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# Production of nanostructure copper by planar twist channel angular extrusion process



M. Shamsborhan <sup>a, \*</sup>, M. Ebrahimi <sup>b, \*\*</sup>

- <sup>a</sup> Department of Engineering, Mahabad Branch, Islamic Azad University, Mahabad, Iran
- <sup>b</sup> Department of Mechanical Engineering, University of Maragheh, Maragheh, Iran

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#### ABSTRACT

This study challenges experimentally, the feasibility of new severe plastic deformation method entitling planar twist channel angular extrusion (PTCAE) for fabrication of nanostructure pure copper. The results indicated, deformed copper has 71%, 69% and 113% higher yield strength, ultimate tensile strength and hardness magnitude as compared to the as-received condition. Additionally, the fractography of tensile sample showed the mechanism alteration from multiple dimples to cleavage planes, associating the production of brittle type material. Moreover, the reduction of bulk formability index of the processed copper in comparison with the initial condition confirms the decrease of material ductility due to the strain hardening and grain refinement phenomena. The grain size of about 270 nm is also attained after the single pass processing. Eventually, a grain size correction coefficient is proposed to the strength to hardness ratio as a rule of thumb for estimation of strength by means of hardness irrespective of the grain size of the pure copper material.

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#### 1. Introduction

Ultrafine grain (UFG) and nanostructure (NS) metals and alloys are recently well established because of their significant and unique properties in comparison with conventional coarse grain (CG) counterparts [1,2]. It is widely accepted that one of the most prosperous methods of top-down approach for achievement of the UFG and even NS materials is severe plastic deformation (SPD) method in which very intense plastic strain is imposed to a specimen without any considerable change at the cross-sectional area [1–4].

A comprehensive study on the various existing severe plastic deformation literature reveals that there are some concerns about their capability on the various industrial and medical applications. These worries return to the problems, including the inability of the method to scale-up, complexity of die geometry and discontinuity of the material production, especially in the form of sheet and strip [5–8]. Accordingly, several SPD techniques and methods have been proposed, developed and experimented for elimination of these

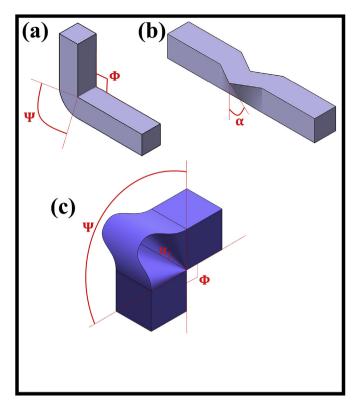
E-mail addresses: m.shamsborhan@iau-mahabad.ac.ir (M. Shamsborhan), ebrahimi@maragheh.ac.ir (M. Ebrahimi).

limitations such as equal channel angular pressing (ECAP) [9], elliptical cross-section spiral equal-channel extrusion (ECSEE) [10], equal channel forward extrusion (ECFE) [11], accumulative back extrusion (ABE) [12], multi axial forging (MAF) [13], repetitive upsetting (RU) [14], planar twist extrusion (PTE) [15], simple shear extrusion (SSE) [16], tubular channel angular pressing (TCAP) [17], constrained groove pressing (CGP) [18] and accumulative roll bonding (ARB) [19]. Recently, the authors have offered a new SPD method entitling planar twist channel angular extrusion process in order to conquer the as-mentioned SPD constraints as far as possible [20–22]. In other words, this method has been introduced by this duty.

The schematic representation of PTCAE method and efficacious processing parameters have been shown in Fig. 1 beside the both PTE and ECAP methods. During this process which combines two ECAP and PTE methods in one deformation zone, intensive shear strain is simultaneously imposed to the three orthogonal faces of the work-piece. According to the previous investigation of PTCAE method, the main advantages of this process in comparison with the other SPD methods are simultaneous imposing of plastic shear strain in three perpendicular planes in one single deformation zone, small volume of plastic deformation zone, less required pass numbers to attain material with the saturated grain size, low required extrusion load due to the reduction of contact surfaces

<sup>\*</sup> Corresponding author.

