Influence of crystallographic texture on the strength and electrical conductivity of ultrafine-grained copper

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Abstract: The paper covers the study of the influence of equal-channel angular pressing (ECAP) on the structure, crystallographic texture, mechanical properties and electrical conductivity of Cu-ETP copper (Russian analogue – M1), as well as the dependence of these characteristics on the orientation of the measurement direction relative to the cross-section (from -45 to 90°). The specific electrical conductivity and strength characteristics of the material in the as-delivered condition (hot-rolled) and the effect of annealing at a temperature of 450 °C of the original sample are investigated. Mechanical tests for uniaxial tension, a study of microhardness using the Vickers method and a study of specific electrical conductivity based on measuring the parameters of the vortex field excited in the surface layers of the body are carried out. It is found that ECAP processing leads to a significant increase in the ultimate tensile strength to 425 MPa compared to the initial state of 300 MPa. The maximum tensile strength of 425 MPa is achieved at orientation angles relative to the ECAP cross-section of -45° . A significant increase in microhardness to 1364-1405 MPa, tensile strength to 350-425 MPa and electrical conductivity to 101.4-102.4 % IACS is a consequence of the selected directions of cutting the samples relative to the ECAP axis. This indicates the dependence of both mechanical and electrical properties of ultrafine-grained samples on the crystallographic texture orientation. A Cu-ETP copper sample subjected to ECAP with a cutting angle deviating from the ECAP cross-section of the sample by 7.5° has the most optimal crystallographic orientation. In this case, the values of microhardness and electrical conductivity reached 1405 MPa and 102.4 % IACS, respectively.

Keywords: crystallographic texture; strength; electrical conductivity; ultrafine-grained copper; equal-channel angular pressing; structure.

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INTRODUCTION

Copper and low-alloyed copper alloys, due to their high electrical conductivity, are widely used in mechanical engineering for the manufacture of contacts and wires. Copper parts must have a unique combination of properties: high electrical conductivity, strength, ductility, and corrosion resistance. Good technological properties and relatively low cost determine the wide application of copper in industry both in the form of alloys and in pure form. In the work [1], it is shown that the mechanical strength and electrical conductivity of these materials are primarily controlled by their microstructure, the most important parameters of which are the grain size and dislocation structure. Dislocations and grain boundaries make a large contribution to increasing the yield strength, but a smaller contribution to increasing the specific electrical resistance [2].

In recent years, a promising research area has been the formation of an ultrafine-grained (UFG) structure with an average grain size of less than 1 μ m, which contributes to the manifestation of unique mechanical properties (high strength, increased fatigue limit) [3; 4]. At the same time, it is known that treatment by severe plastic deformation (SPD) is accompanied by active movement of dislocations and twinning, which leads to reorientation of grains and formation of developed crystallographic textures [5; 6]. Crystallographic texture usually occurs as a result of

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directed external mechanical action, in this case – the SPD process. The presence of a preferred orientation enhances the anisotropy of the material properties and can significantly change the performance characteristics of the product. Therefore, the possibility of texture formation should be taken into account when carrying out various deformation and heat treatments [7]. In particular, in samples of pure copper subjected to SPD, it was found that at the initial stages of deformation, a strong preferred orientation of crystallites occurs, which is characteristic of the simple shear texture [5]. At the same time, an increase in the degree of accumulated deformation contributes to the blurring of texture maxima, which is of interest for studying the influence of crystallographic texture on the strength and electrical properties of UFG copper.

The works are known that consider the influence of crystallographic texture on the strength and electrical conductivity of UFG copper produced by rotational forging [8] and electrodeposition [9], but the studies in them were carried out using the example of samples in the form of a wire or films with a different crystallographic texture.

The purpose of the study is to analyze the structure and crystallographic texture in ultrafine-grained samples of Cu-ETP copper produced by equal-channel angular pressing (ECAP) in order to identify structural factors leading to achieving higher strength while maintaining high electrical conductivity of the material.

METHODS

Materials and methods of research

A Cu-ETP (Russian analogue M1) copper commercial rod with a diameter of 20 mm, GOST 859-2001 (Table 1), was selected as the material for research.

