

# Severe plastic Deformation of Nanostructured Cu-30%Zn tubes at Increased Temperatures

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**Abstract:** Severe plastic deformation (SPD) methods were developed for producing of metals and alloys with ultrafine grained (UFG) microstructures having high strength. Parallel tabular channel angular pressing (PTCAP) as a noble severe plastic deformation (SPD) method was used to produce ultrafine grained (UFG) and nanostructured Cu-30%Zn tubes. In this paper, the effect of PTCAP process temperature on the deformation microstructures and mechanical properties were investigated using experimental tests. Optical microscopy (OM) and scanning electron microscopy (SEM) were used to evaluate microstructural evolutions and fractured surface analysis. Microhardness and tensile tests were employed to mechanically characterize the PTCAP processed samples. The results showed the strength and the hardness decrease with increasing process temperature up to 100°C, but at 200°C, strength and hardness increase in comparison to that in 100°C. The rise in the strength and hardness of the sample processed at 200°C compared to that at 100°C is because of the partial recrystallization, forming new fine grains with high angle boundaries and twin boundaries. Twinning is dominant deformation mechanism of brass material in order to low stacking fault energy (SFE). Observations revealed that the failure mode in PTCAPed brass was a ductile rupture with the existence of deep dimples. It also indicates that the temperature has no obvious effect on the fracture mood.

**Keywords:** Cu-30%Zn tube, PTCAP, Microstructure, Mechanical properties, Temperature, Ultrafine grained.

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## 1 INTRODUCTION

In the last two decades, methods of severe plastic deformation (SPD) are developed with the aim of improving the microstructure and producing of metals and alloys with ultra fine grain (UFG) microstructures and high strength and superplasticity behavior [1]-[3]. SPD with applying high level of shear strain to material leads to coarse grain of material transform to UFG or nano grain one. Temperature has a crucial rule in this method and can influence the properties of materials with preparing required energy for grain growth and recrystallization. Equal channel angular pressing (ECAP) [3]-[5] due to providing bulk UFG materials that large enough for real structures attracts especial interests.

Several experiments have been carried out exploring the mechanical behaviour and microstructural characteristic of SPD processed materials. Horita et al. studied the mechanical properties of Al alloys produced using ECAP [6]. Microstructures and mechanical properties of aluminum alloys processed by ARB were reported in [7]. Terada et al. studied the microstructure and mechanical behavior of commercial purity titanium severely deformed by ARB process [8]. Characterization of nanostructured pure aluminum tubes produced by TCAP was investigated by Mesbah et al. [9]. The researches above were performed at room temperature although the higher pressing temperature has an indisputable effect on microstructure and mechanical properties. Wang et al. examined the effect of deformation temperature on the microstructure developed in commercial purity aluminum processed by ECAP [10]. They reported that an increase in the deformation temperature caused subgrain size to increase and subgrain shape to become more equiaxed. Effect of process temperature on tensile behavior and microstructure of low carbon steels processed by ECAP were studied in [11]. It declared that due to the rapid recovery process, coarser grains with high angle boundaries were formed by high-temperature processing. Hong-Ying et al. examined the effect of tempering temperature on the microstructure, and mechanical properties of AISI 6150 steel and the result show that the microstructure of tempered sample at 200°C mainly consists of tempered martensite [12].

The parallel tubular channel angular pressing (PTCAP) is being considered as one of the most viable SPD techniques that are capable of fabricating UFG cylindrical metals [13]. PTCAP does not require any special equipment and enables the production of large amounts of UFG tube shape materials. Few techniques such as tubular channel angular pressing (TCAP) [14] and high-pressure torsion twisting (HPTT) [15] and parallel tubular channel angular pressing (PTCAP) have been developed to impose the severe plastic strains to the tubular samples [16]. Among them, the PTCAP process has some advantage over the other techniques including better strain homogeneity and needing lower pressing load [13]. Fig. 1 shows the schematic of

PTCAP. The principle of PTCAP is that a sample, in the form of a tube, experienced two cycles. In the first cycle, it is pressed through a die containing two axisymmetric shear zones, which intersect an angle  $\varphi$ , and diameter of the tube is increased. According to the philosophy of SPD methods, no change in dimension, in the second cycle, tube press back to the initial dimension. Intense plastic straining is introduced to the sample as it passes through the shear zone. Since the final dimension is the same as the primary dimension, repetitive pressings can be undertaken to obtain very high total strain. Though, an extensive study of microstructural features in fcc metals with low to moderate stacking fault energies such as Cu and stainless steel were carried out by various researchers [9], [17]-[22], few effort have been undertaken to investigate the mechanical behavior of them at elevated temperatures. This omission motivated the present investigation. In the present study, microstructural evolutions and mechanical properties of UFG Cu-30%Zn tubes are investigated as a function of the PTCAP pressing temperature.

