Evolution of particles of secondary phases in alloys of the Cu-Cr-Zr system in the state of a supersaturated solid solution in the process of deformation-heat treatment

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Abstract. In this work, the evolution of particles of secondary phases in the process of equal-channel angular pressing and subsequent heat treatment of the Cu-1.0 wt.% Cr-0.7 wt.% Zr-0.2 wt.% Al alloy, preliminarily subjected to quenching from the pre-melting temperature, is studied. It is shown that in the process of deformation of the samples, partial dissolution of small particles of the secondary phases occurs. As a result of the subsequent aging, the Cr and Al atoms that have passed into the solution are again separated into small particles. Additionally, there was a partial separation of Cr atoms from the solid solution into small particles of chromium.

1. Introduction
Copper alloys are widely used for the manufacture of electrical conductors, which requires them to have high electrical conductivity. It is known that particles of secondary phases in the alloys affect the electrical conductivity and strength of conductors [1]. In this regard, studies of their evolution in the process of deformation-heat treatment of alloys are relevant [2, 3].

The aim of this work was to analyze the evolution of particles of secondary phases in the process of equal-channel angular pressing (ECAP) and subsequent aging of the Cu-1.0 wt.% Cr-0.7 wt.% Zr-0.2 wt.% Al alloy preliminarily subjected to high-temperature heat treatment with subsequent quenching basing on the known experimental data on the character of the change in its specific electrical conductivity [3].

2. Experimental and theoretical approaches
A detailed description of the experiments, the results of which were used in analytical modeling, are presented in [3]. Samples of the Cu-1.0 wt.% Cr-0.7 wt.% Zr-0.2 wt.% Al alloy in the initial states (IS) with an average grain size equal to $d=90\pm4\ \mu$m were obtained by annealing at a temperature of 1050°C for 5 h with subsequent quenching in water to form a supersaturated solid solution (SS). Samples of coarse-grained (CG) alloy obtained in this way were deformed using 8 passes of ECAP on equipment with a channel intersection angle of 90° via route Bc at room temperature. As a result, an ultrafine-grained (UFG) structure (8ECAP state) was formed in the alloy. Samples of the UFG alloy were subjected to subsequent aging at a temperature of 450 °C for 0.5 h, which led to the formation of the 8ECAP+A state.
The microstructure was studied by optical metallography, scanning and transmission electron microscopy. The conductivity measurements were performed using an eddy current device at room temperature.

During the preliminary heat treatment of the alloy, an increase in the size of particles that did not dissolve at a temperature of 1050 °C was observed. According to [3], these particles are most likely intermetallic compounds, which were formed during casting of the alloy. The phase analysis of the microstructure of samples of a Cu-Cr-Zr alloy containing Al atoms subjected to thermomechanical treatment showed that they could contain dispersed particles of secondary phases of different sizes and compositions, such as Al₃Zr₅, AlCrZr, ZrAl₂, ZrAl₃ and Zr₂Al₃ [2], the size and density of which change depending on the external influence. In the IS, against the background of the obtained α-solid solution, large inclusions with the size $D=4.0\pm0.4 \, \mu m$ were observed. The distance between them $L$ was established based on the available images of the microstructure and turned out to be equal to 8.3 μm. Small particles were also detected. Their size distribution is shown in figure 1a. The average distance between them $L$ amounted to 300.0±7.0 nm.

After 8 ECAP passes, the grain size decreased to 200.0±5.0 nm. The average distance between small particles increased to 340.0±8.0 nm. The fraction of small particles with a size of ≈10 nm has increased (figure 1b) [3].

![Figure 1. Distribution of particle sizes in the initial CG state, $q_j$ – the fraction of particles of radius $r_j$ (a); distribution of particle size in the 8ECAP state, $q'_j$ – the fraction of particles of radius $r'_j$ (b).](image)

Subsequent aging resulted in the decomposition of the SS. The most probable size of small particles lays in the range of 15–20 nm; the distance between the particles decreased to 155.0±3.5 nm [3].

The specific electrical resistance of the studied alloy in its various structural states is due to the scattering of electrons on various defects of the crystal lattice, alloying atoms, thermal vibrations of the lattice and grain boundaries [4 - 8].

