Microhardness versus. Equivalent plastic strain in multi-passes PTCAP processed tubes compared with other researchers [27-30] on SPD of 99.95% Cu tube.

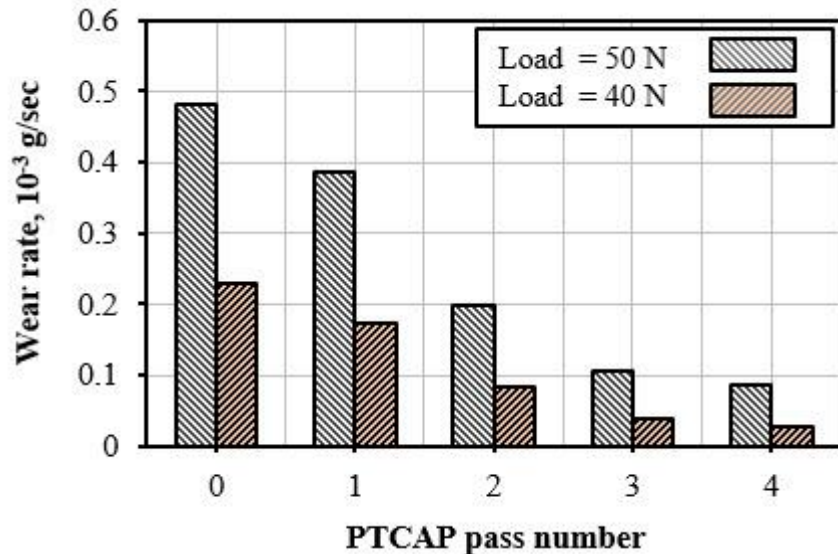


Figure (9), Effect of PTCAP pass number on the wear rate of 99.95% Cu tube.

4. CONCLUSION

Pure copper tubes were severely deformed by PTCAP for up to four passes. The effects of SPD on the microstructure, mechanical properties and the main results are summarized as follows:

1. The PTCAP technique results in material strengthening as well as grain refinement. About 33% and 187% enhancements have been attained for the proof strength after the first and fourth passes compared with that for the annealed state. Also, the value of ultimate tensile strength increased about 30% and 67% for first and fourth passes

passes are decreased close to the ~79% and ~82% over the corresponding as-received condition.

2. Remarkable increase of the microhardness of pure 99.95% Cu tubes with number of PTCAP passes. Microhardness of 99.95% Cu tube is increased to about 123.52 and 144.03 HV after the first and fourth passes 82% and 112% from an initial value of 67.83 HV.

3. Microstructural investigations show a notable decrease in the grain size with increase the number of PTCAP pass.

4. SEM study of fracture surface indicates that, ductile fracture with large necking zone and many big dimples occurs in annealed samples, while fine dimples and limited

ductile fracture features are observed notable decreased size of dimples with increase the number of PTCAP processed samples.

5. The wear resistance of 99.95% Cu tubes is improved with increase the number of PTCAP pass, especially after first pass.

