Investigation of thermal stability of the structure and properties of ultra-fine-grained copper alloys obtained by ECAP

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Abstract. The paper describes the results of experimental studies of the effect of small chromium additives on the temperature of recrystallization (TR) of ultrafine-grained (UFG) copper obtained by equal channel angular pressing. Our investigation shows that the process of recrystallization in UFG CuCr copper alloys has a three-stage character - anomalous grain growth at annealing temperatures not exceeding the temperature of chromium particles (T<sub>ann</sub>&lt;T<sub>1</sub>), "fixation" of grain boundaries by the released particles (T<sub>1</sub>&lt;T<sub>ann</sub>&lt;T<sub>2</sub>), and regular grain growth at higher temperatures (T<sub>ann</sub>&gt;T<sub>2</sub>), when grain boundaries "detach" from particles of the second stage. It also shows that an increase in the chromium concentration in UFG copper by 0.3-0.5wt.% leads to a decrease in the temperature of the onset of the migration of grain boundaries T<sub>1</sub> in the UFG copper by 40-60°C and an increase in temperature T<sub>2</sub> to ~400 °C. We discovered that low-temperature pre-recrystallization annealing contributes to an increase in the thermal stability of the mechanical properties of CuCr alloys.

1. Introduction

The ultrafine-grained (UFG) metals, the structure of which is formed by equal channel angular pressing (ECAP), are known to have low thermal stability - their temperature of recrystallization (TR) is 0.2-0.3Ts (Ts is the absolute melting point), which is 0.1-0.2Ts lower than that of regular metals. (In particular, after 4-12 ECAP cycles, the temperature of recrystallization of tough pitch UFG copper is ~150-200 °C) [1-3]. Low TR significantly limits the scope of practical application of pure UFG metals, which, in turn, poses a challenge of stabilizing the structure of these materials.

The most significant results on the stabilization of the grain structure of UFG materials were obtained when the refractory nanoparticles were introduced into the alloy at the stage of ingot production. In the case of UFG copper, the use of particles of Al₂O₃ [4], ZrO₂ [5, 6], HfO₂ [7] led to an increase in TR by 250-300 °C. Low TR significantly limits the scope of practical application of pure UFG metals, which, in turn, poses a challenge of stabilizing the structure of these materials.

The stabilization of UFG copper due to the release of particles during aging of a supersaturated solid solution gives a 100-200 °C increase in TR [8-10]. This is due to the fact that particles released during heating primarily along grain boundaries and cores of lattice dislocations grow rapidly, and their stabilization effect is much lower. A further increase of TR in this case, as a rule, is achieved only through the use of complex alloying systems. In particular, the authors of [11] demonstrated the...
possibility of increasing the TR to 500 °C in the UFG alloy Cu-0.63Cr-0.35Ni-0.25W-0.1Ag with a grain size of 200 nm, and in [12] the authors prove that the TR can be increased to ~ 450-500 °C in the Cu-0.7Cr-0.9Hf alloy.

However, in our opinion, the possibilities of application of the second approach to stabilizing the UFG structure are not completely exhausted. In our opinion, there is a lot of potential in the application of “control” of the nonequilibrium level and diffusion properties of grain boundaries of UFG metals, which can have a noticeable effect both on TR of UFG metals and on the intensity of the processes of separation and growth of particles of the second phase during the annealing of UFG alloys.

The aim of the work is to study the UFG copper alloys stabilized by the separation of particles of the second phase from a supersaturated solid solution.

2. Materials and methods

The object of research was the UFG CuCr industrial alloys with different chromium contents: Cu-0.3wt.%Cr, Cu-0.4wt%Cr, and Cu-0.5wt%Cr, as well as CuCrZr industrial chromium-zirconium alloy composition Cu-0.8wt%Cr-0.05wt%Zr, obtained using ECAP at room temperature. The total number of ECAP (N) cycles for MC alloys of the Cu-Cr system was 10, for the CuCrZr alloy, N = 8. ECAP was carried out in the instrument with the angle of intersection of the working and output channels equal to π/2. The deformation rate during ECAP was 0.4 mm/s. Before ECAP, the alloys were quenched in water at a temperature (1040 °C, 40 min).