Alloying atoms $a$ dissolved in the matrix contributes to the resistivity of the alloy, which is directly proportional to their atomic fraction $C_a$ in solution [5]:

$$\rho_s = \frac{k^3h^4n_en_a}{9e^2\pi n_s} \left[ 1 - \frac{\sin 2kl}{2kl} \right],$$

where $n_a$ is the volume concentration of the matrix atoms; $k=2\pi/\lambda$ - wave number, $\lambda=h/m^*u_F$ - electron wavelength, $m^*$ - effective electron mass, $u_F$ - Fermi velocity; $h$ is Planck's constant; $e$ - electron
charge value; \( n_e \) - density of free electrons; \( l \) - size of the region of interaction of an electron with an alien atom, \( l \approx 1.35 b_H \), \( b_H = a \sqrt{2} / 2 \), \( a \) - lattice parameter.

The contribution of lattice dislocations \( \rho_{\text{disl}} \) is directly proportional to their density \( q_c \) [6, 5]:

\[
\rho_{\text{disl}} = \frac{h k^2 q_c}{9 n_e e^2} \left[ 1 - \sin \frac{2 kl}{2kl} \right], \quad l \approx 1.55 b_H.
\]  

To determine the value of \( q_c \), the dislocation density in grain boundaries (GBs) was preliminarily calculated. It was assumed that GBs are formed by one set of edge dislocations spaced apart from each other at a distance \( H = b / 2 \sin (\Theta / 2) \). Then the dislocation density in them will be equal to \( q_u = 1 / H d \).

The average density of dislocations in high-angle GBs of the alloy takes the value

\[
q_{wH} = \frac{8}{(\Theta - 2 \Theta')} \int_{\Theta'}^{\Theta} \frac{1}{d} \sin \frac{1}{d} \sin \Theta d \sin \Theta = \frac{1.054 \cdot 10^9}{d}, \quad \text{m}^{-2},
\]  

where \( \Theta = \pi / 2 \), \( \Theta' = 16^\circ \); the value of the Burgers vector in the case of high misorientation angles \( \Theta \) is equal to \( b = 2 h c \sin (\Theta / 2) \), \( h_c = 7 b_H \) [7]. Then the density of lattice dislocations \( q_c \), taking into account their average value \( q \) and the volume fraction of GBs \( f = 0.03 \) [8], takes the value

\[
q_c = (q_{wH} q_u) / (1 - f).
\]  

Knowing the resistivity of linear defects \( \rho_{x} \), which make up GBs, per unit of their density \( q_u \) and the distance between them \( H, \) it is possible to calculate the specific electrical resistance of GBs per unit of their area \( S \) in a unit volume \( \Omega \):

\[
\rho_{\text{gb}} / N_{gb} = \rho_x / q_u H, \quad N_{gb} = S / \Omega \approx 1 / d.
\]  

High-angle GBs of CG samples increase the resistivity by \( \rho_{gb} \approx 2.67 \cdot 10^{-16} N_{gb} \Omega \cdot \text{m} \) [8]. The contribution of high-angle GBs of UFG alloy samples was determined on the basis of experimental data [9, 8] and amounted to \( \rho_{gb} / N_{gb} \approx 5.60 \cdot 10^{-16} \Omega \cdot \text{m}^2 \).

An electron moving in the lattice with an average velocity \( \nu \) collides with the particles of the secondary phases, approaching the center of the particle at a distance of its radius \( r \). Consequently, the mean free path of an electron is equal to \( \lambda = 1 / n_{ps} \pi r^2 \), where \( n_{ps} \) is the number of particles per unit volume. According to Ohm's law, electrical resistivity is

\[
\rho = \frac{m_e \nu}{n_e e^2 \lambda},
\]  

where \( m_e, n_e \) are the mass and density of electrons, respectively. Taking into account that electrons obey the Fermi-Dirac statistics, we will take the average velocity equal to the Fermi velocity: \( \nu = \nu_F \). In this case, the electron scattering cross section is associated with the geometric cross section of the particle, since its dimensions are much larger than the atomic dimensions. Small and large particles of secondary phases will increase the resistivity of the alloy by \( \rho_{\text{secl}} \) and \( \rho_{\text{secl}} \) [4], respectively:

