doi: 10.18323/2782-4039-2023-2-64-6

The influence of deformation at cryogenic or room temperature followed by annealing on the structure and properties of copper and its Cu–3Pd and Cu–3Pd–3Ag (at. %) alloys

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Received 14.04.2023 *Accepted 09.06.2023*

Abstract: Due to low electrical resistivity, the Cu–Pd and Cu–Pd–Ag system alloys can be used as corrosion-resistant conductors of weak electrical signals. The paper deals with a comparison of the structure and physical-mechanical properties of Cu, Cu–3Pd and Cu–3Pd–3Ag (at. %) alloys after deformation at room or cryogenic temperature followed by annealing. The authors studied specimens in different initial states: quenched, deformed at room and cryogenic temperatures. To study the processes of structure rearrangement and the evolution of properties, annealing was carried out in the temperature range from 100 to 450 °C, followed by cooling in water. The duration of heat treatments was 1 h. The dependences of the yield strength and elongation to failure on the annealing temperature showed that cryodeformation significantly increases the thermal stability of the structure of both pure copper and the Cu–3Pd–3Ag ternary alloy. According to the temperature dependence of specific electrical resistivity of the deformed Cu–3Pd–3Ag alloy during heating at a rate of 120 deg./h, it was found that the decrease in electrical resistance caused by recrystallization begins at above 300 °C. The dependences of specific electrical resistivity on true strain showed that the structure rearrangement mechanisms during deformation are different for pure copper and the Cu–3Pd–3Ag alloy. The results of mathematical processing of the peaks in the diffraction patterns established that two phases appear in the Cu–3Pd–3Ag alloy after cryodeformation and annealing, one of which is silver-enriched, and the other is depleted. The study showed that during annealing of the deformed (especially after cryodeformation) Cu–3Pd–3Ag alloy, an anomalous increase in strength properties is observed. It was identified that alloying copper with palladium and silver leads to an increase in the recrystallization temperature. Thus, copper alloys with small palladium and silver additives are obviously attractive for practical applications, since they have improved strength properties, satisfactory electrical conductivity, and a higher recrystallization temperature compared to pure copper.

Keywords: Cu; Cu–Pd; Cu–3Pd–3Ag; copper alloy with small additives of palladium and silver; copper alloying with palladium and silver; cryodeformation; anomaly of strength properties; resistometry.

Acknowledgements: The work was carried out within the state assignment, on the topic “Pressure”, state registration No. 122021000032-5. X-ray structure analysis was performed using the equipment of the “Composition of Compounds” Shared Access Center of the IHTE UB RAS.

The paper was written on the reports of the participants of the XI International School of Physical Materials Science (SPM-2023), Togliatti, September 11–15, 2023.

For citation: Novikova O.S., Kostina A.E., Salamatov Yu.A., Zgibnev D.A., Volkov A.Yu. The influence of deformation at cryogenic or room temperature followed by annealing on the structure and properties of copper and its Cu–3Pd and Cu–3Pd–3Ag (at. %) alloys. *Frontier Materials & Technologies*, 2023, no. 2. DOI: 10.18323/2782-4039-2023-2-64-6.

INTRODUCTION

The production of high-strength and corrosion-resistant electric current conductors is an important applied research task. Many foreign companies and domestic researchers pay great attention to the development of methods aimed at the improvement of the strength properties of copper alloys, without a significant decrease in their electrical conductivity and while maintaining sufficient plasticity. Alloying is one of the ways to solve this issue. For example, the introduction of beryllium, niobium, etc. into copper can significantly improve the strength properties [1; 2]. However, the toxicity and high cost of beryllium (as well as the niobium density) hinder the application of such alloys in industry. Works on copper matrix strengthening by introducing nanodispersed particles of various metals (for example, chromium) using the severe plastic deformation (SPD) methods should also be mentioned [3]. Currently, it is generally accepted that silver is an optimal alloying element to create strong copper-based conductors. As is known,

silver is slightly soluble in copper; therefore, even its small additions (3–5 at. %) lead to significant hardening due to the precipitation of dispersed Ag-based particles in the copper matrix.

