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BACKPRESSURE AT ECAP AS A WAY FOR DECREASING GRAIN SIZE AND INCREASING DUCTILITY OF UFG MATERIALS

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The investigation of the backpressure effect during equal-channel angular pressing (ECAP) on formation of ultrafine-grained (UFG) structure in pure copper has revealed that the increased hydrostatic pressure can contribute to formation of a more homogeneous microstructure with finer grains. ECAP with backpressure also exerts a favorable effect on strength and ductility characteristics of the UFG material.

Introduction

Up to the present the problem of processing the defectless samples with homogenous UFG structure by ECAP has not been solved completely. The main not overcome difficulties are the presence of unstable and insufficiently high hydrostatic pressure (HP) in the zone of deformation at pressing of piece samples and the using of radii in the site of channel interfacing. These factors lead to increased strain non-uniformity and, as a consequence, structure heterogeneity [1]. It is known [2] that the increase of HP promotes dislocation slip and concurrently suppresses the recovery process. For example, the increase of HP in the process of direct pressing is used for processing billets with a finer structure and without external and internal cracks [1].

The increase of HP at ECAP in the case of the minimum radii of channel interfacing leads to a significant reduction of the zone of deformation and makes the process of deformation more uniform [3]. One can expect that using backpressure at ECAP for increasing HP with the radii of channel interfacing of about zero would be an efficient tooling both for formation of a finer homogeneous UFG structure and production of defectless samples [4].

In this connection the goal of the present paper is to investigate the influence of high HP on features of formation of UFG structure and mechanical properties for pure copper.

Experimental procedures

The coarse-grained (CG) copper of 99.8% purity was taken as initial material. The billet, 20 mm in diameter and 100 mm in length, was pressed in a die tooling under constant backpressure. The angle of channel intersection was 90° and the radius of channel interfacing was close to zero (Fig. 1). ECAP was generated using a hydrostatic 1.6 MN force press with the traverse rate of 6 mm/s. Pressing was performed at room temperature by Route C, at which after each pass the billet was turned around its longitudinal axis through an angle of 180° [4]. The samples were subjected to 2 and 16 cycles of ECAP both with backpressure and without the same.

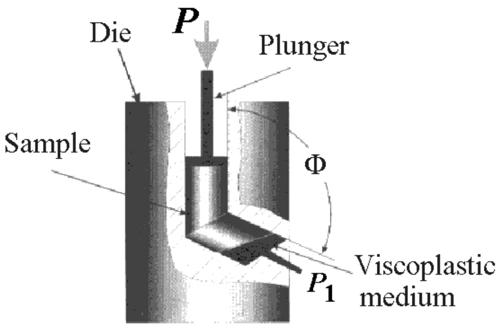


Fig. 1. Scheme of ECAP facility: P – load, P_1 – backpressure, Φ – angle between channels

Microstructure was attested by using a transmission electron microscope JEM-100. The grain size was determined by TEM images of the microstructure at 20000× and more magnification. Electron diffraction patterns were taken from the foil area of 1 μm^2. The surface of the deformed and fractured samples was investigated by means of the LEO 1530 FEG-SEM at an accelerating voltage of 1 kV.

The mechanical tensile properties of the samples at room temperature were measured using the samples with a gauge section of 0.2 × 1.5 × 2.5 mm cut out along the axis of pressing on the universal dynamometer at the initial strain rate of $2\cdot10^{-3}$ s^{-1}. The measuring error was ±5%.

Results and discussion

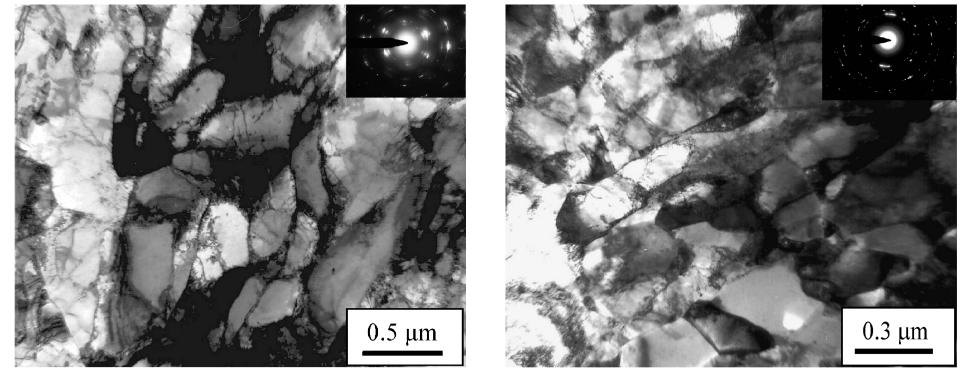
Electron microscopy

The initial annealed copper (99.98%) had a coarse-grained structure with a mean grain size of about 70 μm.