\[
\rho_{\text{secl}} = \frac{m_e \nu_F}{n_e e^2} \sum f_{\text{secl}} \frac{\pi R^2 (j)}{V_{\text{pl}}}, \quad \rho_{\text{secl}} = \frac{m_e \nu_F}{n_e e^2} f_{\text{secl}} \frac{\pi R^2}{V_{\text{pl}}},
\]

where \( f_{\text{secl}}, V_{\text{pl}} \) - volume fraction and volume of small particles of radius \( r(j) \), respectively; \( f_{\text{secl}}, V_{\text{pl}} \) is the volume fraction and volume of large particles; \( R \) is the size of the region of interaction of an electron with a large particle, which is equal to its radius \( D / 2 \).

It was assumed that the processes of electron scattering by various defects of the crystal lattice are independent. Therefore, the resistivity of the alloy was determined according to the Matthiessen rule as the sum of their contributions.

3. Results and discussion
Electrical resistivity is a structurally sensitive quantity that can be easily measured. Therefore, it has been used to detect the character of changes in the particles of secondary phases as a result of ECAP and subsequent thermal effect on the alloy.
In the cast state, the structure of the alloys of the Cu-Cr-Zr system consists of dendrites of a solid solution based on copper with phase particles distributed along the boundaries of the dendrites in accordance with the phase diagram. In alloys containing Cr, chromium-based particles with an average size of ≈1.5 μm, which are identified as Cr particles, precipitate from the SS. Alloys containing Zr atoms have large (≈3.5 μm) particles of the intermetallic compound CuZr. Subsequent hot forging and quenching lead to the formation of a more uniform structure and to the partial dissolution of the second phases [10]. The solubility of Cr in Cu at 1050 °C is 0.6 wt.% (0.73 at.%), the solubility of Zr in Cu is 0.15 wt.% (0.10 at.%). The solubility of Al in Cu at 1050 °C is 6.9 wt% (14.8 at.%). It decreases with increasing temperature [1].

Since the content of alloying elements exceeds their solubility in Cu at the quenching temperature, particles of undissolved phases appear in the structure of the alloys. It was assumed that, as a result of small particles of different phases, as well as the probabilities of detecting large particles of different number of small and large particles, respectively, Cr and Zr, respectively. From expression (7) it follows that the number of large particles in the sample is

\[ N_{pl} = \frac{g}{p} \frac{W_{secCr}}{N_{ps} p_{Crs} \mu_{Cu}} \alpha \left( \sum j q_j \gamma_j^3 N_{ps} N_{pCrs}(j) + g \gamma_j^3 N_{ps} p_{Crs} \right) \]

where  \( g \) and  \( p \) are the number of phases of small and large particles, respectively,  \( g=5 \) (table 1),  \( p=2 \).  \( N_{ps}(j) \) – the number of Cr atoms in one small particle of the secondary phase  \( i \) and radius  \( r_i \) (table 1);  \( N_{pCrs} \) – number of Cr atoms in one large particle of the secondary phase,  \( N_{Crs} = \frac{4}{p} \left( \frac{D}{2} \right)^3 \Omega_{Cr} \). The volumes of Al, Cr, and Zr atoms are equal, respectively:  \( \Omega_{Al} = \frac{\mu_{Al} \mu_{pAl}}{\rho_{Al}} \approx 16.60 \times 10^{-30} \) m\(^3\),  \( \Omega_{Cr} = \frac{\mu_{Cr} \mu_{pCr}}{\rho_{Cr}} \approx 12.00 \times 10^{-30} \) m\(^3\),  \( \Omega_{Zr} = \frac{\mu_{Zr} \mu_{pZr}}{\rho_{Zr}} \approx 23.28 \times 10^{-30} \) m\(^3\), where  \( \rho_{Al} \) – Al density,  \( \rho_{Cr} \) – Cr density,  \( \rho_{Zr} \) – Zr density,  \( m_0 \) – atomic mass unit;  \( \mu_{Al} \),  \( \mu_{Cr} \),  \( \mu_{Zr} \) – relative atomic masses of Al, Cr and Zr, respectively. From expression (7) it follows that the number of large particles in the sample is

\[ N_{ps} = \frac{g p W_{secCr} \alpha N_{Crs} \mu_{Cu}}{\mu_{Cr} \left( \sum j q_j \gamma_j^3 N_{ps} N_{pCrs}(j) + g \gamma_j^3 N_{ps} p_{Crs} \right)} \]