Previously, it was shown that alloying copper with silver leads to the production of an electrically conductive material with high strength properties [4–6]. The work [5] established that the redistribution of dissolved substances upon annealing of a cold-rolled Cu–3Ag (at. %) alloy, leads to the formation of a heterogeneous dislocation structure, where there are areas with low (due to a decrease in the content of silver atoms) and high (due to preservation of the excess silver concentration) dislocation density. Such a heterogeneous microstructure allowed improving the plasticity while maintaining the high strength of the annealed material [7].

As is known, SPD effectively refines the grain structure of metals and alloys and, therefore, is used as well to solve issues of increasing the strength properties of copper conductors [8]. However, the grinding process during SPD slows down after reaching a certain amount of accumulated strain, and the average grain size asymptotically tends to a certain minimum reachable value (which is usually in the submicrocrystalline range). Deformation at very low temperatures – the so-called cryogenic, or low-temperature, deformation can become one of the ways to solve this problem. It is established that very low deformation temperatures, firstly, prevent grain growth, and secondly, hinder the redistribution of dislocations, which contributes to an increase in their density and, as a result, leads to an increase in internal stresses. All this stimulates further microstructure refinement [8]. In the Cu–Ag alloys, the possibility of increasing the strength properties due to preliminary cryodeformation, for example, cryorolling [9–11] or cryodrawing [12; 13] is identified.

Recently, additional requirements for corrosion resistance have been imposed on conductors. For example, to solve this issue, copper conductors are coated with a thin palladium layer [14]. However, during the operation of such wires, palladium diffuses rather quickly, forming a set of ordered phases at the interface. In the work [15], the authors identified that alloying with palladium (less than 10 at. %) allows increasing the copper strength properties, due to solid solution hardening while simultaneously increasing corrosion resistance. However, under the conditions of increasing requirements for producing high-strength electric current conductors, hardening by alloying with one component is not enough. Therefore, to form high functional properties of alloys, currently, alloying with two or more elements is used to combine two hardening mechanisms (for example, solid solution and dispersion ones due to the second phase precipitation [16]) or simultaneous occurrence of several phase transformations [17]. Viewed in this way, Cu–Pd alloys can serve as a matrix for their additional hardening with silver.

Previously, we have shown that the application of cryodeformation before annealing leads to the formation of a high-strength ordered state of the Cu–47Pd (at. %) alloy with an ultrafine-grained structure and low specific electrical resistivity [18]. These results allow suggesting that preliminary cryodeformation will lead to additional grain refinement of the Cu–Pd–Ag ternary alloy, which, together with dispersion strengthening due to the precipitation of silver particles, will improve the mechanical properties. The influence of cryodeformation on the structure and properties of Cu alloys alloyed with silver and palladium has not been considered before.

The purpose of this work is to study the influence of deformation at room and cryogenic temperatures on the physical and mechanical properties of the Cu–3Pd–3Ag (at. %) copper alloy with small additions of palladium and silver.

METHODS

The Cu–3Pd–3Ag alloy (at. %) was smelted from copper, palladium, and silver with a purity of 99.98; 99.99, and 99.99 % respectively. To compare the structure and properties, samples of pure copper and Cu–3Pd alloy (at. %) were used. Smelting was carried out in a vacuum of 10^{-2} Pa with casting into a graphite crucible. The chemical composition of the alloys was checked using a JEOL JCXA-733 X-ray microanalyser. All heat treatments were performed in evacuated glass or quartz ampoules.

An ingot Ø5 mm was homogenized at a temperature of 800 °C for 3 h, quenched by cooling in water, and cut into two parts. From one part of the ingot, a wire Ø1.5 mm was produced by drawing, from which samples for mechanical tensile tests were cut. Further drawing to a diameter of 0.22 mm allowed obtaining a thin wire for resistometry. The other part of the ingot was rolled to produce plates with a thickness of 0.3 mm, which were used for phase composition assessment at various stages of processing.