Structural studies were performed using transmission electron microscope JEM 2000EX microscopes, scanning electron microscope Jeol JSM-6490 with Oxford Instruments INCA 350 EDS microanalyzer, and atomic force microscope Accurex TMX-2100. To study the mechanical properties of UFG alloys, we used the standard method for measuring microhardness $H$ (Duramin Struers -5 microhardness tester, load 50 g), as well as the compression stress relaxation test for micro samples, which makes it possible to determine the values of macroelasticity ($\sigma_0$) and yield strength ($\sigma_y$) [13].

To study the process of decomposition of a solid solution and the separation of particles of the second phase during annealing of UFG copper alloys, we used the standard four-probe method for measuring specific electrical resistivity (SER). The decomposition kinetics of the solid solution was described using the Johnson-Mel-Avrami-Kolmogorov equation: $f_v(t,T)=f_{v0}[1-\exp(-t/\tau)]$ [14, 15], where $f_{v0}$ is the maximum volume fraction of second-phase particles released from a solid solution at a given temperature T, $\tau=\tau_0\exp(Q/kT)$ is the process time scale, Q and n are the activation energy and a numerical parameter (decay intensity coefficient), respectively [15-17].

Assuming that the change in the SER of the alloy ($\Delta \rho$) is proportional to the volume fraction of the released particles $f_v(t,T)=\Delta \rho(t,T)/\rho_0$, the parameters n and Q were determined from the experimental dependences of the resistivity plotted in double logarithmic coordinates ln[ln(1-$\rho$/$\rho_0$)]-ln t. The slope of these dependences gives the value of the parameter n, and the free coefficient was equal to $n\ln\tau$ [16]. The activation energy of the process of particle separation was determined by the slope of the lnT-T dependence [16].

3. Experimental results

3.1. Microcrystalline Cu-Cr alloys

Studies of the state of the structure of Cu-Cr alloys after ECAP (N = 10) showed that severe plastic deformation (SPD) leads to the formation of a homogeneous UFG structure with a grain size of 0.2-0.3 μm. The effect of chromium additives on the grain dispersion limit is negligible.

Studies of the state of the mechanical properties of UFG CuCr alloys after ECAP (N = 10) show that the microhardness of the Cu-0.3%Cr alloy is $H_\mu=1170$-1220 MPa. An increase in the chromium content to 0.4% and 0.5% leads to an increase in the microhardness of the UFG alloy to 1280–1300 MPa and 1450–1480 MPa, respectively.
Figure 1a shows the dependencies of the hardness on temperature during 10-, 30-, 60-, and 180-minute annealing of an UFG alloy with 0.4% chromium, and Figure 1b shows the dependencies of microhardness on the temperature during an 1-hour annealing of UFG alloys with 0.3 and 0.4% chromium.

Figure 1. Dependences of the microhardness of the UFG Cu-0.4Cr alloy on the temperature during 10-, 30-, 60-, and 180-minute annealing (a), as well as dependences of the microhardness on the temperature during 1-hour annealing of UFG CuCr and CuCrZr alloys.

Figure 1 shows that the dependence $H_{\mu}(T_{\text{ann}})$ has a 4-stage character. Annealing of UFG alloy with 0.3%Cr at temperatures $T_1 \sim 140-160 \, ^{\circ}C$ does not lead to a significant increase of microhardness. Heating to a temperature of 160-180 $^{\circ}C$ is accompanied by a slight decrease in microhardness (~10-15% of the initial value). Holing of UFG Cu-0.3% Cr alloy in the temperature range $T_1<T_2<T_3$ increases microhardness to the values typical for UFG alloys after the ECAP. A further increase in the annealing temperature to $T_2 \sim 360-380 \, ^{\circ}C$ is accompanied by softening of the UFG metal to values typical for the standard coarse-grained state. The $H_{\mu}(T_{\text{ann}})$ dependences are similar for UFG alloys with 0.4% and 0.5% Cr.