To analyze the initial microstructure, two samples were studied, one of which was in the as-delivered condition – hot-rolled. The sample in the initial condition was annealed at 450 °C for 2 h. Before annealing, the initial sample was immersed in a melt of a mixture of KOH and NaOH salts to prevent oxidation of the material surface.

The formation of the UFG structure in a billet with a diameter of 20 mm and a length of 150 mm was carried out by the ECAP method in eight passes along the B_C route, which involves rotating the sample between two subsequent cycles counterclockwise for an angle of 90° around the longitudinal axis [6]. The passes of the billets were carried out on equipment with an angle of intersection of the channels of 120° at a temperature of 20 °C. Preparation of samples for metallographic analysis included cutting out samples (Fig. 1) on an ARTA-120 electrical discharge cutting-off machine taking into account the angles relative to the cross-section of ECAP of the billet (0°; 7.5°; $\pm 15^\circ$; $\pm 22.5^\circ$; $\pm 45^\circ$; 90°), grinding, polishing, and etching.

Grinding of samples was carried out on a NERIS grinding and polishing machine with a stepwise decrease in the grain size of the sanding paper from P100 to P4000 at a machine speed of 500–600 rpm.

Polishing was carried out on diamond paste with a gradual decrease in its grain size from 7/5 to 3/2. When moving to the next paste number, the paste residues were carefully removed from the metallographic section using alcohol, and the polishing direction was changed by 90° to ensure complete disappearance of the scratches from the previous paste.

To identify the microstructure, the sample was etched. The etchant composition: perchloric acid (HCl) -50 %, nitric acid (HNO₃) -25 %, acetic acid (CH₃COOH) -25 %. The etching mode was selected experimentally. The sample was etched for 2–3 s by dipping into the etchant, then washed with distilled water and dried with filter paper.

Structural studies

Microstructure images were obtained using a JEM-6390 scanning electron microscope and a JEM-2100 transmission electron microscope. Thin foils were prepared using a Tenupol-5 device by jet electrolytic polishing at 22–24 V using an electrolyte of the following composition: 920 ml of water (H₂O), 70 ml of orthophosphoric acid (H₃PO₄), 15 ml of glycerol (C₃H₅(OH)₃). The ECAP structure of the samples was studied in cross-section. The grain size was calculated using the GrainSize program based on the obtained structure images.

After ECAP processing, the Cu-ETP copper sample was cut with the following orientations (angles) relative to the ECAP cross-section of the billet: 0°; 7.5°; ±15°; ±22.5°; ±45°; 90°; thickness 1.5–2.5 mm, diameter 20 mm. The analysis of texture formation processes in copper was performed using a DRON-3m diffractometer equipped with an automatic texture attachment. Filtered Cu– $K_{\alpha 1}$ X-ray radiation (0.15406 nm) was used to shoot pole figures. Reflection imaging was carried out within the range of radial γ angle from 0 to 75° and azimuthal δ angle from 0 to 360°. The diameter of the irradiated area was 0.6 mm. In the case of ECAP, the study was carried out in the geometric centre of the longitudinal section of the billet.

Table 1. Chemical	composition of Cu-ETP grade copper
Таблица 1.	Химический состав меди марки М1

Content, %												
Cu	Fe	0	Pb	S	Zn	Ag	Sb	As	Ni	Sn	Bi	
99.9	≤0.005	≤0.05	≤0.005	≤0.004	≤0.004	≤0.003	≤0.002	≤0.002	≤0.002	≤0.002	≤0.001	



Fig. 1. Scheme of orientations of cutting ECAP samples **Рис. 1.** Схема ориентаций вырезки РКУП образцов

The result was a set of intensities of reflected X-rays. The experimental results calculated using the LaboTEX software package (<u>www.labosoft.com.pl</u>) are presented as complete pole figures in the shear plane.

Microhardness study

The measurements were carried out on a MicroMet 5101 device using the Vickers method in the cross-section of the ECAP samples under a load of 100 g, the indenter holding time was 10 s. The results were recorded along the sample diameter.