The alloy cryodeformation was carried out between two stainless steel plates. This construction was placed in liquid nitrogen for about 1 min, after which rolling was performed. This operation was repeated. Part of the wires and plates deformed at room temperature were annealed at a temperature of 700 °C during 1 h and cooled in water. Thus, the authors studied samples in several initial states: quenched, deformed at room temperature, and deformed at cryogenic temperature. The value of the true deformation of the samples (e) was determined by the equation: $e=\ln(S_o/S_f)$, where S_o and S_f are the cross-section areas of the sample in the initial and final states.

To study the processes of structural rearrangement and evolution of properties, annealing was carried out in the temperature range from 100 to 450 °C (with a step of 50 °C) followed by cooling in water. The duration of heat treatments was 1 h.

To measure specific resistivity (ρ), the standard four-contact method was used (DC value is I=20 mA). To increase the accuracy in calculating the cross-section area of the sample, the wire diameter was measured by optical methods with an error of ±1 μm. When determining the conductor length, a special conductor was used with a set of contact points, the distance between which (from 120 to 150 mm) was measured with an accuracy of 0.1 mm. The specific electrical resistivity was calculated as the average value of 5 measurements between different pairs of contacts. The electrical resistivity temperature dependences were obtained by heating and cooling the samples at a rate of 120 deg/h.

Mechanical tests were carried out on a ZD 10/90 tensile testing machine at a tensile rate of 3 mm/min; the length of the working part of the samples was 30 mm. For each structural state, at least five samples were tested.

X-ray diffraction analysis (XRD) was performed using a DMAX 2200 diffractometer (Rigaku) in the shooting mode at a speed of 4 °/min, Cu-*K*α radiation was monochromatised by a graphite single crystal. The average size (D_0) of coherent scattering regions (CSR) in the studied alloys deformed to the same degree at different temperatures was estimated based on the X-ray diffraction data using the Williamson–Hall method [19]. The authors carried out mathematical processing of several peaks on X-ray diffraction patterns of copper, Cu–3Pd alloy, and Cu–3Pd–3Ag ternary alloy. It involved deconvolution, i.e., inverting the signal convolution with the broadening function of the device. This inversion was carried out by solving the convolution integral equation, using the regularisation method for inverse problems [20]. The lattice parameter corresponding to each component of the original peak was calculated for both peaks of its doublet and averaged.

RESULTS

The obtained XRD results show that the introduction of small palladium or silver additions into copper causes an increase in the fcc lattice parameter (Fig. 1, Table 1). For visual clarity, the inset in Fig. 1 shows the peaks (200) for pure copper, the Cu–3Pd binary alloy, and the investigated Cu–3Pd–3Ag ternary alloy. It is clearly seen that alloying leads to a shift of this peak towards smaller angles.

Moreover, Fig. 1 shows that the plastic deformation of the studied alloys by the same degree ($e\approx3.5$) causes a different broadening of the X-ray peak. One can conclude that alloying leads to refinement of structural elements.

As we can see from Table 1, the CSR size in deformed binary and ternary alloys is several tens of nanometers. Cryorolling further refines the structure. Therefore, the studied Cu–3Pd and Cu–3Pd–3Ag alloys after deformation ($e\approx3.5$) can be attributed to nanostructured materials.

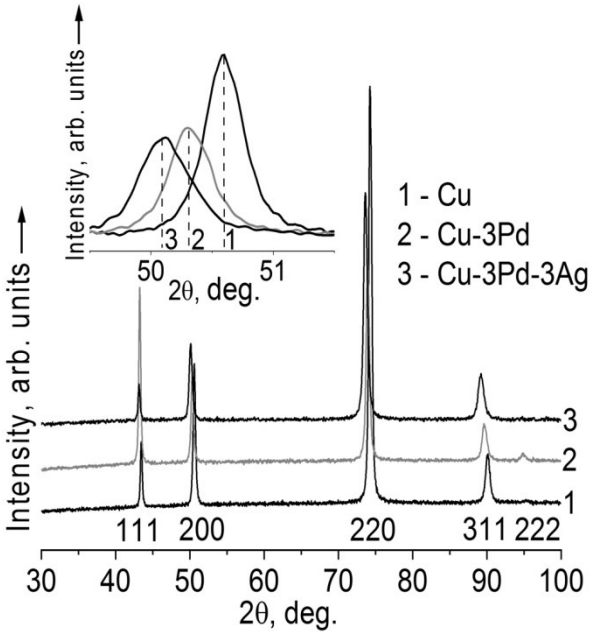


Fig. 1. *Diffraction patterns of copper (1), Cu–3Pd (2) and Cu–3Pd–3Ag (3) alloys after cryodeformation (e≈3.5)*

