Microstructure and mechanical properties of nanostructured Cu-Zn alloys by ECAP and rolling

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Abstract. The results of investigation of the Cu–Zn system alloys with 10 wt.% and 30 wt.% Zn, the stacking fault energy (SFE) of which differs by a factor of 2.5, are presented. ECAP and subsequent flat rolling resulted in the refinement of the microstructure. Flat rolling with the reduction degree 95% resulted in the decrease of the concentration of nanotwins formed during ECAP. Flat rolling of ultra-fine grained (UFG) alloys leads to the strength increase and ductility decrease explained by the refinement of the microstructure and decrease in the concentration of deformation twins.

1. Introduction

Since the 90s of the last century, bulk nanostructured metallic materials obtained by the severe plastic deformation (SPD) methods and characterized by attractive properties cause great interest of researchers \cite{1}. Nanostructured billets obtained by SPD in the manufacture of industrial products must take the required dimensions and shape. The method of flat cold rolling is widely used to produce sheet blanks from coarse grained (CG) metal blanks on an industrial scale. At the same time, investigations of influence of flat rolling on the microstructure and properties of bulk nanostructured materials obtained by SPD are relevant.

Alloys of the Cu-Zn system belong to brass and have different SFE values. As a result, they are characterized by different microstructures, deformation mechanisms and level of properties \cite{2}. In reports \cite{3, 4}, the influence of SFE on the microstructural changes of materials subjected to SPD was investigated. In some reports, the effect of SPD methods such as equal channel angular pressing (ECAP) \cite{5} and high-pressure torsion (HPT) \cite{6–8} on the microstructure and properties of Cu–Zn alloys was investigated. In addition, the influence of flat rolling at room or cryotemperatures on the formation of UFG structure in these alloys was studied \cite{9–11}. However, the effect of using a combined deformation treatment scheme, including ECAP with subsequent flat rolling, remains little investigated.

In this paper the results of investigation of the alloys of Cu–Zn system with a content of 10 wt.% and 30 wt.% Zn are presented. The SFE of these alloys differs by a factor of 2.5. The alloys were subjected to ECAP and subsequent flat rolling in order to investigate their influence on the microstructure and mechanical properties.
2. Experimental

The materials used in this work belong to the Cu-Zn system and contain 10 and 30 wt.% Zn. The SFE of these alloys is equal approximately 35 mJ m\(^{-2}\) and 14 mJ m\(^{-2}\), correspondingly. The rods of 60 mm in length and the edge of the square cross of 8 mm were subjected to 2 ECAP passes using route B\(_c\) (90° clockwise rotation around the billet axis between the consecutive passes) at a temperature of 150 °C for Cu-10% Zn alloy and 300 °C for Cu-30% Zn alloy correspondingly. The pressing rate was 37 mm min\(^{-1}\). The inner and outer die channel intersection angles were selected as \(\varphi = 90^\circ\) and \(\psi = 0^\circ\) correspondingly.

After ECAP, the samples were rolled at the same temperatures into thin ribbons with different reduction degrees from 30% to 95%. The rolling direction was parallel to the longitudinal axis of the rods. Also, the initial rods of the alloys were rolled under the conditions described above.

Thin foils to prepare the samples for transmission electron microscope (TEM) investigations were cut from the planes of the ribbons. The thin foils were examined using a TEM JEM-2100 in dark and bright fields at an accelerating voltage of 200 kV. Final thinning of the foils was performed by the inkjet electrolytic polishing method in a Tenupol-5 special unit using the electrolyte of the following composition: 25% phosphoric acid, 25% ethanol and 50% deionized water with a current of 50-80 mA.

The tensile specimens were cut by electronic spark-cutting technique from the planes of the ribbons along the longitudinal axis and had dog-bone shape with 12 mm in gauge length and the initial working part of 4 mm. The tensile tests were carried out on an Instron 8801 tensile testing machine at room temperature with a strain rate of 0.24 mm/min. Microhardness by Vickers was determined on a Struers Duramin equipment, for which a load of 100 grams was applied for 10 seconds.

3. Results and analysis

3.1. Microstructure

The initial microstructure of the rods, obtained by homogenizing annealing, is characterized by equiaxed grains with an average size of 117 ± 5 µm for the Cu–10 wt.% Zn alloy and 93 ± 3 µm for the Cu–30 wt.% Zn alloy. Figure 1 a,b presents dark-field images of the microstructures of Cu-10 wt.% Zn and Cu-30 wt.% Zn alloys after two ECAP passes. The calculation the subgrains/grains sizes was produced by the standard secent method on dark-field images. After two ECAP passes, the size of structural elements for the Cu-10 wt.% Zn alloy was 800 ± 66 nm, for the Cu-30 wt. Zn alloy - 290 ± 55 nm.