Here we should note that the grain growth process in the case of UFG CuCr alloys is anomalous in nature, similar to what was previously observed in UFG copper M1. The presented figures demonstrate that the annealing of the samples of CuCr UFG alloys at temperatures above $T_1$ is accompanied by the emergence of large (~1-2 $\mu$m) recrystallized grains against a rather stable UFG matrix. A further increase in the annealing temperature leads to a slight increase in the average grain sizes of the UFG matrix and the coarse grains, but the structure of the UFG alloys remains quite stable - the average grain size of the matrix in the UFG Cu-0.4Cr alloy after annealing at 350 $^{\circ}C$ (10 h) is ~1 $\mu$m. Annealing of UFG CuCr alloys at temperatures above 400 $^{\circ}C$ leads to intensive grain growth and the formation of a coarse-grained structure (average grain size of more than 10 $\mu$m).

Figure 2. Dependence of the microhardness and changes in the SER of the UFG Cu-0.4Cr alloy on the temperature during 1-hour annealing.
Figure 2 shows the dependence of the change in SER $\Delta \rho = \rho_{\text{init}} - \rho(t)$ and microhardness of the UFG alloy with 0.4% Cr on the temperature during 1-hour annealing. Note that the decrease in SER is traditionally associated with the process of decomposition of a solid solution. Figure above shows that although the softening point of the UFG alloy is close to the point, where SER starts to decrease, it is still 20-40 °C lower than the temperature, at which the intensive change in resistivity begins. This indicates that the process of grain boundary migration begins earlier than the process of chromium particle release.

Figure 3a shows the dependences of the SER on the annealing time of the UFG Cu-0.4Cr alloy. An analysis of the dependences of the SER on annealing time in the double logarithmic coordinates $\ln[\ln(1-\rho/\rho_0)]-\ln t$ shows that these dependences have shapes of straight lines. The coefficient $n$, determined by the slope, is 0.21-0.49. The activation energy of the process of particle separation is 10.6-11.9 kTm.

3.2. Microcrystalline CuCrZr alloys

After ECAP, the microhardness $H_a = 1550-1600$ MPa. The average grain size of the UFG CuCrZr alloy is 0.45-0.5 µm. The value of SER during ECAP does not change and amounts to $\rho_0 = 5.80 \mu\Omega\cdot$cm. Figure 3b shows the dependencies of the SER of the CuCrZr alloy on the annealing time at various temperatures. The Figure presented shows that heating of the UFG alloy to a temperature above $T \sim 150-200$ °C leads to a decrease in SER due to the decomposition of a supersaturated solid solution of chromium in copper. The coefficient $n$, determined by the slope, is 0.21-0.38. The activation energy of the process of particle separation is 10.8-12.1 kTm.

Figure 4 shows the dependence of the microhardness increment of the CuCrZr alloy on the time of annealing at temperatures of 250 (a), 450 (b), and 500 °C (c), which was previously subjected to pre-recrystallization annealing at $T = 100$ °C for various periods of time. The low-temperature annealing times are shown in the figures.
Figure 4b shows the dependence of the microhardness of the CuCrZr alloy on the temperature during 1-hour annealing. The figure shows that the decomposition of the solid solution in the CuCrZr provides for an increased (in comparison with copper and CuCr alloys) microhardness increment during annealing - annealing of the CuCrZr at T = 420 °C for 1 h leads to increase in microhardness from 1600 MPa to ~2100 MPa. The obtained value ($H_\mu = 2100$ MPa) is ~ 2 times greater than the microhardness of copper and CuCr alloys after ECAP (~1150-1250 MPa) and more than 2.5-3 times higher than the hardness of these materials (~800-850 MPa) after similar annealing modes.