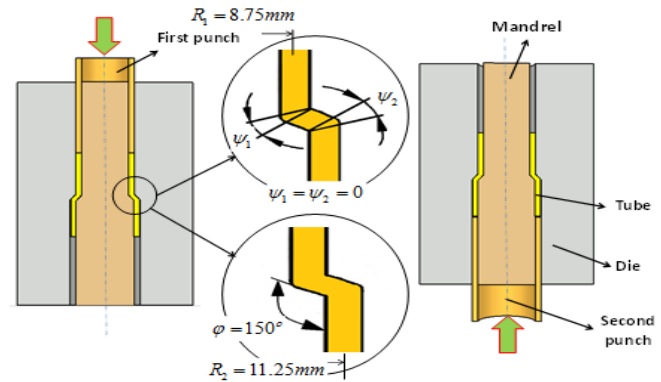


Fig. 1 Schematic of PTCAP and die parameters

## 2 EXPERIMENTAL PROCEDURE

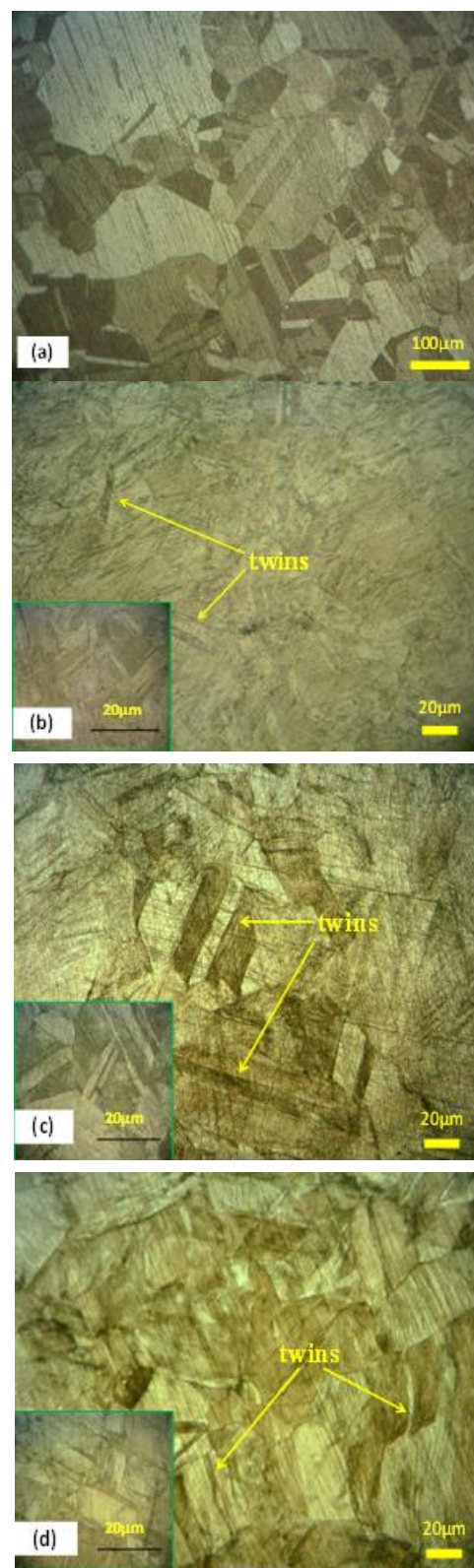
A commercial Cu-30%Zn alloy drawn tubes with a thickness of 2.5mm, the outer diameter of 20mm and length of 40mm were used. Tubes were annealed at 600°C for 1 hour to obtain fully recrystallized homogeneous microstructure. A PTCAP die with parameters shown in Fig. 1 was manufactured [23]. According to usual plasticity formulas and geometry of PTCAP die, total accumulated strain after  $N$  passes can be calculated by following equation [13]:

