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Effect of SPD on the structure formation and mechanical properties of the Cu-10% Zn alloy

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Abstract. In this paper, we report on the microstructure characteristics and strength properties of the Cu-10 wt.% Zn alloy subjected to equal-channel angular pressing (ECAP) and subsequent flat rolling (FR), in order to form an ultrafine-grained (UFG) structure and enhance the strength properties. It is shown that the formed subgrain structure increases the strength characteristics. An increase in the degree of reduction at FR resulted in an increase in the dislocation density and also in an increase in strength. The presence of high-angle grain boundaries increases the ductility of the alloy.

1. Introduction

Today, considerable scientific interest in structural bulk ultrafine-grained (UFG) materials is based on the fact that their mechanical, physical and performance properties differ considerably from the properties of coarse-grained analogues. Features of the structure of UFG materials (grain size, fraction of high-angle boundaries, etc.) are determined by methods of production and have a significant impact on their properties. The UFG structure of the materials is formed by severe plastic deformation (SPD) methods. Equal-channel angular pressing (ECAP) [1, 2], high pressure torsion [3, 4], screw extrusion [5], etc. which belong to the SPD methods are widely applied in scientific research. The SPD forms the UFG structure and increases the density of defects. As a rule, an increase in strength occurs along with a decrease of ductility. Consequently, low ductility becomes a critical limitation in the application of these metals.

The aim of the conducted research was to obtain an increased strength and sufficient ductility in the Cu-10 wt.% Zn alloy by means of ECAP and subsequent rolling.

2. Experimental

The material used in this work is the Cu-Zn alloy with 10 wt.% Zn. The stacking fault energy (SFE) of this alloy is equal to 35 mJ/m\textsuperscript{2}. The rods of the alloy with a square cross section 8\times8 mm\textsuperscript{2} were cut into sections 60 mm long, annealed at 800 °C for 2 hours, slowly cooled to 500 °C and then quenched in water to obtain a homogeneous microstructure before pressing. Then the treated the so-called “initial rods” were processed by ECAP for 8 passes via route \textsuperscript{B}\textsubscript{c} (90° clockwise rotation around the axis of the billet between consecutive passes) at a temperature of 150 °C with a pressing rate of 37 mm/min using a die with the inner and outer channel intersection angles \(\phi = 90^\circ\) and \(\psi = 0^\circ\), correspondingly. The as-processed rods were flat rolled into thin plates with a reduction of thickness of 30%, 40%, 60%, 80%, 90% and 95% (thereinafter designated as samples 8 ECAP + 30% rolling, 8 ECAP + 40% rolling, 8 ECAP + 60% rolling, 8 ECAP + 80% rolling, 8 ECAP + 90%
rolling, 8 ECAP + 95% rolling) at a temperature of 150 °C. The rolling direction was parallel to the longitudinal axis of the rod. Besides, the initial rods of the alloy were rolled under the conditions described above (thereinafter designated as samples SS + 30% rolling, SS + 40% rolling, SS + 60% rolling, SS + 80% rolling, SS + 90% rolling, SS + 95% rolling).

Thin foils were cut from the longitudinal plane to prepare samples for transmission electron microscope (TEM) investigations. The final thinning of the foils was performed by the method of ink-jet electrolytic polishing in a special unit Tenupol-5 using the electrolyte of the following composition: 25% phosphoric acid, 25% ethanol and 50% deionized water with a current of 50-80 mA. Thin foils were examined by a JEM-2100 transmission electron microscope in dark and bright fields at an accelerating voltage of 160-200 kV. The surface density of the twins was analyzed by calculating the twin area, assuming that it was rectangular with respect to the investigated TEM image area. For statistical analysis, over 15 TEM images were analyzed.

X-ray diffractograms were obtained on a RigakuUltima IV diffractometer with the "Bragg-Brentano" scheme, using Cu Ka radiation generated at a voltage of 40 kV and a current of 40 mA. The lattice parameter, the X-ray crystallite size $d_{XRD}$, the dislocation density $\rho$, and the proportion $m_{\text{exp}}$ of edge dislocations among all dislocations were determined. At that, the diffractograms were analyzed within the framework of the Whole Powder Pattern Modeling algorithm implemented in the PM2K program.

The tensile specimens were cut by electric spark-cutting technique from the longitudinal plane and had the shape of a dog-bone with a total length of 12 mm and a gauge length of 4 mm. Tensile tests were carried out on an Instron 8801 tensile testing machine at room temperature with a ram speed of 0.24 mm/min. The values of tensile strength and yield stress were obtained from the engineering stress-strain curves.

3. Results and analysis

3.1. Microstructural evolution

Figure 1 shows TEM images of the initial coarse-grained (CG) samples with equiaxial grains with an average size of about 100 ± 40 µm after FR with 95% reduction and the sample after 8 passes of ECAP with subsequent FR to the same reduction.

Deformation twins are clearly observed in the fine structure after FR of the initial state with 95% reduction (figure 1 a). The surface density of twins is 1.2%. The average grain size, determined by the dark-field image is 170 ± 60 nm.

Grains are refined to a size of 210 ± 60 nm in the 8 ECAP + 95% rolling state (figure 1 b). Also, there are deformation twins in the structure. The surface density of twins is 0.5%.