Table 1. The number of atoms of aluminum  \( N_{pAl} \), chromium  \( N_{pCrs} \) and zirconium  \( N_{pZrs} \) in one small particle of the secondary phase  \( i \).

|   | ZrAl\(_3\) | Zr\(_2\)Al\(_3\) | ZrAl\(_2\) | AlCrZr | Al\(_2\)Zr\(_5\) |
|---|---|---|---|---|---|
| \( N_{pZrs} \) | \( V_{pZr} \) | \( 2V_{pZr} \) | \( V_{pZr} \) | \( 5V_{pZr} \) | \( - \) |
| \( N_{pAl} \) | \( 3\Omega_{Zr} + 3\Omega_{Al} \) | \( 2\Omega_{Zr} + 3\Omega_{Al} \) | \( \Omega_{Zr} + 2\Omega_{Al} \) | \( \Omega_{Al} + \Omega_{Cr} + \Omega_{Zr} \) | \( 5\Omega_{Zr} + 3\Omega_{Al} \) |
| \( N_{pCrs} \) | \( - \) | \( - \) | \( - \) | \( V_{pCrs} \) | \( - \) |
| \( i \) | 1 | 2 | 3 | 4 | 5 |
The volume fraction of small particles of radius \( r_q \), the fraction of which in the total number of small particles is equal to \( q_i \) (figure 1a), was calculated based on its definition:

\[
f_{scq} = \frac{V_{ps}N_{ps}}{V_s} = \frac{V_{ps}q_i \gamma^3 N_{pl}}{V_s} = \frac{gpV_{ps}q_i \gamma^3 W_{secC} \alpha \mu_{Cu}}{(nCr,\Omega_{Cu} + nZr,\Omega_{Zr} + nAl,\Omega_{Al} + \Omega_{Cu}) \mu_{Cu}(p \sum q_i \gamma^3 N_{pcCu}(j) + gN_{pCu})},
\]

where \( N_{ps} \) - number of small particles of radius \( r_q \); \( V_s \) - volume of sample, which was considered equal to the sum of the volumes of its constituent atoms; \( \Omega_{Cu} \) - volume of Cu atom, \( \Omega_{Cu}=\mu_{Cu}m_0/\rho_{densCu}=11.82\cdot10^{-30}, \rho_{densCu} \) - Cu density. According to the calculations \( f_{sec} = \sum_{j} f_{scq} = 5.87\cdot10^{-4} \). The volume fraction of large particles was determined by the expression

\[
f_{scL} = \frac{V_{pl}N_{pl}}{V_s} = \frac{gpV_{pl}W_{secC} \alpha \mu_{Cu}}{(nCr,\Omega_{Cu} + nZr,\Omega_{Zr} + nAl,\Omega_{Al} + \Omega_{Cu}) \mu_{Cu}(p \sum q_i \gamma^3 N_{pcCu}(j) + gN_{pCu})},
\]

where \( n_{ei}=W_{muCu}/\mu_i(1-W_{Zr}-W_{Cr}-W_{Al}), k=Cr, Zr, Al; W_{Cr}, W_{Zr}, W_{Al} \) - mass fractions of Zr, Cr, Al atoms in the alloy. According to the calculations \( f_{scL}=1.42\cdot10^{-2} \).