Table 1. *Crystal lattice parameter and the average size of the areas of coherent scattering of deformed (e≈3.5) specimens of the studied alloys and copper*

Specimen	Lattice parameter *a*, nm	CSR average size D_0, nm
Cu*	0.3619	>100
Cu–3Pd	0.3627	66±7
Cu–3Pd*	0.3628	50±5
Cu–3Pd–3Ag	0.3639	58±6
Cu–3Pd–3Ag*	0.3645	40±4

** Specimens produced by cryodeformation.*

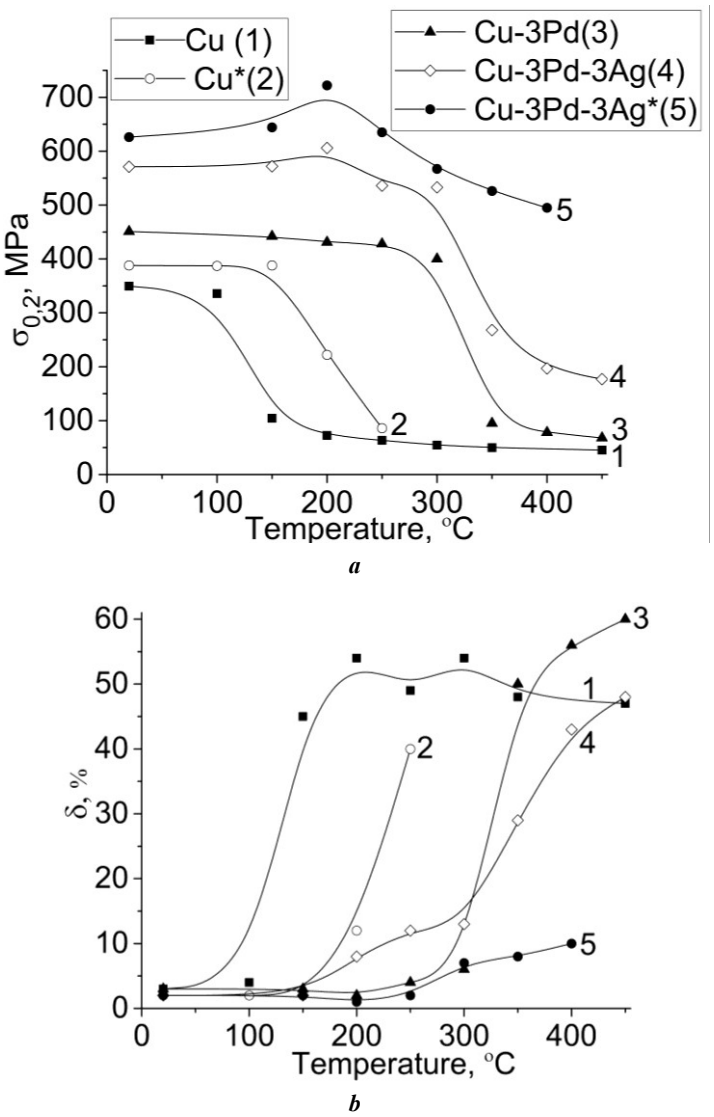
The change in yield strength ($\sigma_{0.2}$) and relative elongation to failure (δ) after exposure (for 1 h) of pre-deformed samples of Cu–3Pd and Cu–3Pd–3Ag alloys in the temperature range from 100 to 450 °C is shown in Fig. 2 a and 2 b, respectively.