Figure 5 shows the dependences of the magnitude of the microhardness increment $\Delta H_\mu(t, T) = H_\mu(t,T)-H_\mu(0)$ for the CuCrZr alloy during two-stage heating. At the first low-temperature stage, the UFG material was held at a temperature of 100 °C for various periods; at the second stage - at temperatures of 250 °C, 450 °C and 500 °C for various periods. An analysis of the experimental data shows that low-temperature pre-recrystallization annealing leads to a certain degree of softening of the quenched UFG alloy, but at the same time, the use of a two-stage annealing allows for an additional increase in the strength of the UFG alloy at the stage of high-temperature isothermal holding, as well as for a decrease in its softening intensity at elevated temperatures and long exposure times (Figure 5).

4. Summary results

1. For UFG CuCr alloys, the TR decrease as the chromium content in the solid solution goes up. The experimental studies we conducted show that an increase in the concentration of chromium in solid solution to 0.5% leads to a decrease in the temperature $T_1$ by 40-60 °C compared with UFG copper.

2. Although, the decomposition point of the solid solution during annealing of the UFG CuCr alloys is close to the TR, it exceeds it by 40-60 °C. This leads to a decrease in hardness at the beginning of the grain growth process. These grains, in turn are “fixed” by the particles released during the second phase. The release of chromium particles leads to an increase in the microhardness of the UFG alloy and provides increased thermal stability up to the annealing temperature $T_2$, at which the grain boundaries “detach” from the particles of the second phase. For Cu-0.3Cr alloy, the value of $T_2$ is 360-380 °C. Moreover, an increase in the chromium content from 0.3 to 0.5% leads to an increase in $T_2$ temperature by 40-60 °C.

3. An experimental study of solid solution decomposition processes showed that the kinetic parameters of the Johnson – Mel – Avrami – Kolmogorov equation (coefficient $n = 0.25–0.5$ and activation energy $Q \sim 10–12 \text{ kT}_{\text{m}}$) are close to the theoretical values typical for particle separation on migrating grain boundaries or lattice dislocations [14, 15].

4. Low-temperature pre-recrystallization annealings of the CuCrZr alloy samples after ECAP contribute to a decrease in the intensity of softening of the UFG alloy during subsequent annealing at higher temperatures.

5. Discussion

The grain growth in an UFG alloy with particles requires the effective driving force of grain boundary migration ($P_m$) to be above zero: $P_m = P_\rho P_2 \geq 0$, where $P_\rho$ is the force that drives the movement of the
boundaries and is proportional to the volume density of lattice dislocations $\rho_v$: $P_z = \beta_1 G b^2 \rho_v$, where $\beta_1$ is the numerical coefficient, $b$ is the Burgers vector, $G$ is the shear modulus, $P_z$ is the deceleration force of migration, which results primarily from the deceleration of boundaries on dispersed particles of the second phase (Zener force): $P_z = f_v \gamma_b / R_v$, where $f_v$ is the particle volume fraction of the second phase at a given temperature, $\gamma_b$ - surface energy of a boundary, $R_v$ - a characteristic average size of second phase particles.

The equation for the speed of migration of grain boundaries in this case can be written as:

$$\dot{d} = M (\beta_1 G b^2 \rho_v - f_v \gamma_b / R_v)$$  \hspace{1cm} (1)

where $\beta_1 = 0.5 - 1$ is the numerical coefficient.

We should note that the migration temperature point in the alloy will differ from the temperature of recrystallization in the pure metal $T_1$.