The electrical resistivity of the alloy in the IS is due to thermal vibrations of atoms in the nodes of the crystal lattice and defects in its structure. When calculating the electrical resistivity \( \rho_T \) due to thermal vibrations of the crystal lattice, the contribution of the regions occupied by large Cr particles was taken into account: \( \rho_T=(1-f_{scL}) \rho_{TCu}+f_{scL} \rho_{TCr} \), where \( \rho_{TCu}=1.72\cdot10^{-8} \) \( \Omega \cdot m \) is the electrical resistivity of pure CG copper; \( \rho_{Cr}=2.70\cdot10^{-8} \) \( \Omega \cdot m \) is the electrical resistivity of chromium at RT. It was assumed that the resistivity of the particles of the CuZr compound has a value close to that for the matrix, which somewhat underestimates the value of \( \rho_T \). The contribution of the regions occupied by fine particles was neglected. The density of lattice dislocations (4), taking into account the density of dislocations in GBs (3) \( q_{WW}=1.17\cdot10^{13} \text{ m}^{-2} \) and the average density of dislocations equal to \( q=3.00\cdot10^{12} \text{ m}^{-2} \) [11], takes the value \( q_{Wi}=2.70\cdot10^{12} \text{ m}^{-2} \). The contribution of factors that increase the electrical resistivity of the alloy in the IS is shown in table 2.

| Table 2. Specific electrical resistivity \( \rho \), calculated \( \delta \) and experimental \( \delta_{exp} \) values of specific electrical conductivity, contribution of thermal vibrations of atoms of crystal lattice and defects in the crystal structure of Cu to the specific electrical resistivity of the Cu-1.0 wt.% Cr-0.7 wt.% Zr-0.2 wt.% Al alloy in its various structural states. |
|------------------|------|------|------|
| \( \rho_{sec} \) (\( \Omega \cdot m \)) | Initial | 8ECAP | 8ECAP+A |
| \( \rho_{sec} \) (\( \Omega \cdot m \)) | 3.86\cdot10^{-11} | 8.07\cdot10^{-12} | 5.78\cdot10^{-10} |
| \( \rho_{scL} \) (\( \Omega \cdot m \)) | 1.76\cdot10^{-12} | 1.76\cdot10^{-12} | 1.76\cdot10^{-12} |
| \( \rho_L \) (\( \Omega \cdot m \)) | 1.73\cdot10^{-8} | 1.73\cdot10^{-8} | 1.74\cdot10^{-8} |
| \( \rho_{Cr} \) (\( \Omega \cdot m \)) | 2.51\cdot10^{-8} | 2.62\cdot10^{-8} | 4.81\cdot10^{-9} |
| \( \rho_{Al} \) (\( \Omega \cdot m \)) | - | 0.80\cdot10^{-8} | - |
| \( \rho_{dis} \) (\( \Omega \cdot m \)) | 4.66\cdot10^{-13} | 1.48\cdot10^{-10} | 2.51\cdot10^{-11} |
| \( \rho_{gb} \) (\( \Omega \cdot m \)) | 2.97\cdot10^{-12} | 0.28\cdot10^{-8} | 0.24\cdot10^{-8} |
| \( \rho \) (\( \Omega \cdot m \)) | 4.25\cdot10^{-8} | 5.45\cdot10^{-8} | 2.52\cdot10^{-8} |
| \( \delta \) (% IACS) | 40.6 | 31.6 | 68.4 |
| \( \delta_{exp} \) (% IACS) | 42±1 | 32±1 | 70±1 |
After 8 passes of ECAP, the distance between small particles increased and the fraction of particles with a size of \(>10\) nm increased [3]. Consequently, their fragmentation took place. At the same time, atoms of alloying elements were released into solution from small particles of secondary phases as a result of their partial dissolution. The deformation by the ECAP method created favorable conditions for this. If we assume that the distance between micrometric particles has not changed significantly, then the number of small particles will be \(\gamma' = L_v / L_s\) times the number of large particles: \(N_{p=\gamma'} N_{p=1}\). Let us assume that Al atoms are released into the solution. The number of Al atoms in small particles changed by \(\Delta N_{Al} = N_{p} \left[ \sum_j \gamma_j^3 q_i N_{pAl}(j) - \sum_j \gamma_j^3 q_i' N_{pAl}(j) \right] / g\). The number of Cr atoms released from small particles was \(\Delta N_{Cr} = N_{p} \left[ \sum_j \gamma_j' q_i N_{pCr}(j) - \sum_j \gamma_j' q_i' N_{pCr}(j) \right] / g\). According to calculations, the atomic fraction of the released elements is equal to \(\Delta C_{Al} = \Delta N_{Al} / N_{Cu} = 1.56 \times 10^{-3}\), \(\Delta C_{Cr} = \Delta N_{Cr} / N_{Cu} = 2.07 \times 10^{-4}\), where \(N_{Cu}\) – the number of Cu atoms in the alloy. \(N_{pAl}\), \(N_{pCr}\) is the number of Al and Cr atoms in one small particle of the secondary phase \(i\) and radius \(r_j\) in the 8ECAP state, respectively (table 1). After ECAP, the fraction of Cr atoms in the solution reached the value \(C_{Cr} \approx 5.08 \times 10^{-3}\), the fraction of Al atoms – \(C_{Al} \approx \Delta C_{Al}\). It was also assumed that the Zr atoms were not released into the solution. Zr atoms are strongly associated with crystal lattice defects. The volume fractions of small and large particles, according to the calculations, are equal to \(f_{secCrs} = 1.36 \times 10^{-3}\) and \(f_{secAl} = 1.42 \times 10^{-2}\), respectively. The dislocation density after 8 ECAP passes reached the value \(q \approx 1.00 \times 10^{15} \text{ m}^{-2}\) [11]. The average dislocation density in high-angle GBs, according to (3), was equal to \(q_{wh} \approx 1.054 \times 10^{14} / d = 5.27 \times 10^{15} \text{ m}^{-2}\). The average density of lattice dislocations is \(q = 0.87 \times 10^{15} \text{ m}^{-2}\). The electrical resistivity in this state was determined mainly by the Cr and Al atoms dissolved in the matrix, the thermal vibrations of atoms, and GBs (table 2).