It is seen (curve 1, Fig. 2 a) that to remove cold hardening in copper specimens, annealing at a temperature of 150 °C for 1 h is sufficient. The introduction of 3 at. % palladium increases the recrystallisation temperature to ~300 °C (curve 3, Fig. 2 a), and the additional introduction of 3 at. % silver increases the recrystallisation temperature by another ~50 °C (curve 4, Fig. 2 a).

Plasticity of all heavily deformed samples is low: their elongation to failure is $\delta \approx 2 \div 3$ %. After the completion of recrystallisation processes, as a result of annealing, the plastic properties of most alloys increase significantly – up to $\delta \approx 50$ %. It can be assumed that in the cryodeformed Cu–3Pd–3Ag alloy annealed at 400 °C during 1 h, the recrystallisation processes have not yet been completed (curve 5 in Fig. 2 b). Therefore, cryodeformation significantly increases the thermal stability of the structure of both pure copper (curves 1 and 2) and the Cu–3Pd–3Ag ternary alloy (curves 4 and 5).

Since the decrease in the structure defectiveness during recrystallisation leads to a drop in the electrical resistivity, the thermal stability of the deformed structure can be understood by measuring the temperature dependences of the electrical resistivity. Fig. 3 shows the dependences of the change in electrical resistivity obtained during heating and cooling of a thin wire sample of the Cu–3Pd–3Ag alloy deformed at $e \approx 7.1$. The temperature dependence of the electrical resistivity of the initially quenched alloy has a linear form, since in this case recrystallisation processes do not occur in the alloy and are not presented in the work. The dependences of the electrical resistivity during cooling are the same regardless of the initial state of the samples and are also linear (dashed line in Fig. 3).

The temperature of recrystallisation of the Cu–3Pd–3Ag alloy can be determined in Fig. 3 as the point of deviation of the electrical resistivity dependence from the linear form during heating. Here it should be noted that the recrystallisation temperature depends on the temperature-time processing conditions [21]. Therefore, one can argue that, upon heating at a rate of 120 deg/h, the decrease in the specific



a

b

Fig. 2. *The dependences of yield strength (**a**) and elongation to failure (**b**) on the temperature of annealing of specimens of Cu, Cu–3Pd and Cu–3Pd–3Ag alloys pre-deformed at room and cryogenic (*) temperatures (e≈2.3)*

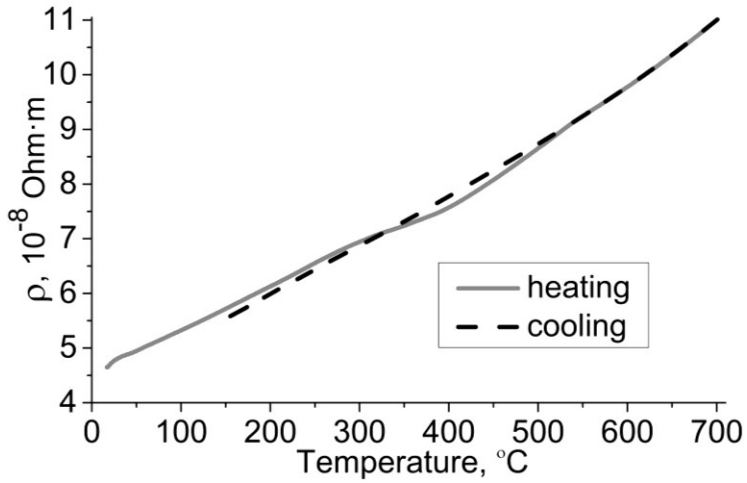


Fig. 3. *Change in electrical resistivity at heating and cooling at a rate of 120 deg./h of the Cu–3Pd–3Ag alloy specimen drawing-deformed at room temperature (e≈7.1)*

electrical resistivity of the studied alloy caused by recrystallisation begins upon heating above 300 °C.

At room temperature, the specific electrical resistivity of the quenched Cu–3Pd–3Ag alloy is $\rho=4.5\cdot10^{-8}$ Ohm·m (i.e. the alloy electrical conductivity is 39 % IACS). The electrical resistivity of the deformed Cu–3Pd–3Ag alloy is slightly higher (this also follows from the course of the "heating – cooling" curves in Fig. 3).