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REFERENCES

- [1] L. Kunz and L. Collini, "Mechanical properties of copper processed by equal Channel Angular Pressing - a review", *Frattura ed Integrità Strutturale*, vol. 19, pp 61-75, (2012). [doi:10.3221/IGF-ESIS.19.06](https://doi.org/10.3221/IGF-ESIS.19.06)
- [2] M.M. Sadawy and M. Ghanem, "Grain refinement of bronze alloy by equal-channel angular pressing (ECAP) and its effect on corrosion behaviour", *Defence Technology*, vol. 12, pp 316-323, (2016). <http://dx.doi.org/10.1016/j.dt.2016.01.013>
- [3] F. Djavanroodi, A.A. Zolfaghari and M. Ebrahimi, "Experimental investigation of three different tube equal channel angular pressing techniques", *Kovove materialy*, vol. 53, pp 27-34, (2015). [doi:10.4149/km-2015-1-27](https://doi.org/10.4149/km-2015-1-27)
- [4] M. Lipińska, P. Bazarnik and M. Lewandowska, "The electrical conductivity of CuCrZr alloy after SPD processing", *Materials Science and Engineering*, vol. 63, pp 012119, (2014). [doi:10.1088/1757-899X/63/1/012119](https://doi.org/10.1088/1757-899X/63/1/012119)
- [5] S.R. Agnew and J.R. Weertman, "Cyclic softening of ultrafine grain copper", *Materials Science and Engineering A*, vol. 244, pp 145-153, (1998). [https://doi.org/10.1016/S0921-5093\(97\)00689-8](https://doi.org/10.1016/S0921-5093(97)00689-8)
- [6] O. Nejadseyfi, A. Shokuhfar and V. Moodi, "Segmentation of copper alloys processed by equal-channel angular pressing", *Transactions of Nonferrous Metals Society of China*, vol. 25, pp 2571-2580, (2015). [https://doi.org/10.1016/S1003-6326\(15\)63877-8](https://doi.org/10.1016/S1003-6326(15)63877-8)
- [7] R.Z. Valiev and T.G. Langdon, "Principles of equal-channel angular pressing as a processing tool for grain refinement", *Progress in Materials Science*, vol. 51, pp 881-981, (2006). <https://doi.org/10.1016/j.pmatsci.2006.02.003>
- [8] X.Z. Ling, X.J. Jia, H.Y. Shi and W.X. Lei, "Influence of processing temperature on microstructure and microhardness of copper subjected to high-pressure torsion", *SCIENCE CHINA Technological Sciences*, vol. 53, pp 1534-1539, (2010). [doi: 10.1007/s11431-010-3157-7](https://doi.org/10.1007/s11431-010-3157-7)
- [9] E.O. Hall, "The deformation and ageing of mild steel: III discussion of results", *Proceedings of the Physical Society. Section B*, vol. 64, pp 747, (1951). <http://stacks.iop.org/0370-1301/64/i=9/a=303>
- [10] F.H. Torre, A.A. Gazder, C.F. Gu, C.H. Davies and E.V. Pereloma, "Grain size, misorientation, and texture evolution of copper processed by equal channel angular extrusion and the validity of the Hall-Petch relationship", *Metallurgical and Materials Transactions A*, vol. 38, pp 1080-1095, (2007). [doi:10.1007/s11661-007-9103-z](https://doi.org/10.1007/s11661-007-9103-z)
- [11] W.C. Peng, L.F. Guo, W. Lei and Q.H. Juan, "Review on modified and novel techniques of severe plastic deformation", *SCIENCE CHINA Technological Sciences*, vol. 55, pp 2377-2390, (2012). [doi:10.1007/s11431-012-4954-y](https://doi.org/10.1007/s11431-012-4954-y)
- [12] V.M. Segal, "Materials processing by simple shear", *Materials Science and Engineering A*, vol. 197, pp 157-164, (1995). [https://doi.org/10.1016/0921-5093\(95\)09705-8](https://doi.org/10.1016/0921-5093(95)09705-8)
- [13] M. Kamachi, M. Furukawa, Z. Horita and T.G. Langdon, "A model investigation of the shearing characteristics in equal-channel angular pressing", *Materials Science and Engineering A*, vol. 347, pp 223-230, (2003). [https://doi.org/10.1016/S0921-5093\(02\)00589-0](https://doi.org/10.1016/S0921-5093(02)00589-0)
- [14] L.S. Toth, M. Arzaghi, J.J. Fundenberger, B. Beausir, and R. Arruffat-Massion, "Severe plastic deformation of metals by high-pressure tube twisting", *Scripta Materialia*, vol. 60, pp 175-177, (2009). <https://doi.org/10.1016/j.scriptamat.2008.09.029>
- [15] M.S. Mohebbi and A. Akbarzadeh, "Accumulative spin-bonding (ASB) as a novel SPD process for fabrication of nanostructured tubes", *Materials Science and Engineering A*, vol. 528, pp 180-188, (2010). <https://doi.org/10.1016/j.msea.2010.08.081>
- [16] G. Faraji, M.M. Mashhadi and H.S. Kim, "Tubular channel angular pressing (TCAP) as a novel severe plastic deformation method for cylindrical tubes", *Materials Letters*, vol. 65, pp 3009-3012, (2011). <https://doi.org/10.1016/j.matlet.2011.06.039>
- [17] G. Faraji, M. Ebrahimi and A.R. Bushroa, "Ultrasonic assisted tubular channel angular pressing process", *Materials Science and Engineering A*, vol. 599, pp 10-15, (2014). <https://doi.org/10.1016/j.msea.2014.01.069>
- [18] G. Faraji, M.M. Mashhadi, A.R. Bushroa and A. Babaei, "TEM analysis and determination of dislocation densities in nanostructured copper tube produced via parallel tubular channel angular pressing process", *Materials Science and Engineering A*, vol. 563, pp 193-198, (2013). <https://doi.org/10.1016/j.msea.2012.11.065>