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

To ensure reduced friction between die and tube, MoS<sub>2</sub> was used as a lubricant [24]. The samples and die were preheated to desired temperature for processing at higher temperatures. Tube samples were processed at room temperature (RT), 100°C and 200 °C. The pressing speed was 10 mm/min in all cases. Tensile tests were carried out on unprocessed, and PTCAP processed samples using SANTAM tensile test machine at a strain rate of  $5 \times 10^{-4} \text{ s}^{-1}$  at RT. These samples were prepared with gage length 10mm and 1mm in width. Conventional metallography method and optical microscopy were organized in order to study of the microstructure of the samples. Before the fractographic tests using SEM, the samples were cleaned for 3 minutes in an ultrasonic bath in ethanol. The Vickers microhardness values of annealed sample and PTCAP process samples were measured by applying a load of 1 kg for 20 s. Six points were determined, and the average was chosen as hardness. To investigate the homogeneity of plastic deformation and mechanism of fracture mode, the fracture surface of the tensile sample was examined using SEM.

### 3 RESULTS AND DISCUSSION

The fully recrystallized homogeneous microstructure of the original annealed material with a mean linear intercept grain size of 70  $\mu\text{m}$  was illustrated in Fig. 2(a). Fig. 2(b)–(d), show the microstructural changes during the PTCAP process in the longitudinal cross section direction at room temperature, 100°C and 200°C respectively. The reduction of grain size after PTCAP proves that current PTCAP process is effective in refining the microstructure of the brass alloy. As expected, microstructure after the process is significantly different from the initial state. It was seen that after PTCAP process at RT, elongated fine grain appeared which is in agreement with previously reported results [25]–[27]. Naturally, the size of grains in the process at high temperature is coarser in comparison with RT [28] and the grain shape become more equiaxed. Significant noting is the grain size at 200°C a little finer than 100°C. It can be concluded that the process temperature cannot provide atomic mechanism and recrystallization at 100°C, but at 200°C, new strain-free grains nucleate, grow and cause to produce finer grains. As is mentioned [29], the minimum temperature for the brass alloy to occurrence recrystallization is  $\sim 200^\circ\text{C}$ . Though, this temperature may affect the primary straining and the initial grain size. Significant noting is plenty of twinnings exists in all processed specimens as shown in Fig. 2. It demonstrated that activation of deformation twins competes with dislocation slip and affects the mechanical response of the material.



**Fig. 2** Optical micrograph of (a) annealed state and PTCAPed brass at (b) RT, (c) 100°C and (d) 200°C

As studied in [19] The dominant deformation mechanism in 70/30 brass is twinning because of its low stacking fault energy (SFE). Brass deforms by both slip and twinning mechanisms but because of lower activation energy of twinning, the mechanical twinning in low SFE materials is preferred to slip mechanism [30]. Because of the activation of more slip systems at high temperature, it is supposed that an increase in PTCAP temperature decreases the amount of twinning. However, remarkable noting is the presence of more twinings in the PTCAPed specimens at increased temperatures (Fig. 2). PTCAP processing at higher temperatures leads to the formation of more coarse grains than that of lower temperatures. Meyers et al. [31] illustrated that the necessary stress for activation of twinning decreased by increasing the grain size and, as a result, twinning takes place more easily in coarser grains. Indeed, because of the high temperature and possibility of simultaneous recovery, recrystallization or grain growth co-occurred with twinning, these twinings may not be called mechanical twinning with certainty.

Fig. 3 represents engineering stress-strain curves obtained for the samples processed at three temperatures of RT, 100°C and 200°C. Engineering stress-strain curves of the coarse-grained annealed brass alloy and the PTCAP processed alloy with a different temperature are compared. According to diagram curves, after the process, different curve appeared compared with annealed one. Apparently, after passes in all temperatures yield and ultimate strength were significantly increased whereas elongation to failure was decreased that similar to other SPD methods [11]. Maximum strength takes place at RT after deformation. Surprisingly, the tensile strength increases from 350 MPa to ~580 MPa. However, the elongation was dramatically decreased about 75 % in all cases. This indicates a very distinct loss of formability and suggests that strain hardening mechanism alone could not explain the present phenomenon. Cu-30%Zn has a very small SFE ( $14 \text{ mJ.m}^{-2}$ ), so deformation twinning frequently occurs during its plastic deformation and results in substantial work hardening [32]. The strengthening can be attributed to two different physical mechanisms. The most common clarification is the reduction of free slip line distance due to the presence of twins. This is similar to the Hall-Petch effect: the twin matrix interface acts as a barrier to dislocation pile-up formation, and the strain accommodation is more complex due to the reduction of the mean slip length. Fig. 4 illustrates the changes in mechanical properties of the PTCAP 70/30 brass. Tensile strength reduced to ~478MPa at PTCAP-100°C. As expected, with increasing the temperature, strength should be reduced [33]. Outstanding noting is more strength was occurred at 200°C process than 100°C. As declared in Fig. 2, existence of more twinings in PTCAP-200°C may also contribute to the restriction of the dynamic recovery because the dislocations is hard to annihilate at the twin boundary with perfect coherency [34], and it leads to higher strength.

