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Study of Copper Microstructure after ECAP and Rolling

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In this article, the evolution of copper grade microstructure after equal-channel angular pressing (ECAP) and rolling is investigated. There is a relatively equiaxed ultrafine-grained structure formed after rolling after 10 passes of ECAP transformed into a lamellar structure with smaller grain-boundary spacing. After 10 passes of ECAP, the grain-boundary spacing is 180 nm, and it is reduced to 110 nm after rolling. Microstructure grinding during rolling almost does not occur. There is an increase in the share of large-angle boundary by 20% compared to the states after 10 passes of ECAP.

У цій статті досліджується еволюція мікроструктури мідних сортів після рівноканального кутового пресування (РККП) та вальцювання. Спостерігається відносно рівноосьова ультрадрібнозерниста структура, що утворюється після вальцювання, яка після 10 проходів РККП перетворюється на пластинчасту структуру з меншою віддаллю між межами зерен. Після 10 проходів РККП віддаль між межами зерен становить 180 нм, а після вальцювання вона зменшується до 110 нм. Подрібнення мікроструктури під час вальцювання майже не відбувається. Спостерігається збільшення частки великокутової межі на 20% порівняно зі станом після 10 проходів РККП.

Key words: microstructure, severe plastic deformation, pressing, rolling.

Ключові слова: мікроструктура, інтенсивна пластична деформація, пресування, вальцювання.

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1. INTRODUCTION

Nowadays, copper and copper alloys are widely used in industry due to their excellent physical and chemical properties. They make a great contribution to modern life: in magnetic circuits, power and communication cables, and other electrical devices. Recent achievements in the electronics industry have drawn the attention of many researchers to the development of copper-based alloys. Such alloys should have high strength and good electrical conductivity. However, high strength and good electrical conductivity are the two opposite conditions of all alloys.

Much works have been done in adding alloying elements (such as Cr, Ag, Zr, Nb and Co) to pure copper die. Such conventional hardening methods lead to various types of defects (*e.g.*, dislocations, hardening particles, point defects) in the copper die, which increase the scattering of conduction electrons and increase the electrical resistivity. Studies by K. X. Wei *et al.* [1] and C. Zhu *et al.* [2] have shown that the addition of Mg to copper-based alloys yields favourable results, and a reduction in production costs is achieved when Cu–Mg alloy contact wires are used.

Traditional metal forming techniques, including rolling and drawing, are commonly used to manufacture copper wires. High tensile strength (> 500 MPa) and good conductivity ($> 60\%$ IACS) are the most important properties required for wires [2]. Cold working improves the microstructure and increases the strength of alloys.

A high dislocation density is required to transform a coarse-grained material into an ultrafine-grained material, and a high stress shall be applied to increase the dislocation density [3–6]. At the same time, grain boundaries are reorganized to form an ultrafine-grained (UFG) or nanostructures. Conventional metal processing methods, such as extrusion or rolling, are limited in their ability to produce nanostructures for two important reasons. First, a limit to the amount of stress can be applied using these methods. Secondly, the stresses applied in conventional processes are insufficient to form nanostructures due to the low machinability of metal alloys under ambient conditions and low temperatures. As a result of these limitations, alternative machining methods based on the application of extreme deformation are needed. That means that very high stresses are applied at low temperatures without changing cross-sectional dimensions.

A new process of extreme plastic deformation, equal channel angular pressing (ECAP), can be used to obtain nanostructures with high strength and good electrical conductivity [7–11]. ECAP process has proven to be an efficient method for grinding metal grains and composites and has the advantage of being scalable for the production of large volume samples. Although ECAP is an effective processing tool for laboratory research, it is labour-intensive and not easy to use in industrial conditions [12, 13].

In copper, high structure grinding and growth of mechanical properties are achieved during ECAP. Similar changes are observed when other metals are treated with ECAP, as well as with other methods of severe plastic deformation (SPD) [14–18]. Further microstructure grinding is only possible when followed by another strain method. Therefore, combinations of ECAP and other metal forming techniques began to be used later on [19–22]. The most widespread is the combination of ECAP with subsequent rolling. It allows obtaining sheet semi-finished products with ground structure and increased strength properties, which can be used in industry.

From the presented literature data, it can be concluded that copper is significantly hardened after SPD due to the grinding of its microstructure, and additional rolling after ECAP will further grind the microstructure and prepare the work piece for further use.

Therefore, the purpose of this work is to study the peculiarities of nanostructures formation in copper using ECAP and rolling.

2. EXPERIMENTAL METHODS

M2 grade copper was used for the study and was supplied as a bar with a diameter of 25 mm and a length of 120 mm. M2 grade copper contained 99.7% Cu.

