



# Experimental study on pure copper subjected to different severe plastic deformation modes



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## ABSTRACT

Equal channel angular pressing (ECAP), elliptical cross-section spiral equal-channel extrusion (ECSEE) and torsion deformation (TD) have been proven as efficient SPD methods for grain refinement. In order to compare the characteristics of grain refinement by these technologies, experimental researches on microstructure evolution and mechanical properties have been conducted by optical microscopy (OM), transmission electronic microscopy (TEM) and microhardness tests. OM observation shows a significant decrease and non-uniformed distribution in grain size on the cross-section of the processed samples, which agrees well with the result of strain distribution. TEM observation shows a similar refinement process undergoing the forming of shear bands, dislocation forest, large-angle grain boundaries and subgrains. The different morphological structures by TEM have been discussed in terms of the effect of deformation modes including bending–torsion, extrusion torsion and pure torsion on microstructure evolution. Microhardness distribution of pure copper after 6-passes deformation agrees well with the microstructure observed by OM. However, ECAP is different with ECSEE and TD in microhardness distribution along the radial and circumferential directions.

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## 1. Introduction

Nanostructured metals with the structural feature of less than 100 nm in at least one dimension are usually obtained in alternative ways: a two-step approach, such as inert gas condensation [1,2]; a one-step approach, such as severe plastic deformation (SPD) [3]. In fact, the two-step approach often have defects in producing nanostructured metals, such as porosity and cracks, that lead to very low ductility [4]. In comparison, the one-step approach can produce 100% dense and defect-free nanostructured metals. Based on the pioneering works on production of ultrafine grained structures by SPD processing [5,6], many SPD techniques have attracted wide attention and experienced further development. These techniques are equal channel angular pressing (ECAP), torsion deformation (TD) and elliptical cross-section spiral equal-channel extrusion (ECSEE) (Fig. 1).

For ECAP process, the die is made up of two channels with an equal cross-section intersecting at an angle that can alter typically between 90° and 135° [7–9]. For TD process, the samples are twisted along the axis [10]. Unlike above SPD methods, the die structure of ECSEE is composed of three channel regions: round–ellipse cross-section transitional channel  $L_1$ , elliptical cross-section

torsion transitional channel  $L_2$  and ellipse–round cross-section transitional channel  $L_3$ . According to the principle of ECSEE, the deformation can be identified as two basic forms: compression deformation, corresponding to the round–ellipse/ellipse–round cross-section transitional channel and torsion deformation, corresponding to the elliptical cross-section torsion transitional channel. Significant reduction in grain sizes is attributed to the high pressure accompanying torsion process during channel  $L_2$ . These SPD process leads to the presence of a high fraction of high angle boundaries ( $> 15^\circ$ ), which contributes to a significant improvement of the mechanical properties, offering great potential for the advent of the superplastic behavior in a given material.

More in-depth understanding of fundamental mechanisms of different SPD methods probably brings us closer to the revolutionary use of nonmaterial for structural and functional applications. Special emphasis is laid on the relationship between microstructure features and properties [11]. Mishra et al. [12] obtained that the first few passes of ECAP are the most efficient in grain refinement and the microstructure becomes more equiaxed as the number of passes increases by undergoing five successive stages: homogeneous dislocation distribution; elongated sub-cell formation; elongated subgrain formation; break-up of subgrains into equiaxed units; sharpening of grain boundaries and final equiaxed ultrafine structure. Li et al. [13] conducted experimental and simulated study on the micro-structural evolution subjected to combined tension–torsion deformation for pure copper, and