The presence of deformation twins was observed in the structure after ECAP of both alloys (white arrows indicated in figure 1 a,b). Double diffraction points on the captured electron diffraction patterns for these states confirm the presence of twins in the microstructure (inserts in the lower right corner of figure 1 a,b). However, in the Cu-30 wt.% Zn alloy after two ECAP passes, twins of different orientations are observed, which is probably associated with different SFEs. In the Cu-10 wt.% Zn alloy, the average twin length is 1000 ± 100 nm and their thickness is 40 ± 4 nm. The calculated twin concentration (as the ratio of the twin area to the area of the observed photo) is \(\rho_{tw} = 3.3\%\). In the Cu-30 wt.% Zn alloy, the average length of twins is 290 ± 38 nm, the thickness is 20 ± 3 nm, and twin concentration \(\rho_{tw} = 5.3\%\).

Thus, plastic deformation leads to the nanoscale grain refinement and formation of deformation twins. It should be noted that in the alloy with low SFE value, the grain size is smaller, and twin concentration is higher. This can be explained by the fact that in the nanoscale range, the twinning occurs via the emission of partial dislocations by grain boundaries, which differs from the twinning mechanism in the CG state [12]. Low value of SFE makes easier for the dissociation of a dislocation into two partial dislocations, as a result a wider stacking fault ribbon is formed between them. It acts as a barrier for full dislocation, and the dislocation either cross slip or climb when it encounters a barrier. This promotes the formation of narrower twins [9].

Further flat rolling of the obtained UFG states for different reduction degrees leads to further changes in the microstructure. Figure 1 c,d shows the images of the investigated alloys subjected to 2 ECAP passes and subsequent flat rolling with a reduction of 95%.
In both alloys, in the body of some grains, there is a clear alignment of the dislocation walls in low-angle boundaries and the formation of a subgrain structure. The average size of the structural elements is 220 ± 30 nm and 130 ± 10 nm for alloys with a content of 10 wt.% and 30 wt.% Zn, respectively. As can be seen, subgrains and grains contain nanoscale deformation twins. In the Cu-10 wt.% Zn alloy, the average twin length is 150 ± 10 nm, the thickness is 4.0 ± 0.4 nm, and the twin concentration $\rho_{tw} = 0.8\%$. In the Cu–30 wt.% Zn alloy, the average twin length is 100 ± 8 nm, the thickness is 3.0 ± 0.3 nm, and the twin concentration $\rho_{tw} = 1.3\%$. Thus, further flat rolling leads to the microstructure refinement and formation of the nanotwins. The concentration of twins is reduced compared with ECAP, but remains higher in the alloy with a lower SFE value. It can be assumed that as reduction degrees increases, the dislocation density increases, a significant number of dislocations pile up at the twin boundaries, since they act as a barrier for dislocation slip. As a result of the interaction, the original atomically-flat coherent twin boundaries are bent and transformed into incoherent ones. Further straining transforms the incoherent boundaries into high-angle grain boundaries. This leads to the detwinning [13].

3.2. Tensile properties
The mechanical tensile test results, including Vickers microhardness measurements, are presented in table 1 for conditions after ECAP and ECAP with subsequent flat rolling with reduction 95%. As can be seen from table 1, the values of microhardness after rolling increase for two alloys compared to ECAP.

For an alloy with a lower SFE value, the ultimate strength is 1.2 times higher after flat rolling. This can be explained by the fact that at low SFE value, the dislocations cross slip is limited due to a wider stacking fault ribbon between partial dislocations. As a result, plastic deformation occurs with a large contribution of twinning, which is confirmed by the results of TEM investigations described above.

The ultimate elongation of the alloys decreases as a result of rolling. This is explained by the decrease in the twins concentration, as well as by the structure refinement.
Table 1. Mechanical properties of Cu-Zn alloys after ECAP and ECAP with subsequent flat rolling.

|          | ECAP-2p     | ECAP-2p+roll 95% |
|----------|-------------|------------------|
| Cu-10 wt.% Zn | 145±9       | 216±6            |
| σut, MPa  | 413±78      | 670±120          |
| ε, %      | 18±5        | 9±3              |
| Hv        | 222±7       | 250±6            |
| Cu-30 wt.% Zn | 50±79       | 812±130          |
| σut, MPa  | 12±3        | 8±2              |

In the Cu-10 wt.% Zn and Cu-30 wt.% Zn alloys, after flat rolling, the concentration of twins decreases by 4 times, the structural elements are refined by 2-4 times. At the same time, the ultimate elongation for the Cu-10 wt.% Zn alloy is halved to 9 ± 3%. For the Cu-30 wt.% Zn alloy, the elongation decreases to a lesser extent from 12 ± 3% to 8 ± 2%.

4. Conclusions
Flat rolling of UFG Cu-10 wt.% Zn and Cu-30 wt.% Zn alloys leads to the refinement of the microstructure and the formation of nanotwins, the concentration of which decreases as the reduction degree increases. However, the concentration of nanotwins remains higher in the alloy with a lower SFE value. In the future, such microstructural changes contribute to an increase in strength of the material and, however, they reduce its ductility.

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