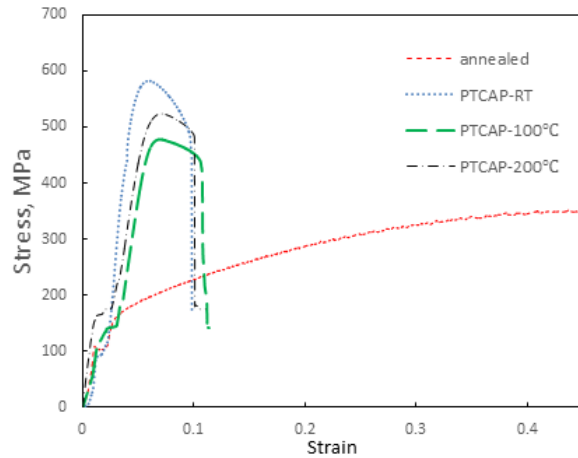


Fig. 3 Engineering stress-strain curves of unprocessed and PTCAP processed samples at different temperatures

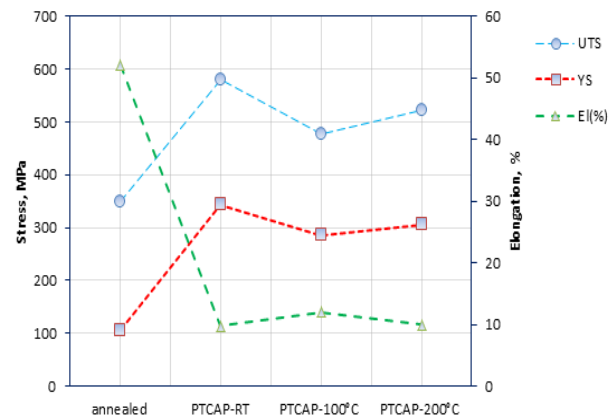


Fig. 4 Variation of UTS, yield strength and elongation as a function of process temperature