The microstructure evolution under the influence of deformation is well revealed by the change in specific electrical resistivity. To compare, Fig. 4 shows the dependences of specific electrical resistivity on the true deformation of samples of pure copper and Cu–3Pd–3Ag alloy. During plastic deformation, the electrical resistivity of copper (Fig. 4, curve 1) increases by ~4 % due to an increase in the structure defectiveness; the maximum value of the electrical resistivity corresponds to the true deformation $e\approx3.5$. Further deformation of copper does not lead to an increase in its electrical resistivity, since a dynamic equilibrium occurs between the generation of defects, and their annihilation due to recovery/recrystallisation processes. That is why at large deformations, the dependence of the specific electrical resistivity of copper looks like a plateau (curve 1 in Fig. 4).

Fig. 4 (curve 2) clearly demonstrates that during plastic deformation, the electrical resistivity of the Cu–3Pd–3Ag alloy increases by approximately 7 %, the maximum value of the electrical resistivity corresponds to the true deformation $e\approx4.3$. Further deformation leads to a decrease in the specific electrical resistivity of the ternary alloy. Therefore, the mechanisms of structural rearrangement during deformation are different for pure copper (curve 1) and the Cu–3Pd–3Ag alloy (2).

Fig. 5 shows the diffraction patterns of the Cu–3Pd–3Ag alloy samples deformed by $e\approx3.5$ at room and cryogenic temperatures and then annealed at 250 °C for 1 h.

All diffraction patterns contain the lines of only the fcc phase; no reflections of other phases were found. It is seen that a decrease in the deformation processing temperature

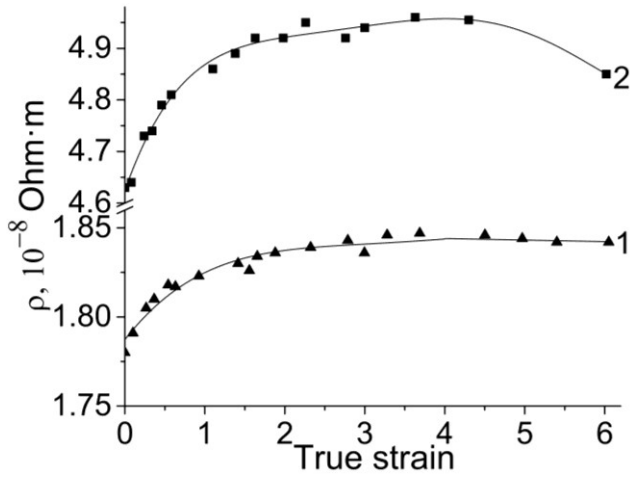


Fig. 4. *The dependences of specific electrical resistivity on true strain of pure copper (1) and Cu–3Pd–3Ag alloy (2)*

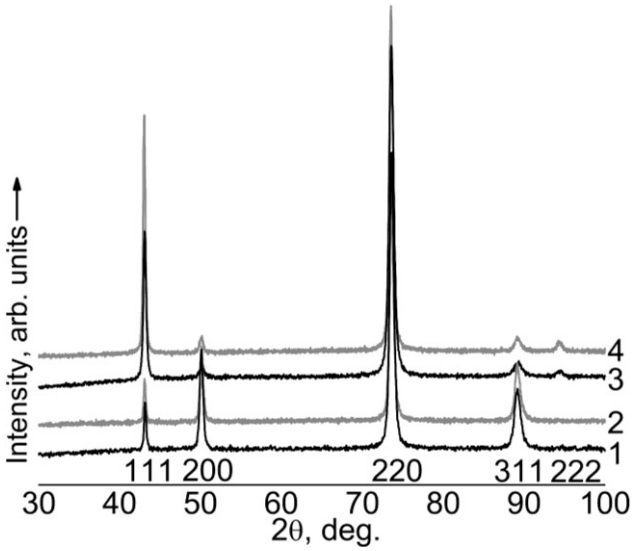


Fig. 5. *Diffraction patterns of the Cu–3Pd–3Ag alloy specimens:*
1 – deformation at room temperature (e≈3.5); 2 – treatment (1) + 250 °C, 1 h, cooling in water;
3 – cryodeformation (e≈3.5); 4 – treatment (3) + 250 °C, 1 h, cooling in water

causes an additional broadening of the X-ray peaks (diffractograms 1 and 3). Obviously, to reveal possible weak lines of the second phase, the use of methods of mathematical processing of X-ray peaks is required.