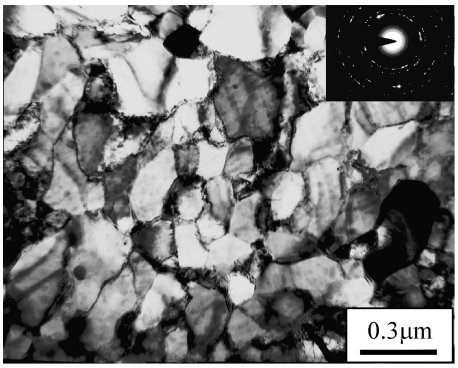
Electron microscopic studies testify that at ECAP of pure copper there occurs essential transformation of structure with increasing strains. The common features of these transformations are an essential decrease in structure component size and growth of elastic microdistortions of a crystal lattice. The results of X-ray structural analysis methods given in [5] also confirm this statement.

A fragmented structure with low density of dislocations inside of fragments and a mean fragment size of about 0.4 μm was formed (Fig. 2,*a*) in the samples subjected to 2 pass ECAP with backpressure. Fragment boundaries look like wide dark bands. According to [6] the density of dislocations at such boundaries is

10^{10}–10^{11} cm^{-2}. The Selected Area Electron Diffraction (SAED) pattern testifies to the presence of low and moderate angle misorientations in such a structure and essential strain stress inside of fragments [6]. Evidently, fragments with high density of defects at boundaries and a small amount of dislocations within the fragment volume are formed at this stage of ECAP.



a *b*



c

Fig. 2. TEM micrographs and corresponding SAED of Cu: *a* – 2 passes of ECAP with backpressure, *b* – 16 passes of ECAP without backpressure, *c* – 16 passes of ECAP with backpressure

After 16 passes of ECAP without backpressure the structure of copper is a bit heterogeneous and its grains are slightly elongated. The mean grain size is 0.28 µm. Grains with separate dislocations inside of them occupy the bulk of the structure volume. Azimuth spreading of spots observed on the SAED pattern testifies to the presence of internal stresses in grains. Formation of high-angle misorientations is typical of such structure that is confirmed by the presence of spots with large azimuth distances (Fig. 2,*b*).

The using of backpressure at ECAP with 16 passes leads to formation of homogeneous UFG structure with equiaxed grains having a mean size of 0.19 µm (Fig. 2,*c*). As seen on the structure image, the grain boundaries (GB) consist of defects in the form of dark bands without extinction contours. Some grains possess a spreading of the extinction contours. It testifies to the non-equilibrium of GB structure with high density of defects. In comparison with material after 16 passes of ECAP without backpressure the SAED of this state contains a larger amount of spots uniformly arranged on the circles. Such an appearance of the SAED is typical of structures with predominantly high-angle misorientations and enhanced supplemental energy [8].

The electron microscopic data obtained show that the using of high HP in the centre of deformation at ECAP with 16 passes leads to a more intensive multiple dislocation slip and formation of a more disperse, homogeneous and equiaxed structure. At the same time, the using of backpressure also contributes to the formation of more non-equilibrium GB with high-angle misorientations and significant energy accumulated during the process of ECAP. All these features of strongly deformed structure essentially influence the nature and deformation behavior of the material.

Mechanical tests

Table shows that ECAP leads to a significant growth of strength properties and decreases essentially ductility of the material. After two passes of ECAP the ultimate tensile strength is increased by a factor of 2.4 and the ductility is dropped by a factor of four as compared to the CG copper. Such changes in the properties occur due to the high density of dislocations inside boundaries of fragments and suppression of dislocation slip owing to small size of fragments. Weak strain hardening at tension and sharp drop of ductility are typical of materials with strongly fragmented structure [6] and one can observe all these in the investigated pure copper (Fig. 3).

The increase in the number of passes from 2 to 16 improves both the strength and ductile characteristics of copper. As compared to the CG copper the strength of copper subjected to ECAP with 16 passes without backpressure is increased 3 times up to 440 MPa and ductility is decreased by a factor of 2.7 down to 15%. Using of backpressure at ECAP with 16 passes leads to the increase of not only the strength up to 470 MPa but also the ductility up to 25% as compared to the material after ECAP without backpressure (Table).