A tooling with a channel-crossing angle of 90° was used to perform the ECAP. The speed of movable die was 5 mm/sec. Pressing was carried out at room temperature using the Vs route for 10 cycles. The rolling direction after ECAP was perpendicular to the extrusion direction during pressing. At the same time, before rolling, the samples, which had a cylinder shape after pressing, were milled to obtain two parallel flat surfaces 15 mm apart. Rolling was carried out with compression rates per pass of 5–10%. The final thickness of samples after rolling was equal to 1.5 mm, *i.e.*, the total compression was of 90%, which corresponds to the degree of true deformation of 2.7.

Optical microscope, transmission electron microscope and EBSD analysis were used to analyze the microstructure of the samples. Samples for metallographic analysis were thinned on a Tenupol-5 device using the electrolyte composition: 250 ml of H_3PO_4, 250 ml of ethyl alcohol, and 500 ml of distilled water. EBSD measurements were performed on a JOEL field emission electron microscope (at 20 kV) using the Oxford software package.

3. RESULTS AND DISCUSSION

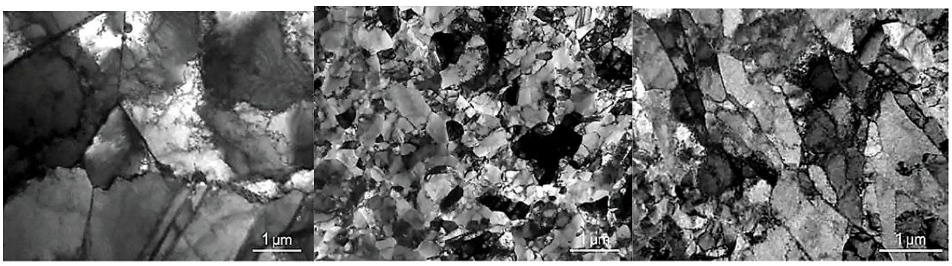
The microstructure of the initial bar consisted of large grains of 120 μm. Twins were observed in the body of many grains (Fig. 1, *a*). After 10 passes, clear thin boundaries appeared in the structure with presumably high-angle disorientation. The shape of grains has become more regular. The structure consisted of both fairly equiaxed grains and grains elongated in the shear direction. The grain size was of 350 nm (Fig. 1, *b*). A large number of twins were also observed in the structure.

EBSD analysis showed that the structure became much more equiaxed after 10 cycles. However, even after such a number of ECAP cycles, the structure remained extremely heterogeneous as small submicron grains were adjacent to areas consisting predominantly of subgrains. In general, no significant differences in microstructure in different planes were found, except for metallographic texture.

Metallographic analysis showed that the microstructure after rolling is significantly different from the obtained microstructure after ECAP (Fig. 1, *c*). A distinct fine-grained lamellar microstructure is observed. Lamellar boundaries parallel to the rolling plane are straight and clear. Many lamellae are internally divided into individual fragments by transverse boundaries, often thick and eroded. In the rolling plane the structure is characterized by sufficient equiaxiality, most grains are irregularly shaped; their boundaries are thick and indistinct.

EBSD confirms the data of metallographic analysis and shows that the majority of lamellar boundaries acquire high-angle disorientation with increasing number of cycles, and the structure becomes quite homogeneous. However, the transverse boundaries within the lamellae retain predominantly low-angle disorientation.

In our case, the Vs route carried out pressing, so, the shear direction in each subsequent pressing cycle was different. In such a



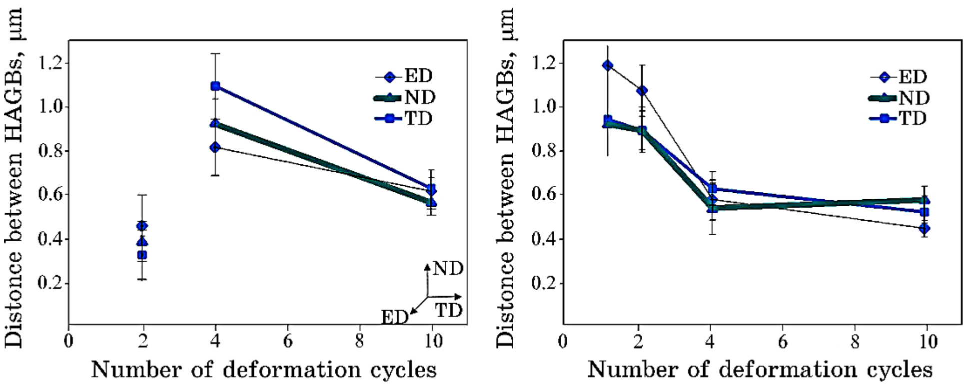
a *b* *c*

Fig. 1. Graphs of microhardness distribution along the bar section.

case, the newly formed shear bands are likely to intersect the bands formed during the previous cycle. Therefore, a rather equiaxed structure can occur at the intersection of shear zones that are perpendicular to each other (since the sample has been rotated at 90°C). The number of such intersections increases with each new pass and the volume fraction of equiaxed structures increases. In addition, the temperature of shear zone is probably much higher than the temperature of material volume. Therefore, the dynamic processes of return and recrystallization are more active and the fraction of large-angle boundary increases.