Electrical conductivity study

The specific electrical conductivity was determined at room temperature by the eddy current method using a VE-27NTs device with a relative measurement error of 2 %. Annealed pure copper with an electrical conductivity of 58 MS/m (electrical resistance of 0.017241 $\mu\Omega$ ·m) corresponds to the 100 % IACS designation according to the IACS (International Annealed Copper Standard) international standard. The results of electrical conductivity measurements in this paper are presented in % IACS, i. e. as a percentage of the electrical conductivity of pure copper.

Uniaxial tensile tests

The tests were carried out on a small sample deformation device at room temperature at a rate of $3 \times 10^{-3} \text{ s}^{-1}$.

In each state, two samples with working base dimensions of $6.0 \times 1.0 \times 0.7$ mm (Fig. 2) cut from the initial, annealed and ECAP billets were tested.

RESULTS

The obtained microstructure images are shown in Fig. 3. The initial structure of Cu-ETP copper consists of large irregular-shaped grains with an average size of 7 ± 4 µm (Fig. 3 a). The grain distribution histogram shows that most grains are located in the range of 2.5–10 µm (Fig. 3 b). The microhardness value in the initial state is 1211±65 MPa, the electrical conductivity value was 101.3±1.36 % IACS. Such electrical conductivity values were obtained due to the presence of a relative error of the measuring device, therefore, for the correct experiment and possible comparison of the results, the value obtained using the VE-27NTs device was taken.

After heat treatment at 450 °C, the grain size increased to $10.2\pm2.3 \mu m$ (Fig. 3 c). The grain distribution histogram shows that the majority of grains are located in the range of $5.5-15.5 \mu m$ (Fig. 3 d). The microhardness value is 773 ± 37 MPa, and the electrical conductivity is $102.2\pm1.79 \%$ IACS.

After ECAP processing, the grain size decreased to 300 nm on average (Fig. 3 e). The grain distribution histogram shows that the great number of grains are located in the range of 250–324 nm (Fig. 3 f).



Fig. 2. The geometry of a specimen for mechanical tensile testing Puc. 2. Геометрия образца для механических испытаний на растяжение



Fig. 3. Results of microscopy and grain size calculation:
a – structure of the initial sample; b – grain size (initial sample);
c – microstructure of the annealed sample; d – grain size (annealed sample);
e – dark-field TEM image of copper (ECAP); f – grain size (ECAP)
Puc. 3. Результаты микроскопии и расчета размеров зерна:
a – структура исходного образца; b – размер зерна (исходный образец);
c – микроструктура отожженного образца; d – размер зерна (отожженный образец);
e – темнопольное изображение ПЭМ меди (РКУП); f – размер зерна (РКУП)

As a result of texture analysis, direct pole figures were obtained (Fig. 4), which were rearranged for further analysis with a 90° rotation along the equatorial plane (Fig. 5). After eight ECAP passes at T=20 °C, on the (111) pole figure, clearly defined maxima are visible, the arrangement nature of which is quite ordered (Fig. 5). The crystallographic texture after eight ECAP passes can be described by ideal orientations (Fig. 6) corresponding to the state after simple shear taking into account the rotation by an angle of 60° counterclockwise. The (111) pole figure is characterized by a set of seven maxima (Fig. 5 d): six maxima are symmetrically located on the pole figure periphery and one in its centre. These maxima correspond to the A {111}<uvw>, B {hkl}<110>, and C {001}<110> components of the simple shear texture. Their intensity increases with increasing shear angle (from 0 to 22°).

The crystallographic textures of all sections of the copper sample subjected to eight ECAP passes are identical and are characterized by dominant {110}<111> components (Fig. 5). At the same time, the pole figures obtained for different sections are characterized by the fact that the A_{10}^* , A_{20}^* and C_0 maxima located on the periphery of the pole figure shift toward its centre with an increase in the cutting angle. In general, the finally formed texture can be described by the main textural (111)[$\overline{1}$ $\overline{1}$ 2], (111)[$11\overline{2}$], ($1\overline{1}$ 1)[110], ($\overline{1}$ 1 $\overline{1}$)[$\overline{1}$ $\overline{1}$ 0], ($1\overline{1}$ 2)[110], ($\overline{1}$ 12)[$\overline{1}$ $\overline{1}$ 0], and (001)[110] maxima characteristic of simple shear textures.