Let us present the results of such processing of one of the peaks. After the peak deconvolution (220) of the Cu–3Pd–3Ag alloy (cryodeformation (e≈3.5) + + 250 °C, 1 h, cooling in water), it is seen (Fig. 6) that it is clearly divided into two components, each of which is a doublet of Cu-$K\alpha_1$ and Cu-$K\alpha_2$. All other peaks in Fig. 6 are undescriptive, and are either noise or a consequence of the approximate solution of inverse problems by the regularisation method. For comparison, the authors carried out the same mathematical processing of the peak (220) of pure copper and the Cu–3Pd alloy: in contrast to the ternary alloy peak (220), after deconvolution they are divided only into a doublet Cu-$K\alpha_1$ and Cu-$K\alpha_2$, without revealing signs of the presence of the second component.

For phase 1 (left doublet, more intense), the lattice parameter was a_1=0.3646 nm. For phase 2 (right doublet, less intense), a_2=0.3632 nm. As expected, both parameters exceed the crystal lattice parameter of pure copper a=0.3619 nm.

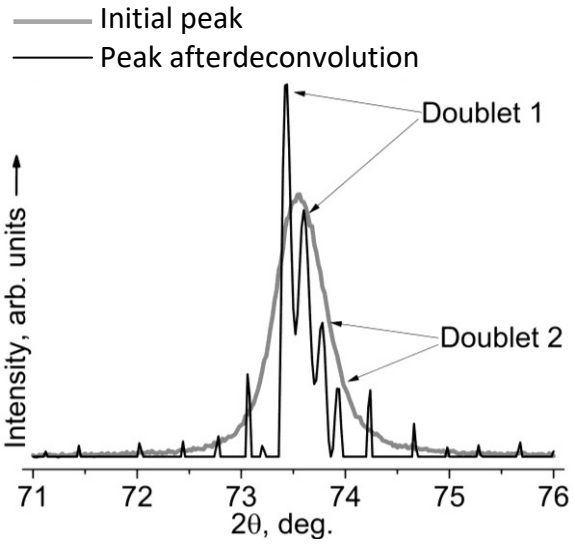


Fig. 6. *The initial peak (220) and peak after deconvolution on the diffraction pattern of the Cu–3Pd–3Ag alloy, cryodeformation (e≈3.5) + 250 °C, 1 h, cooling in water*

DISCUSSION

The most interesting result obtained during the study is the detection of a temperature anomaly in the yield strength during annealing of the Cu–3Pd–3Ag alloy. As a rule, a decrease in strength properties can be expected when annealing an initially deformed material. However, Fig. 2 a demonstrates that the annealing of the ternary alloy at a temperature of 200 °C "switches on" some additional hardening mechanism compared to the binary alloy. Indeed, in the Cu–3Pd alloy, no increase in strength occurs as a result of annealing (Fig. 2 a, curve 3 shows a plateau). In turn, the annealing of the Cu–3Pd–3Ag alloy at 200 °C after deformation at room temperature increases its yield strength by ~40 MPa, while cryodeformation increases the yield strength by ~100 MPa (compare curves 4 and 5 in Fig. 2 a). After such thermomechanical treatment, the yield strength of a low-alloyed copper-based Cu–3Pd–3Ag alloy ($\sigma_{0.2}$=720 MPa) becomes 2 times higher than the yield strength of heavily deformed pure copper ($\sigma_{0.2}$=350 MPa).