Figure 2, *a* shows the dependence of the large-angle boundary spacing (LABs) on the number of passes in different planes of the sample. Since after 1–2 passes the structure contains only a small number of grains whose size can be measured, the results of such measurements are very unreliable, so, they are not given at all. After 4 cycles, the average distance between large-angle boundaries was about 1 μm, whereas, after 10 cycles, it decreased to 700 nm. The distance between low-angle boundaries (SABs) as a function of the number of cycles varies similarly to the grain size, decreasing after the first two passes and then remaining almost constant at approximately 400 nm (Fig. 2, *b*).

In general, the obtained data on copper microstructure after ECAP coincide quite well with the known literature data. Thus, the transformation of elongated subgrains with predominantly low-angle boundaries formed after the first pass into a fairly equiaxed grain-subgrain structure with increasing number of passes was observed in the paper [9].



a *b*

Fig. 2. Distances between boundaries after ECAP according to EBSD analysis: *a*—distance between large-angle boundaries; *b*—distance between low-angle boundaries.

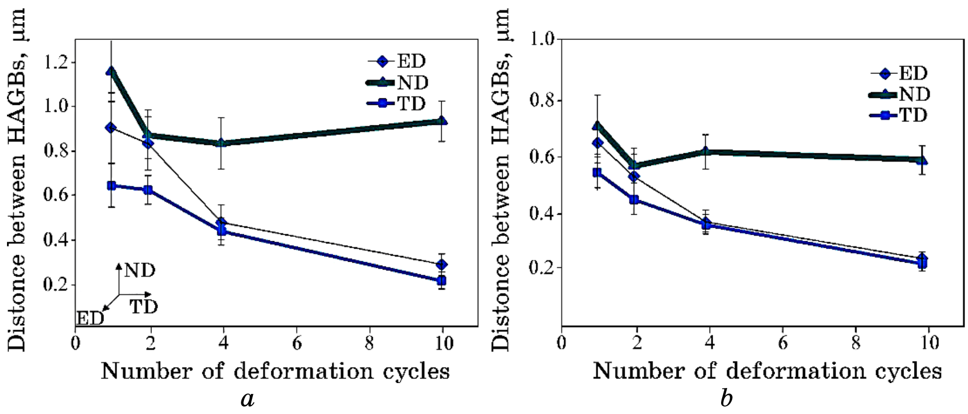


Fig. 3. Distances between boundaries after ECAP according and rolling to EBSD analysis: *a*—distance between large-angle boundaries; *b*—distance between low-angle boundaries.

The numerical values obtained from EBSD analysis after rolling show a significant difference between the microstructure in the rolling plane and in other planes. Thus, in the ED and TD planes, the distances between large-angle boundaries and low-angle boundaries decrease monotonically enough with increasing number of ECAP cycles, reaching values of 200–300 nm after 10 passes (Fig. 3). In the rolling plane, the distances between the boundaries almost do not change with the change in the number of passes, and are about 1000 nm for large-angle boundaries, and about 600 nm for low-angle boundaries. In the rolling plane, the portion of large-angle boundaries is basically unchanged after the first 4 cycles, amounting to 25–30%, and it increases to 45% after 10 cycles. In the ED and TD planes, in all cases except for 1 pass of ECAP, the portion of large-angle boundaries was almost the same and much higher than in the rolling plane. Large-angle boundaries portion increased by 20% after rolling compared to the values after ECAP.

4. CONCLUSION

The following conclusions can be drawn from the results obtained. Morphologically, the microstructure after ECAP does not differ from the typical microstructure formed in cubic metals during cold straining. The elongated microstructure becomes equiaxed after 10 straining cycles and the large-angle boundaries portion increases. Transverse grain size almost does not decrease with increasing number of passes. Rolling leads to a further reduction in the size of the structural components, at least in the RD and TD planes.

The conducted studies have shown that rolling after ECAP leads to extremely serious changes in microstructure. Instead of a relatively equiaxed structure, a lamellar structure is formed typical of cold-rolled b.c.c. metals.

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