<sup>\*\*</sup> Corresponding author.



**Fig. 1.** Schematic representation of a) equal channel angular pressing, b) planar twist extrusion and c) planar twist channel angular extrusion.

between the sample and the die and eventually, processing efficiency. The method details have been found elsewhere [20–22]. In this research, PTCAE die which is suitable for induction of extremely large strain to the sample in the single deformation zone has been designed and manufactured. Afterwards, mechanical properties and microstructural evolution of one pass processed copper have been experimentally investigated.

#### 2. Experimental procedures

All experimentations were performed on annealed commercial pure (CP) copper with a length of 70 mm and square cross-section of 10 mm  $\times$  10 mm. PTCAE process was conducted using a split die with channel angle, outer corner angle and planar twist angle of 90°, 90° and 35°, respectively. This process was carried out at ambient temperature with a ram speed of about 1 mm s $^{-1}$  and application of molybdenum disulfide as lubricant [23]. Fig. 2 represents the used PTCAE die and the copper sample after the first pass. The effective plastic strain during one pass of PTCAE process with the die parameters used for this study is equal to 1.95 [20–22], which is higher than from the conventional equal channel angular pressing  $((1/\sqrt{3})|2\cot(\Phi+\Psi/2)+\csc(\Phi+\Psi/2)]$  with the die

configurations of  $\Phi=90^\circ$ ,  $\Psi=90^\circ$ ) [9] and also, planar twist extrusion  $((2/\sqrt{3})\tan(\alpha))$  with the die configuration of  $\alpha=35^\circ$ ) [24] processes. It is better to mention that the magnitudes of strain for each pass of ECAP and PTE processes are equal to 0.577 and 0.808, respectively.

After the process, various mechanical and microstructural examinations were carried out on the CP copper sample to investigate the capability of the method in comparison with the other SPD processes. Therefore, Vickers microhardness (HV) measurements with a load of 300 gf and dwell time of 10 s were accomplished on a plane normal to the extrusion direction (Newage MS-HMVG20SV microhardness testing system). It should be noted that 16 HV tests with the step of 2 mm were recorded and the average magnitude was reported. In addition, room temperature tensile test by means of Instron 5582 universal tester in accordance with the ASTM E8 was done to evaluate tensile strength and elongation of the extruded copper sample as compared to the annealed one. It is better to know that the tensile testing sample was prepared at the middle part of the billet by means of CNC wire cut electrical discharge machining with its tensile axis parallel to the extrusion direction. Two tensile tests were carried out for each circumstance at the constant strain rate of  $3 \times 10^{-3}$  s<sup>-1</sup> and the average magnitudes were recorded. Afterwards, scanning electron microscopy (SEM) using Jeol JSM-IT100 instrument was utilized to size up breakage surface morphology of the tensile samples. Finally, the samples were respectively subjected to SEM and Jeol JEM-ARM200F transmission electron microscopy (TEM) at the crosssectional section for the samples of the as-received and the deformed conditions to confirm the grain refinement of pure copper material after the PTCAE process. To attain TEM image, thin foil was prepared by use of mechanical grinding and polishing and also, twin jet electropolishing machine at a voltage of 60 V and a temperature of -35 °C.

#### 3. Results and discussion

The engineering stress – strain curve of the pure copper sample before and after the PTCAE process has been represented in Fig. 3. By considering that there is not any well determined yield occurrence at the tension behavior of Cu samples before and after the process, the yield strength of the 0.2% offset was utilized. It is apparent that imposing severe plastic deformation by planar twist channel angular extrusion leads to the improvement of both yield strength and ultimate tensile strength and to the reduction of elongation to failure. It means that PTCAE process has significant influence on the mechanical properties of pure copper. For the deformed material, the strength increases dramatically with strain up to the peak point and then, it decreases gradually with strain increment till to the breaking. The quantitative analyses of the results reveal that 71% and 69% higher yield strength and ultimate tensile strength and a 48% lower elongation to failure are achieved after the first pass of PTCAE process in comparison with the initial condition. It should be useful to define a formability index for the



Fig. 2. The utilized PTCAE die and deformed copper sample after the first pass.

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