- [19] H. Abdolvand, G. Faraji, M.K. Besharati-Givi, R. Hashemi and M. Riazat, "Evaluation of the microstructure and mechanical properties of the ultrafine grained thin-walled tubes processed by severe plastic deformation", *Metals and Materials International*, vol. 21, pp 1068-1073, (2015). [doi:10.1007/s12540-015-5261-5](https://doi.org/10.1007/s12540-015-5261-5)
- [20] D.E. Lozano, R.D. Mercado-Solis, A.J. Perez, J. Talamantes and F. Morales, "Tribological behaviour of cast hypereutectic Al-Si-Cu alloy subjected to sliding wear", *Wear*, vol. 267, pp 545-549, (2009). <https://doi.org/10.1016/j.wear.2008.12.112>
- [21] M.I. Abd El-Aal, "The effect of the pre-homogenization treatment on the fracture characteristics and wear properties of ECAPed Al-Cu alloys", *Materials Science and Engineering A*, vol. 539, pp 308-323, (2012). <https://doi.org/10.1016/j.msea.2012.01.101>
- [22] R.N. Caron, R. G. Barth and D. E. Tyler, "Metallography and microstructures", *ASM Handbook Committee*, vol. 9, pp 1848-1850, (2004).
- [23] H. Abrams, "Grain size measurement by the intercept method", *Metallography*, vol. 4, pp 59-78, (1971). [https://doi.org/10.1016/0026-0800\(71\)90005-X](https://doi.org/10.1016/0026-0800(71)90005-X)
- [24] G. Faraji, A. Babaei, M.M. Mashhadi and K. Abrinia, "Parallel tubular channel angular pressing (PTCAP) as a new severe plastic deformation method for cylindrical tubes", *Materials Letters*, vol. 77, pp 82-85, (2012). <https://doi.org/10.1016/j.matlet.2012.03.007>
- [25] O.F. Higuera-Cobos and J.M. Cabrera, "Mechanical, microstructural and electrical evolution of commercially pure copper processed by equal channel angular extrusion", *Materials Science and Engineering A*, vol. 571, pp 103-114, (2013). <https://doi.org/10.1016/j.msea.2013.01.076>
- [26] J. Yanagimoto, J. Tokutomi, K. Hanazaki and N. Tsuji, "Continuous bending-drawing process to manufacture the ultrafine copper wire with excellent electrical and mechanical properties", *CIRP Annals - Manufacturing Technology*, vol. 60, pp 279-282, (2011). <https://doi.org/10.1016/j.cirp.2011.03.148>
- [27] A. Habibi, M. Ketabchi and M. Eskandarzadeh, "Nano-grained pure copper with high-strength and high-conductivity produced by equal channel angular rolling process", *Journal of Materials Processing Technology*, vol. 211, pp 1085-1090, (2011). <https://doi.org/10.1016/j.jmatprotec.2011.01.009>
- [28] F.H. Torre, E.V. Pereloma and C.H. Davies, "Strain hardening behavior and deformation kinetics of Cu deformed by equal channel angular extrusion from 1 to 16 passes", *Acta Materialia*, vol. 54, pp 1135-1146, (2006). <https://doi.org/10.1016/j.actamat.2005.10.041>
- [29] S.J. Kadri, K.T. Hartwig, in: Z. Horita (Eds.), "Proceedings of the nanomaterials by severe plastic deformation", *Materials Science Forum*, vol. 503-504, pp 1050, (2006). [doi:10.4028/www.scientific.net/MSF.503-504](https://doi.org/10.4028/www.scientific.net/MSF.503-504)
- [30] L. Kommel, I. Hussainova and O. Volobueva, "Microstructure and properties development of copper during severe plastic deformation", *Materials and Design*, vol. 28, pp 2121-2128, (2007). <https://doi.org/10.1016/j.matdes.2006.05.021>
- [31] Z.Y. Zhong, H. Saka, T.H. Kim, E.A. Holm, Y.F. Han and X.S. Xie, "Mechanical properties of copper processed by severe plastic deformation", *Materials Science Forum*, vol. 475-479, pp 3497-3500, (2005). [doi:10.4028/www.scientific.net/MSF.475-479.3497](https://doi.org/10.4028/www.scientific.net/MSF.475-479.3497)
- [32] L.L. Gao and X.H. Cheng, "Microstructure, phase transformation and wear behavior of Cu-10%Al-4%Fe alloy processed by ECAE", *Materials Science and Engineering A*, vol. 473, pp 259-265, (2008). <https://doi.org/10.1016/j.msea.2007.03.072>