The indicated A_{θ} , $A_{1\theta}^*$, $A_{2\theta}^*$, \overline{A}_{θ} , B_{θ} , \overline{B}_{θ} , and C_{θ} ideal orientations lie on the {111} θ and <110> θ fibres (Fig. 6). For the (111) pole figure obtained for a 7.5° cut, a superposition of the recrystallization texture on the simple shear texture is observed (Figs. 5 b and 6 b). The recrystallization process is associated with the absorption of old grains by new equiaxed grains with high-angle boundaries. It is activated when a certain deformation degree is reached. In the case under consideration, in addition to the simple shear texture component on the (111) pole figure, the formation of dominant $R1(\overline{1}11)[113]$, $R2(120)[\overline{2}11]$, $R3(023)[3\overline{3}2]$, and $R4(\overline{1}22)[\overline{2}\overline{2}1]$ components characteristic of the recrystallization texture is observed (Fig. 6 b).

In sections of 15 and 22.5°, a broken symmetry of the crystalline structure is observed. 0° section corresponds to the crystalline texture of simple shear of the metal fcc lattice. In section of 7.5° , the location of texture maxima corresponds to an absolutely symmetrical pattern of the crystal structure, which indicates its greatest ordering and explains the highest electrical conductivity (Fig. 4).

The mechanical test curves were obtained for the initial, annealed initial (Fig. 7 a) and ECAP-treated samples (Fig. 7 b). After annealing the initial samples, a decrease in the ultimate tensile strength from 300 to 210 MPa occurred due to an increase in the average grain size, as well as an increase in plasticity due to an increase in the stage of strain hardening (Fig. 7 a). In the ECAP samples, different values of the ultimate tensile strength were observed in the range from 330 to 425 MPa depending on the angle of cutting relative to the ECAP axis (Fig. 7 b). At the same time, all the studied samples showed close values of relative

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elongation before failure of about 5 %. Figs. 8, 9 show the changes in microhardness, ultimate tensile strength, and electrical conductivity depending on the direction of sample cutting relative to the ECAP axis, which indicates a strong influence of the crystallographic texture on these characteristics of the samples. The maximum (425 MPa) and minimum (330 MPa) ultimate tensile strengths were observed at orientation angles relative to the ECAP cross section of -45° and 15° , respectively. The highest values of microhardness and electrical conductivity were observed at $7.5^{\circ} -$ 1405 MPa and 102.4 % IACS, respectively.

DISCUSSION

An increase in the strength properties of Cu-ETP copper after grain structure refinement has already been observed in the literature using the example of UFG samples produced by SPD [10; 11]. It can be explained by the wellknown Hall–Petch relationship [12; 13], which describes the dependence of the yield strength on the average grain size. In [14], it was noted that in order to achieve a combination of high strength and good electrical conductivity in copper materials, grain refinement to an average size of 200 nm is sufficient. In the present work, a close average grain size of 300 nm was observed in the ECAP samples, as well as a high ultimate strength of 425 MPa close to the values of 450 MPa previously observed in the pure copper ECAP samples [15].

A combination of increased strength and electrical conductivity values was also noted using the example of UFG copper samples produced by electrodeposition [17], multiple rolling [18] and drawing [19], which were characterized by the presence of a crystallographic texture. The features of crystallographic texture in ECAP copper samples were investigated in [20; 21]. In the work [20], it was noted that there is a texture gradient in different directions of equalchannel angular pressing of samples, which can create anisotropy of mechanical properties in them. The work [21] demonstrated that the electrical conductivity of ECAP copper is affected by various structural factors, including grain orientation and crystallographic texture. In this paper, it is shown that the difference in strength and electrical conductivity of ECAP copper samples relative to different crystallographic directions can reach 20–30 and 2–3 %, respectively.

CONCLUSIONS

1. The structure of Cu-ETP copper in the initial state is represented by grains of irregular shape with an average size of 7 μ m, subsequent annealing at 450 °C leads to an increase in the grain size to 10.2 μ m. After ECAP processing, the grain size decreased to 300 nm. ECAP processing led to a significant increase in the tensile strength compared to the initial state (300 MPa). The maximum (425 MPa) and minimum (350 MPa) tensile strengths were observed at orientation angles relative to the ECAP crosssection of -45° and 15°, respectively.