Subsequent heat treatment led to the decomposition of the SS [3]. In the modeling, it was assumed that the atoms of Al and Cr, which passed into the solution during partial dissolution of small particles during ECAP, newly allocated in the secondary phase particles. Accordingly, the volume fraction of small particles gained a value equal to that one in the IS. But at the same time, Cr atoms were additionally separated from the solution into small particles of chromium. Based on the known value of the specific electrical conductivity of the alloy after aging, it can be assumed that the fraction of additionally precipitated Cr atoms was \(\Delta C_{Cr} = 3.94 \times 10^{-3}\). The volume fraction of fine particles of precipitated Cr, calculated according to its definition (9) by the formula \(f_{secCrs} = \Delta C_{Cr} / N_{Cr} = 3.98 \times 10^{-3}\). In the 8ECAP+A state, the fraction of Cr atoms in the solution decreased to \(C_{Cr} \approx 0.93 \times 10^{-3}\).

The average size of small particles slightly increased and became equal to 17.5 nm. It was assumed that the distances between micrometric particles and their sizes did not change significantly. As studies of the thermal stability of the UFG structure of Cu-Cr-Zr bronzes have shown [11], annealing for 1 h at a temperature of 300-500 °C does not lead to any significant changes in the microstructures formed during ECAP. Therefore, the average grain size after annealing was taken equal to 230 nm. The average density of dislocations took the value \(q \approx 2.80 \times 10^{14} \text{ m}^{-2}\) [11]. The average density of lattice dislocations, calculated taking into account the density of dislocations in GBs of the UFG material (3) equal to \(q_{wh} = 4.60 \times 10^{13} \text{ m}^{-2}\), made up \(q = 1.50 \times 10^{14} \text{ m}^{-2}\). The specific electrical resistivity of the alloy in this state in addition to thermal vibrations of atoms, is due to the contribution of GBs, as well as to the alloying Cr atoms. The contribution of small particles increased by two orders of magnitude compared to that in the 8ECAP state (table 2).
4. Conclusions
According to the modeling results, the experimentally observed character of the change in the specific electrical conductivity of the Cu-1.0Cr-0.7Zr-0.2Al alloy during thermo-mechanical treatment is possible if all Zr and Al atoms, as well as some Cr atoms, were in the particles of the secondary phases in the IS. As a result of ECAP, Cr and Al atoms passed from small particles into solution. Their partial dissolution took place. As a result of the subsequent aging, the Cr and Al atoms, which passed into the solution during ECAP, again precipitated into the particles of the secondary phases. At the same time, additional precipitation of fine Cr particles from solution into fine Cr particles occurred.

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