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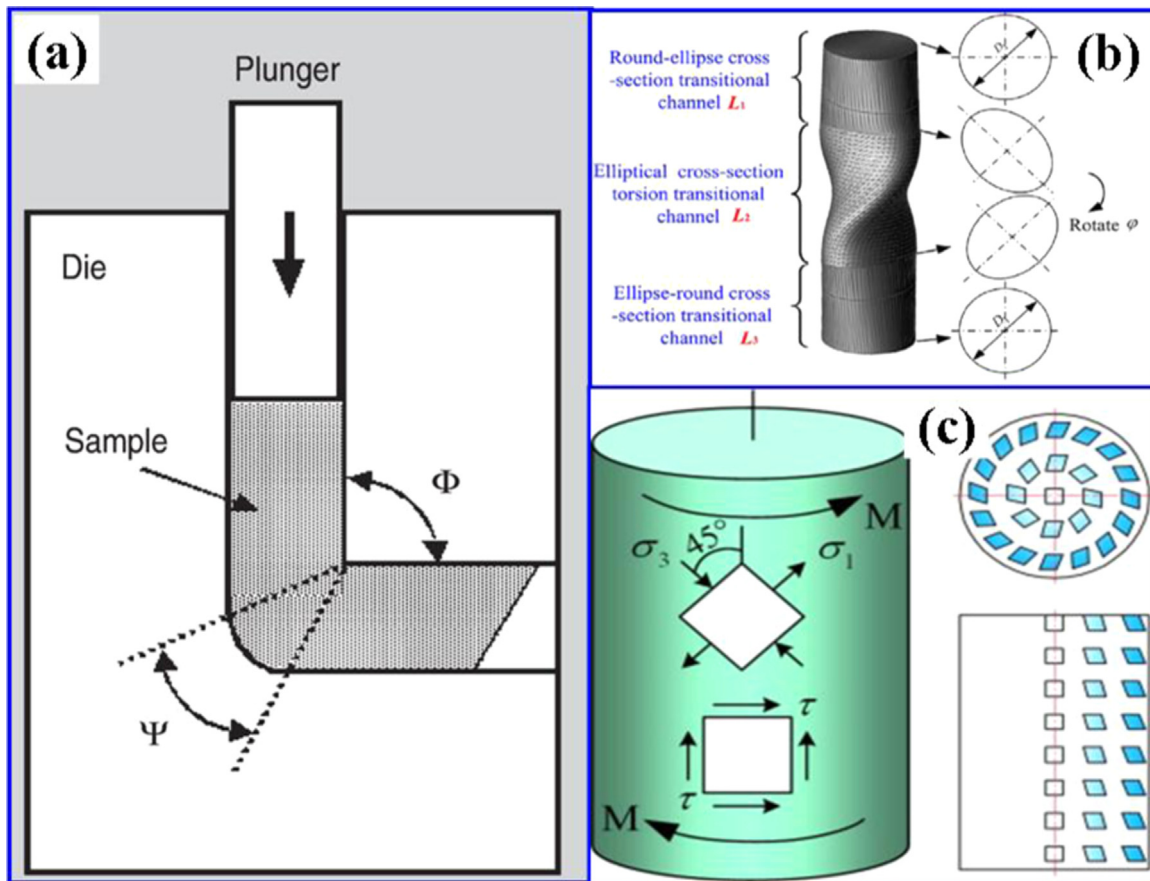


Fig. 1. Three candidate SPD techniques in the study: (a) ECAP [11]; (b) ECSEE [14]; (c) TD [10].

found that a very obvious strain gradient develops on the cross-sections of specimens. Besides, the micro-voids proliferate and grow with the increasing of torsion strain, but the volume of the voids decreases, and voids tend to aggregate when a critical torsion strain is reached. Wang et al. [14] maintained that severe deformation of ECSEE contributed much to the improvement of hardness and refinement of microstructure in the shear stress suffered area of pure copper.

However, scarce attention has been paid on the comparison of different SPD techniques. Some exceptions can be found in the study of Stolyarov et al. [15]. These authors compared the microstructure features produced by ECAP and HPT processes including those obtained by a combination of both (i.e., ECAP followed by HPT). Besides, Stolyarov et al. also studied the effect of deforming Ti-alloys under ECAP combined with HPT at high temperature conditions. Unlike the above studies, the aim of the present study is to compare the microstructural features of deformed pure copper produced by ECAP, TD and ECSEE at room temperature. Also the aim of the present research is extended to evaluate the effect of deformation modes (bending–torsion, extrusion torsion and pure torsion) on the microstructure evolution.

## 2. Experimental materials and procedures

### 2.1. Experimental materials

The chemical composition of the cylindrical commercial copper samples (99.7% in wt%) for the study was 0.001P, 0.002Bi, 0.002Sb, 0.002As, 0.005Fe, 0.002Ni, 0.1Pb, 0.002Sn, 0.005S, 0.005Zn, and 0.02O. With the aim of obtaining a full uniform microstructure, the

experimental samples were preconditioned by annealing at 650 °C for 2 h, and then by furnace cooling to obtain the grain size of 100 μm in Fig. 6a. Those samples were machined with a diameter of 10 mm and length of 30 mm for ECAP and ECSEE.

### 2.2. Experimental deformation procedures

It is important to guarantee that the same equivalent plastic strain is imposed on the billets after one-pass ECAP, ECSEE and TD based on the relationship of equivalent plastic strain and microstructure evolution [16–18]. ECAP was designed with an internal angle ( $\Phi$ ) of 90° and a curvature angle ( $\Psi$ ) of 34° to receive the equivalent plastic strain of  $\sim 1$  after every repetitive extrusion. To obtain the same equivalent plastic strain, the die cavity parameters of ECSEE were set as follows [14]:  $D_1=10$  mm,  $L_1=7$  mm,  $L_2=10$  mm,  $L_3=7$  mm,  $\phi=120$  and  $m=1.55$  ( $m$  was the ratio of major-axis and minor-axis length of the elliptical cross-section). The geometric sketch of samples for TD was shown in Fig. 2, where the equivalent plastic strain is calculated as  $\sim 0.5$  by one-cycle torsion. Therefore, based on the principle of equal equivalent plastic strain, two-cycle torsion is defined as one-pass TD.

The ECAP test was conducted at room temperature from 1 to 6 passes using route Bc with the extrusion speed of 1 mm/s and lard lubrication, and the detailed description for deformation routes of ECAP can be consulted in the references [19–21]. The deformation procedures of ECSEE were the same as those of ECAP. TD tests were achieved in wire torsion testing machine XC-10. The torsion rate is 0.96 turns/min equivalent to a linear velocity of 1mm/s at room temperature.

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