2. A significant dependence of the change in microhardness (1364–1405 MPa), tensile strength (350–425 MPa) and electrical conductivity (101.4–102.4 % IACS) on the directions of cutting of the samples relative to the ECAP



Fig. 4. Direct pole figures of copper after eight ECAP passes in different sections: $a - 0^\circ$; $b - 7.5^\circ$; $c - 15^\circ$; $d - 22.5^\circ$ Рис. 4. Прямые полюсные фигуры меди после 8 проходов РКУП в различных сечениях: $a - 0^\circ$; $b - 7,5^\circ$; $c - 15^\circ$; $d - 22,5^\circ$



Fig. 5. Rearranged direct pole figures in different studied states: $a - 0^\circ; b - 7.5^\circ; c - 15^\circ; d - 22.5^\circ$ Рис. 5. Перестроенные прямые полюсные фигуры в различных исследованных состояниях: $a - 0^\circ; b - 7.5^\circ; c - 15^\circ; d - 22.5^\circ$



Fig. 6. Positions of ideal orientations corresponding to the state after a simple shear taking into account a rotation by an angle of 60° counterclockwise: a – cubic orientation; b – R-orientation Puc. 6. Положения идеальных ориентировок, соответствующих состоянию после простого сдвига

ис. о. положения идеальных ориентировок, соответствующих состоянию после простого совига с учетом поворота на угол 60° против часовой стрелки:

а – кубическая ориентировка; **b** – R-ориентировка

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Fig. 8. Orientation angle dependence curves microhardness and electrical conductivity Puc. 8. Кривые зависимости от угла ориентации микротвердости и электропроводности



Fig. 9. Orientation angle dependence curves strength and elongation **Рис.** 9. Кривые зависимости от угла ориентации прочности и удлинения

axis indicates a strong influence of the crystallographic texture both on the mechanical and on the electrical properties of the UFG samples. The most favourable crystallographic orientation is exhibited by the Cu-ETP copper sample with a cutting angle deviating from the crosssection of the ECAP sample by 7.5°. In this case, the values of microhardness and electrical conductivity reached 1405 MPa and 102.4 % IACS, respectively.

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Влияние кристаллографической текстуры на прочность и электропроводность ультрамелкозернистой меди

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Аннотация: Работа посвящена исследованию влияния равноканального углового прессования (РКУП) на структуру, кристаллографическую текстуру, механические свойства и электропроводность меди марки М1, а также зависимости этих характеристик от ориентации направления измерения относительно поперечного сечения (от -45 до 90°). Исследованы удельная электропроводность и прочностные характеристики материала в состоянии поставки (горячекатаного) и влияние отжига при температуре 450 °С исходного образца. Проведены механические испытания на одноосное растяжение, исследование микротвердости по методу Виккерса и исследование удельной электропроводности, основанное на измерении параметров вихревого поля, возбуждаемого в поверхностных слоях тела. Установлено, что обработка РКУП приводит к значительному увеличению предела прочности до 425 МПа по сравнению с исходным состоянием 300 МПа. Максимальный предел прочности 425 МПа достигается при углах ориентаций относительно поперечного сечения РКУП -45°. Существенный разброс в повышении микротвердости до значений 1364–1405 МПа, предела прочности до 350–425 МПа и электропроводности до 101.4–102.4 % IACS является следствием выбранных направлений вырезки образцов относительно оси РКУП. Это свидетельствует о зависимости не только механических, но и электрических свойств ультрамелкозернистых образцов от ориентации кристаллографической текстуры. Наиболее оптимальной кристаллографической ориентировкой обладает образец меди марки М1, подвергнутый РКУП с углом реза, отступающим от поперечного сечения РКУП образца на 7,5°. В данном случае значения микротвердости и электропроводности достигали 1405 МПа и 102,4 % IACS соответственно.

Ключевые слова: кристаллографическая текстура; прочность; электропроводность; ультрамелкозернистая медь; равноканальное угловое прессование; структура.

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