The effect of an anomalous increase in the yield strength discovered during mechanical tensile tests (Fig. 2) is observed as well when measuring the microhardness of the Cu–3Pd–3Ag alloy [22]. The ternary alloy microhardness increases after annealing at 150 °C, reaches a maximum at 250 °C, and then begins to decrease. Note that the effect of hardening as a result of annealing is more pronounced on the microhardness curves. This is probably associated with the difference in the degrees of preliminary deformation of the samples. Indeed, the pre-deformation of the plates for microhardness measurements was $e\approx3.5$, and the true deformation of the wire for tensile tests did not exceed $e\approx2.3$.

An anomalous increase in strength properties as a result of annealing has already been observed earlier, for example, on cryodeformed samples of Cu–Ag, Cu–Al–Zn, and Mg–Al–Zn alloys [23; 24]. This effect was explained by the segregation of atoms of the released component on various kinds of defects (dislocations, grain boundaries, etc.). Therefore, an increase in the structure defectiveness during cryodeformation should lead to a more pronounced manifestation of the temperature anomaly, which corresponds to our results obtained for the Cu–3Pd–3Ag alloy (Fig. 2).

Based on the results of mathematical processing of XRD data, one can assume that the main volume of the studied alloy is a solid solution of palladium in copper. According to [25], the change in the fcc lattice parameter when alloying copper with palladium completely satisfies the Vegard law. Thus, in full agreement with the results obtained, the crystal lattice parameter of the Cu–Pd matrix should slightly exceed the lattice parameter of pure copper.

In the Cu–5Ag alloy (wt. %), after aging at a temperature of 450 °C, a grid of silver precipitates along the grain boundaries was observed [26]. In the Cu–8Ag alloy (wt. %) after annealing at a temperature of 500 °C for 710 h, larger silver precipitates were found along the grain boundaries and at triple junctions, and fine precipitates were found inside the grains [27]. Taking into account the results of [26; 27], as well as the low solubility of Ag in Cu, one can assume that the segregation of silver atoms occurs along the grain boundaries of the Cu–Pd matrix, as well as inside them, at dislocations. Precipitation of a silver-based phase has been repeatedly observed earlier in Cu–Pd–Ag alloys with a high content of palladium and silver. For example, using the method of field ion microscopy of the Cu–50Pd–20Ag (at. %) alloy, the formation of Pd–Ag particles was observed in an atomically ordered Cu–Pd matrix [28].

In the low-alloyed Cu–3Pd–3Ag alloy studied in this work, no ordered phase is formed. Apparently, after cryodeformation and annealing at a temperature of 250 °C, two regions appear in the alloy, one of which is silver-enriched and the other is depleted. The components of the diffraction peak, which were observed after deconvolution, correspond to this two-phase state.

In further studies of Cu–Pd–Ag ternary alloys, one can follow the way of increasing the palladium content. Silver alloying of Cu–5Pd and Cu–10Pd alloys, in which the formation of nuclei of the ordered Cu_3Pd phase can be expected, will lead both to solid solution strengthening and precipitation age hardening due to the second phase precipitation, and to additional hardening due to atomic ordering processes. Probably, this will make it possible to increase noticeably the strength properties of such alloys, as was proposed for the alloyed ordered alloys based on Cu–Au and Cu–Pd in the work [29].

If to compare binary Cu–Pd and ternary Cu–Pd–Ag alloys, it should be noted that small silver additions affect slightly the electrical conductivity of Cu–Pd alloys at a simultaneous significant increase in strength properties and recrystallisation temperature. For example, the yield strength and tensile strength of the Cu–3Pd–3Ag ternary alloy are higher, and its electrical conductivity is comparable to the characteristics of the Cu–3Pd alloy.

Thus, from a practical perspective, copper alloys with small palladium and silver additions are of obvious interest, since they have increased strength properties, satisfactory electrical conductivity, and a higher recrystallisation temperature compared to pure copper.

CONCLUSIONS

1. An anomalous effect of an increase in strength properties during annealing of a deformed Cu–3Pd–3Ag alloy was identified; cryodeformation enhances significantly this effect.

2. Alloying copper alloys with a low palladium content with a small amount of silver leads to an increase in the strength properties and recrystallisation temperature, while the observed decrease in electrical conductivity is insignificant.

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