Table

Results of mechanical tests at 20°C and tensile rate $2.5 \cdot 10^{-3}$ s^{-1} for ECAP copper (b/p – backpressure, YS – yield stress, UTS – ultimate tensile stress, El – elongation)

State of material	YS, MPa	UTS, MPa	El, %
Initial Cu	100	145	40
Cu 2 with b/p	340	350	10
Cu 16 without b/p	420	440	15
Cu 16 with b/p	440	470	25

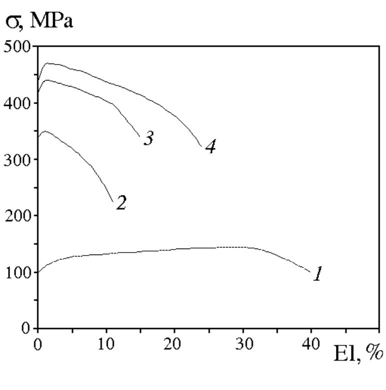
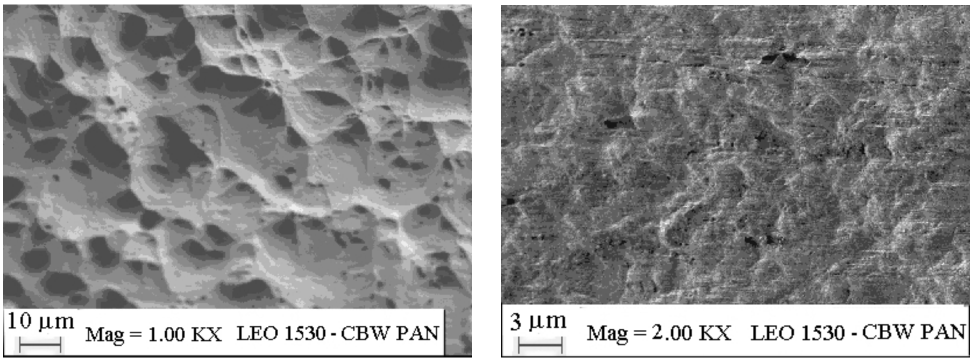


Fig. 3. Dependence curves of flow stress on relative elongation for copper: *1* – CG, *2* – ECAP 2 with backpressure, *3* – ECAP 16 without backpressure, *4* – ECAP 16 with backpressure

So, grain refinement and formation of non-equilibrium structure in the material after ECAP regularly result in the increase of its strength. Whereas the increased ductility observed at ECAP with backpressure in the case of a large number of passes being a bit unexpected. However, a similar effect – the combination of high strength and ductility in UFG copper subjected to high-pressure torsion was revealed recently [7]. Such a behavior is caused by the presence of non-equilibrium grain boundaries in the structure that accelerates dynamic processes at GB [8] Moreover, non-equilibrium GB with high dislocation density can also be responsible for acceleration of GB sliding [9]. The increase in ductility from 15 to 25% in copper samples subjected to ECAP with backpressure is evidently connected with the change in the mechanism of deformation for UFG material. Apparently, due to the large degree of non-equilibrium revealed by TEM studies (see SAED patterns in Figs. 2,*b* and *c*) the contribution of GB to the total deformation is increased.

The high plasticity of this material is also confirmed by the fractographic observations (Fig. 4), where specific dimple relief with the extended edges characteristic of ductile fracture was observed. Zones of deformation localization in the form of intersecting bands are observed on the surface of the sample after tension. These bands are arranged at an angle of 45° with the axis of tension. Similar zones were revealed on the surface of samples deformed at superplasticity and resulted from cooperative GB sliding [10]. So, fractographic studies also confirm the change in the mechanism of deformation.



a *b*

Fig. 4. Fracture (*a*) and deformation relief (*b*) of ultrafine-grained Cu after mechanical test

Thus, the using of backpressure at ECAP contributes to the formation of homogeneous structure with finer grains. EACP with backpressure exerts a favorable effect on strength and ductility increase of the investigated material, as compared to the process without backpressure.

Conclusion

1. The using of backpressure at multiple cycles of ECAP treatment leads to a more efficient grain structure refinement with mainly high-angle misorientations. In case of similar number of passes equal to 16 the using of backpressure leads to the decrease in the grain size from 0.28 to 0.19 μm, the increase in UTS from 440 up to 470 MPa and ductility from 1 5 to 25%.

2. Investigations have shown that after 2 passes of ECAP with backpressure a fragmented structure with a fragment size of about 0.5 μm is formed. The strength of the deformed copper is increased 2.4 times (UTS = 340 MPA), whereas its ductility is decreased by a factor of 4 (El = 10%) as compared to the coarse-grained material.

3. The increase of the number of passes from 2 to 16 at ECAP with backpressure leads to transformation of the fragmented structure with low-angle misorientations to the homogeneous UFG structure with high-angle misorientations and the mean grain size of about 0.2 μm. This results in the increase of both the strength and the ductility, that being an extraordinary experimental fact. As compared to the coarse-grained copper, the strength of copper after 16 passes with backpressure is increased 3.2 times (UTS = 470 MPa), while the ductility is dropped only by a factor of 1.6 (El = 25%).

Acknowledgements

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