Let doping atoms in the initial state be in solid solution. In this case, the volume fraction of particles of the second phase $f_v = 0$, and the Zener force preventing the migration of grain boundaries is absent. When heated to a temperature $T_1$, the boundary begins to migrate and encounters atoms of the alloying element in its path. In the case when the distribution coefficient of this element is less than unity ($K_n < 1$), the atoms tend to “move” to the grain boundary and after some time, as a result of “sweeping” of such atoms at the boundary, a supersaturation may occur, which will lead to the release of dispersed particles at the grain boundary. These released particles can stop the migration and fix the grain boundary on one place. In another case, when the distribution coefficient of the doping element is greater than unity ($K_n > 1$), the atoms “push off” from the boundary, which, and after some time, leads to a supersaturation sufficient to separate particles in the lattice in the volume “in front of” the grain boundary. Particles released in the volume can also stop the migration movement of the grain boundary.

From geometric considerations, it is not difficult to evaluate the migration path for a given concentration of atoms of alloying elements, and the volume fraction and particle size necessary for the fixation described above. It is clear, however, that at ordinary concentrations near the solubility limit in the volume, the path that the boundary will go to before the “stop” in the first and second cases will be small $x < d$, and it is difficult to clearly define it experimentally.

Thus, the boundary that begins to move in the alloy will almost immediately be “fixed” by the released particles, and the temperature of recrystallization in the alloy will be associated with the possibility of “detachment” of this boundary from such particles.

In accordance with (1), this condition can be represented as:

$$\beta_1 G b^2 \rho_v \geq f_v \gamma_b / R_v$$  \hspace{1cm} (2)

To simplify, let us assume that the separation of particles is fully completed and $f_v = f_{v*} = C (\text{at.}%)/n_c$, where $n_c$ is the fraction of alloying atoms in the released particle (for CuCr alloys in which chromium particles are released, $n_c = 1$), then in (2) the only parameter significantly varying in time is the particle size $R_v$. In this case, the condition for the onset of recrystallization - the condition for detaching the boundary from the particles - can be represented as a condition for the particle size:

$$R_v > R_1 = f_{v*} \gamma_b / \beta_1 G b^2 \rho_v$$  \hspace{1cm} (3)

The growth of particles distributed at grain boundaries at $f_v = \text{const}$ can be represented using the equation [14, 15]: $R_v^n - R_0^n = K_1 t$ where $R_0$ is the initial particle size, $m$ is a numerical coefficient, $K_1(D)$ is a parameter depending on the diffusion coefficient along the grain boundaries $D_b$ or along the cores of lattice dislocations $D_d$. We should note that in accordance with the model [15], the value of the coefficient $m$ in the particle growth law (3) is related to the value of the coefficient $n$ in the Johnson – Mel – Avrami – Kolmogorov equation, which describes the kinetics of solid solution decomposition:

$$f_v(t, T) = f_{v0} [1 - \exp(-t/t^0)^n)]$$  \hspace{1cm} (4)

The analysis of the dependences of the SER on annealing in the double logarithmic coordinates $\ln[\ln(1-\rho)/\rho] - \text{Int}$ shows that the coefficient $n$ for the CuCrZr alloy is 0.21-0.38 (average $n = 0.32$), and the activation energy of the process of particle separation is $Q = 10.8-12.1$ kTm.

In accordance with the model [15], these values of the coefficient $n$ and activation energy are close to the theoretical values $n = 0.38$ and $Q = (2Q_b + Q_s)/3$, typical for the decay during simultaneous return
and growth of grains ($p_v \neq \text{const}, \ d \neq \text{const}$), when the kinetics of grain boundary migration is related to the growth of released particles and can be described using a power law: $d^4 - d_0^4 = K_2 t$, where $K_2$ is the parameter that depends on the value of the grain-boundary diffusion coefficient.