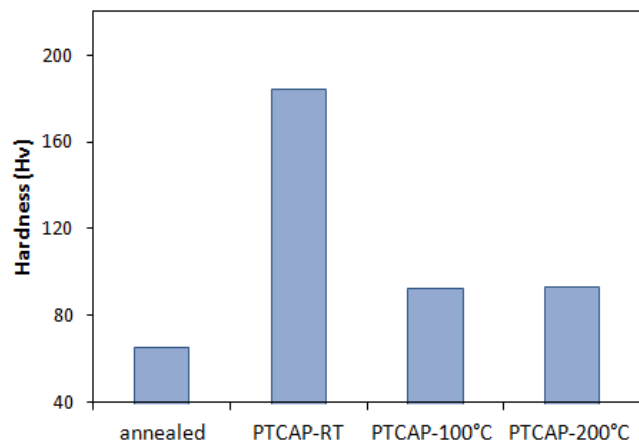
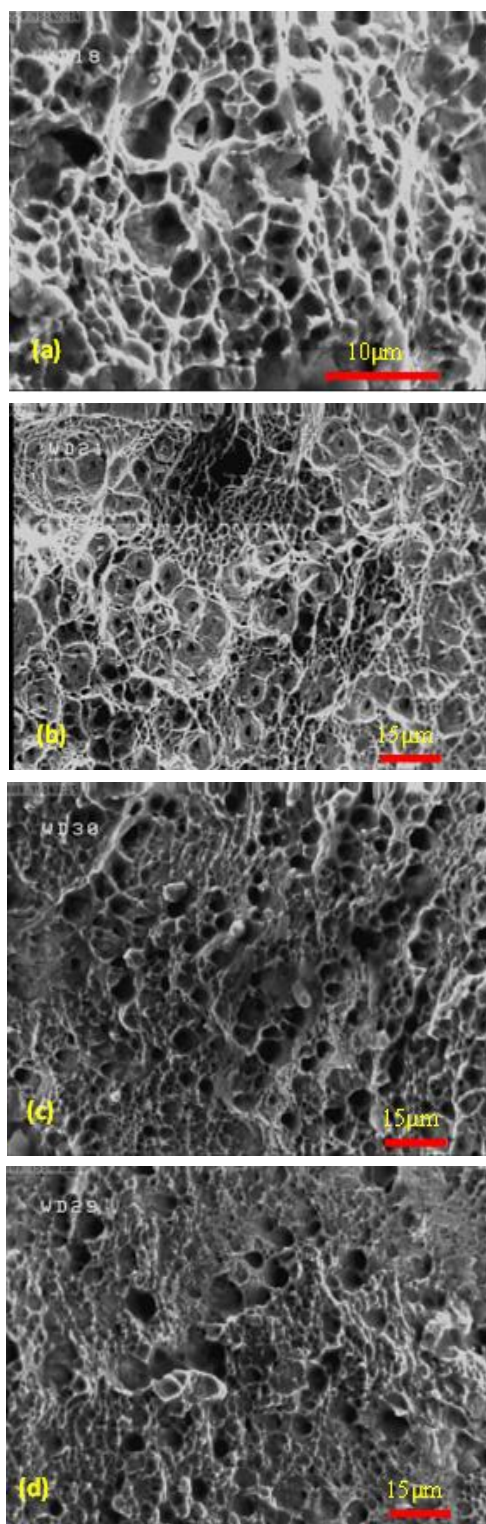


Fig. 5 Microhardness value vs. process temperature

The results of measurements of the hardness versus to process temperature were shown in Fig. 5. A remarkable increase in hardness, which was almost three times greater



than that of the initial value, was achieved after one pass of PTCAP at RT.



**Fig. 6** SEM micrographs of the fractured surface of (a) annealed and PTCAPed samples at (b) RT, (c) 100°C and (d) 200°C

The hardness of as received brass tube was ~65HV and after one pass reached to ~185HV. For the high temperature-UFG

samples, the microhardness decreased abruptly to about 92Hv. The intensification in the brass hardness may be attributed to the strain hardening and grain refinement as indicated in the well-known Hall–Petch relationship. The hardness increased notably as the grain structure was refined, and the dislocations were accumulated significantly after the PTCAP process. As shown in the figure, the hardness of 200°C processed tube is a little more than 100°C processed specimen that is in good agreement with tensile properties.

SEM pictures presented in Fig. 6 are the fractured surface of tensile test samples to correlate the fracture characteristics with the ductility properties. It reveals that all the specimens are fractured in a ductile manner, consisting of well-developed dimples over the entire surface. The average dimple size for the initial material is ~6 μm, and it gets reduced to less than 2 μm after PTCAP process. This drastically decrease in dimple size may be due to the grain refinement and work hardening, which occur during severe plastic deformation of the samples as reported in the earlier literature [35]-[37]. The fractures of the brass are circular due to the typical cracking of bottom-cone characteristic. The spiral edges on the fractures indicate their helical way of formation. The surface crack orientation changes as well as the mechanism of cracking due to the change of the state of stresses to local shearing in the planes of maximum stresses at an angle of 45° from the direction of tension. Fig. 6 (c) and (d) exhibit the morphology of fracture surface for high temperature deform samples. It includes of deep dimples with size of ~5 μm that is indicating ductile fracture mode. It also indicates that the temperature has no obvious effect on the manner of fracture.

## 7 CONCLUSION

The microstructure and mechanical properties of a Cu-30%Zn severely deformed by the PTCAP process at room temperature, 100°C and 200°C were investigated. The key findings are summarized below:

- UFG microstructure was formed in the Cu-30%Zn highly deformed by the PTCAP process.
- Because of low stress activation for twinning, plenty twinning was observed in the samples processed at enhanced temperatures.
- Samples processed at 200°C showed higher strength in comparison with that processed at 100 °C.
- Hardness increase to ~185 Hv from an initial value of ~65 Hv at RT and after processing at high temperature reaches to ~92Hv.
- Ductile fracture mood was happened in all samples.

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