![Figure 1](image1.png)

**Figure 1.** TEM images of the samples in SS + 95% rolling (a) and 8 ECAP + 95% rolling (b) states.
3.2. X-Ray results

Table 1 shows the estimated lattice parameter, the X-ray crystallite size \(d_{\text{XRD}}\), dislocation density and proportion of edge dislocation obtained by X-ray diffraction (XRD). An increase in the rolling reduction of the FR of the initial CG samples leads to a regular decrease in the lattice parameter.

| Microstructure parameter          | Lattice parameter, \(\text{nm}\) | \(d_{\text{XRD}}, \text{nm}\) | Dislocation density, \(10^{15} \text{m}^{-2}\) | Proportion of edge dislocations, % |
|-----------------------------------|-----------------------------------|---------------------------------|-----------------------------------------------|-----------------------------------|
| SS                                | 0.363789(11)                     | 452(79)                         | -                                             | 28                                |
| SS +30% rolling                   | 0.363786(18)                     | 390(54)                         | 0.62(18)                                      | 34                                |
| SS +40% rolling                   | 0.363782(8)                      | 324(61)                         | 0.89(13)                                      | 39                                |
| SS +60% rolling                   | 0.363777(12)                     | 226(44)                         | 1.14(26)                                      | 58                                |
| SS +80% rolling                   | 0.363778(10)                     | 170(57)                         | 2.18(17)                                      | 63                                |
| SS +90% rolling                   | 0.363771(19)                     | 104(23)                         | 2.44(9)                                       | 70                                |
| SS +95% rolling                   | 0.363769(12)                     | 76(19)                          | 2.51(12)                                      | 68                                |
| 8 ECAP                            | 0.363629(7)                      | 35(3)                           | 8.63(2)                                       | 89                                |
| 8 ECAP +30% rolling               | 0.363635(9)                      | 32(2)                           | 8.84(11)                                      | 95                                |
| 8 ECAP +40% rolling               | 0.363619(9)                      | 31(2)                           | 9.22(8)                                       | 90                                |
| 8 ECAP +60% rolling               | 0.363627(9)                      | 26(4)                           | 9.79(12)                                      | 96                                |
| 8 ECAP +80% rolling               | 0.363625(5)                      | 30(3)                           | 9.79(15)                                      | 91                                |
| 8 ECAP +90% rolling               | 0.363623(8)                      | 27(3)                           | 9.72(11)                                      | 95                                |
| 8 ECAP +95% rolling               | 0.363637(5)                      | 29(2)                           | 9.66(19)                                      | 89                                |

The lattice parameter of pure Cu is 0.361491 nm. Thus, it is observed that the lattice parameter tends to the lattice parameter of pure Cu.

The decrease in \(d_{\text{XRD}}\) indicates a grain refinement, which is in good agreement with the results of TEM investigations for the SS + 95% rolling state. The density of dislocations increases as the rolling reduction rises (table 1). According to Taylor's theory \([6]\), \(\sigma \approx \rho\), this process should lead to an increase in the strength characteristics, which is observed experimentally in figure 2. In the SS state the microstructure contains mainly screw dislocations, and after the rolling reduction of 95% the dislocations refer primarily to the edge ones (table 1).

FR of a sample subjected to 8 passes of ECAP does not lead to a regular change in the lattice parameter (table 1). Changing of the \(d_{\text{XRD}}\) parameter confirms the refinement of grains (table 1). The density of dislocations, which belong to the edge type, increases with the rise of the rolling reduction, which agrees with the growth of the strength characteristics of the alloy.

3.3. Tensile properties

Figure 2 shows the engineering stress-strain curves of the investigated states. The values of tensile strength calculated from the presented above engineering stress-strain curves for the initial samples after FR are equal to 410 MPa for reduction of 80%, to 421 MPa for the reduction of 90% and to 515 MPa for the reduction of 95%.

The yield stress of these samples is 380 MPa for the reduction of 80%, 400 MPa for the reduction of 90% and 496 MPa for the reduction of 95%. After 8 passes of ECAP and subsequent FR the values of tensile strength are equal to 682 MPa for the reduction of 80%, to 679 MPa for the reduction of 90% and to 724 MPa for the reduction of 95%, while the values of the yield stress are equal to 661 MPa for the reduction of 80%, to 657 MPa for the reduction of 90% and to 589 MPa for the reduction of 95%.

As can be seen from figure 2, 8 passes of ECAP +95% rolling allow achieving strength which is 1.4 times higher as compared to the SS samples after analogous FR, while the ductility also increases up to 14% from 9% for the corresponding states.
4. Discussion
FR of the Cu-10 wt.% Zn alloy to different reduction values promotes the change in strength and ductility. When rolling the initial CG samples, the strength increases logically, and the ductility varies little. The lattice parameter, crystallite size and dislocation density change monotonically as the rolling reduction increases. FR of the UFG alloy allows achieving high strength and sufficient ductility. The greatest effect is achieved for the state of 8 ECAP + 95% rolling (figure 2 curve 6). Analysis of the microstructure of 8 ECAP + 95% rolling (figure 1b) shows that a slight increased grain size (according to TEM investigations) in this case cannot lead to the observed increase in strength. However, as follows from table 1, while the crystallite size decreases as the rolling reduction increases, the refined subgrain structure increases the strength characteristics. An increase in the density of dislocations as the rolling reduction increases also leads to an increase in strength. According to [7], with SPD, the edge type dislocations are mainly located at the grain boundaries. This statement corresponds to the high fraction of high-angle grain boundaries detected in this report, which corresponds to the increased ductility of the UFG alloy [8]. In the ECAP + 95% rolling state, the highest strength is 724 MPa and sufficient ductility is 14% compared to the initial CG samples after rolling.

5. Conclusions
The formation of the UFG structure in the Cu-10 wt.% Zn alloy provides an increase in both strength and ductility during subsequent FR with an increasing rolling reduction. This behavior of the UFG state is significantly different from that characteristic of the case of rolling the CG state, characterized by an increase in strength and a decrease in ductility. The increased strength and ductility of the UFG state are explained by the developed substructure and the predominance of high-angle grain boundaries.

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Figure 2. Engineering stress-strain curves.
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