The kinetics of particle growth under these conditions can be described using equation [15]:

$$R \sim (3a^2 D_d B_4 \lambda_\ast (C_{od} - C^\ast) / \pi \alpha_1)^{1/3} t^{1/6},$$

where $B_4 = \psi_4 (C_0 d b D_b)^{1/6} / (a^2 D_d)^{2/3}$, $\psi_4 = 0.51 / (\beta_1 f_{vb} b^2) (A \beta_0)^{1/6} (\gamma / G b)^{1/6} (G \Omega / kT)^{1/2}$, $A \beta_0 = 2/3 - \gamma_b^*/2 + \gamma_b^* / 6$, $B_4 = 0.5 \ln(1 / f_b)$, $a$ is the width of the core of the dislocation, $\gamma$ is the surface energy of the particles, $f_b$ is the fraction of the grain boundary occupied by the precipitated phase, $\lambda_\ast$ is the average distance between particles, $\alpha_1$ is a numerical parameter characterizing the fraction of atoms of alloying elements in the released particle (for chromium particles in copper $\alpha_1 = 1$), $C_{od}$ is the initial concentration of atoms of alloying elements near the core of the dislocation, $C^\ast$ is the equilibrium concentration of alloying elements in solid solution, $f_{vb}$ is the volume fraction of particles of the second phase deposited at the grain boundary.

Equating expressions (3) and (5) and assuming that $D_d = D_d 0 \exp(-Q_d / kT)$, let us determine the temperature at which, over time $t$, the particles within the grain boundaries reach size $R_1$ (necessary for “detaching” the boundary from the particles). This temperature, obviously, is the temperature of the new beginning of the boundary - the temperature of recrystallization of the alloy ($T_2$):

$$T_2 / T_m = - (Q_d / kT_m) / \ln \left[ (\gamma b / G b) (f_{vb}^* / \beta_1 \rho_v b)^4 / (6 B_1 a^2 \lambda_\ast C_{od} D_d 0 \rho_v b / \pi \alpha_1) t^{1/3} \right].$$

The resulting expression shows that the nature of the influence of the particles of the second phase on the recrystallization temperature of the UFG alloy depends not only on the diffusion parameters over the nuclei of lattice dislocations $Q_d$ and $D_d 0$, but also on the density of lattice dislocations $\rho_v$ and the maximum volume fraction of the released particles $f_{vb}^*$, the value of which in the case total decay is proportional to the total concentration of alloying elements in the alloy.

We should note that, in accordance with (6), an increase in the volume fraction of particles $f_{vb}^*$ at $D_d = \text{const}$ should lead to an increase in temperature $T_2$ in accordance with the dependence $T_2 \sim \ln(f_{vb}^*)$. Figure 6 shows the dependence of the experimentally determined temperature $T_2$ on the chromium concentration in UFG CuCr alloys at various annealing times. As can be seen from the graph, this dependence can be interpolated by a straight line with good accuracy, which confirms the correctness of expression (6). Let us analyze the effect of low-temperature pre-recrystallization annealing on the recrystallization temperature of the UFG alloy with particles.

As follows from (5), the particle growth rate during annealing of the UFG alloy depends not only on the temperature and time of annealing, but also on the diffusion properties of nonequilibrium grain boundaries. The low-temperature annealing results in a decrease in the density of defects introduced during ECAP into the grain boundaries - orientational mismatch dislocations ($\rho_b \Delta b$) and sliding components of delocalized dislocations [18]. This leads to a decrease in the coefficient of grain boundary

**Figure 6.** The dependence of the temperature $T_2$ on the concentration of chromium in UFG copper
diffusion: $D^*_b = D^*_b 0 \exp(\xi_1 t^{1/3})$ [18] and, accordingly, to a slowdown in the growth of particles of the second phase. A decrease in the particle growth rate leads, on the one hand, to a decrease in the intensity of softening of the UFG alloy in accordance with the Orowan rule and, on the other hand, to an increase in the “drag” force by particles of the migrating grain boundaries $P_z = f_v b / \Gamma_v$ and, as a result, to a decrease in the rate of migration of grain boundaries (see $\Delta H_\mu = \alpha G b \sqrt{\Gamma_v / \Gamma_v}$, (1)). This leads to an increase in temperature $T_2$, at which a “separation” of grain boundaries from particles occurs (see (6)) and an increase in the level of thermal stability of the mechanical properties of the UFG alloy, which is observed in the experiment.

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