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Influence of cold working on microstructure and properties of annealing CuTi4 alloy

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Properties

ABSTRACT

Purpose: The aim of this study was to investigate the effect of cold plastic deformation of the supersaturation on the structure and properties of the CuTi4 alloy after aging.

Design/methodology/approach: CuTi4 alloy of supersaturation temperature of 900°C after heating for 1 hour. After solutioning alloy was processed in two ways: first aged in the temperature range 450-600°C and the second stage rolling reduction with Z=50%, and then aged in the temperature range 450-600°C.

Findings: The results confirmed that the temperature within the range 500-600ºC, the hardness increases with increasing aging time until reaching the maximum, but then with increasing aging time the hardness decreases. By using methods of electron microscopy (SEM, EDS, EBSD, TEM) after aging at 550°C after 1 minute of modulated microstructure was observed - characteristic for the spinodal transformation and lamellar, formed by nucleation and growth.

Research limitations/implications: A widely used method for increasing the strength properties of metal alloys, in addition to cold plastic deformation, is the strengthening of new phases separated particles during aging. The effect of cold rolling operation between solutioning and aging on microstructure and properties of alloyed copper CuTi4. Further examination also included the effect of time and aging temperature.

Practical implications: On the basis of conductivity, the influence of cold plastic deformation and subsequent aging on the hardness and electrical conductivity of the alloy CuTi4. It was found that with increasing aging time and with increasing aging temperature increases electrical conductivity of the alloy. On the basis of X-rays can be concluded that in alloyed copper containing 4% Ti and precipitation hardening metastable phase β'-Cu4Ti is separated, which occurs both in the previously deformed and undeformed cold worked alloy.

Originality/value:

Keywords: Metallic alloys; Electron microscopy; Heat treatment; Cold deformation; CuTi4 alloy

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1. Introduction

Both copper and copper alloys are very flexible and can be plastically cold deformed with severe draft what causes the strengthening of the alloy. To remove the effects of strain annealing is used, whose parameters depend on the degree of draft and chemical composition of the alloy. Large impact on the selection of parameters are also the dimensions of intermediates and type of the furnace [1,2].

Except the cold working a small addition of alloying elements (up to 2%) increases the mechanical properties of copper with a slight reduction in conductivity. These alloys are called alloyed copper. Commonly used copper and a little silver, phosphorus, arsenic, chromium, nickel, tin, zinc, cadmium, beryllium, titanium, sulfur, manganese, iron, silicon, tellurium, and zirconium. In general, these alloys are classified as low-alloyed copper alloys [3-12].

A special group is a copper-beryllium, chromium, titanium and iron and multicomponent alloys - copper-nickel-silicon-chrome. A good application properties such as high tensile strength, good electrical conductivity, stable mechanical properties at elevated and high temperatures now provide a wide range of applications [13].

The best properties of strength and good electrical conductivity of copper alloys are characterized by beryllium bronzes [14]. These alloys are called traditionally beryllium bronzes [15], which, despite the high toxicity and difficulties in its production technology and processing [13] are widely used due to high mechanical properties and resistance to corrosion and abrasion. One of the major advantages of these alloys are not prone to arcing. This advantage predisposes CuBe alloys for industrial production of inflammable and explosive materials, the non-sparking tools in chemical, petroleum, and the inserts for molds [16].

A major disadvantage of eliminating the Cu-Be alloys in industrial applications is the toxicity of beryllium oxides that cause heart and lung disease. Recent studies confirm the deleterious effects of beryllium compounds on the human body even during hot forming and during welding, cutting or grinding of Cu-Be alloys. For this reason, a number of years a total ban on melting of beryllium bronze in the European Union [17].

The result of search for alternative replacements for Cu-Be alloys are working on the CuNiSi and CuTi alloys [18-20]. CuTi alloys compared beryllium bronzes are characterized by similar electrical properties and comparable mechanical properties. The most effective way to increase the strength properties of alloys is precipitation hardening CuTi.

Analysis of microstructure development in alloys Cuti solutioning and aging made based on a literature review, allows the systematization of knowledge about the mechanisms that control recrystallization and precipitation in copper alloy CuTi. Work is also underway on the evolution of the microstructure by the introduction of micro (and even ppm) of alloying elements mainly micro-addition of boron, zirconium, etc.. influencing fragmentation of the microstructure and stability of grain size and sub-grains during use under varying conditions of thermal or mechanical loads [21-24].

Literature data [25-28] allow the conclusion that the most commonly used method of increasing the strength properties of alloys CuTi is precipitation hardening, resulting in the metastable phase Cu4Ti is separated, which determines the properties of utility these alloys. The CuTi alloys solutioning is carried out in the temperature range 800-950°C for 10 hours with subsequent aging at 400-600°C for 1 to 48 hours [29-31].

In addition, work is underway on a variant of combined treatment consisting of heat treatment and hot plastic deformation [32], which heavily increases the strength properties of copper alloy CuTi. However, the greatest strengthening guarantees alternating heat treatment and plastic deformation involving operations sequence solutioning-cold rolling-aging-cold rolling [13].

Despite numerous studies and publications on the changes that occur during heat treatment (solutioning and aging), there is still a need to clarify the description of the separation and recrystallization mechanisms occurring during aging, which was preceded by cold plastic deformation with a high degree piece of draft. As demonstrated by studies [33-35] so carried out alternating heat and cold plastic deformation is beneficial to increase the functional properties of these alloys. The few works devoted to this variant treatment [36-39] make it necessary to clarify the description of the mechanisms of recrystallization and precipitation in alloys subjected to alternating CuTi heat treatment and plastic deformation.

Knowledge of the mechanisms of precipitation and recrystallization is necessary to determine the microstructural changes that should be considered in assessing the functional properties of alloys CuTi.

2. Material and methods

Investigations were carried out on the commercially available CuTi4 alloy. Chemical composition of the industrial CuTi4 alloy is presented in Table 1.

The material preparation procedure for investigation of CuTi4 alloy included:

- hot working with 80% draft to 3.0 mm thickness,
- supersaturation (920°C/1h) in water,
- cold working with 50% draft,
- ageing at temperature of 550°C for 1 min, 60 min, and 420 minutes.

Examinations of microstructure and grains misorientation were made on ZEISS SUPRA 25 scanning electron microscope (SEM) using the EBSD (Electron Backscatter Diffraction Analysis) method and with JEOL 3010 transmission electron microscope (TEM).

X-ray phase analysis of the specimens was made on Panalytical X'Pert diffractometer using filtered radiation of the lamp with cobalt anode. The measurement step was 0.05° and the impulse counting time was 10 sec.

Table 1.
Chemical composition of CuTi4 alloy

Cu	Ti	Zn	P	Pb	Sn	Mn	Ni	Sb	Bi	As	Cd
95.83	**3.95**	0.13	0.065	0.003	0.009	0.030	0.01	0.001	0.001	0.001	0.001

3. Results and discussion

According to study feature, the obtained results can be given in discussion part and/or in the conclusion section.

3.1. Hardness

The results of hardness measurement of CuTi4 supersaturated alloy and then aged shown in Fig. 1. However, the results of hardness measurement CuTi4 supersaturated alloy, cold deformed (Z=50%) and aged shown in Fig. 2. The hardness of the alloy after solution is CuTi4 HV=125, while the deformation after solution and HV=250.

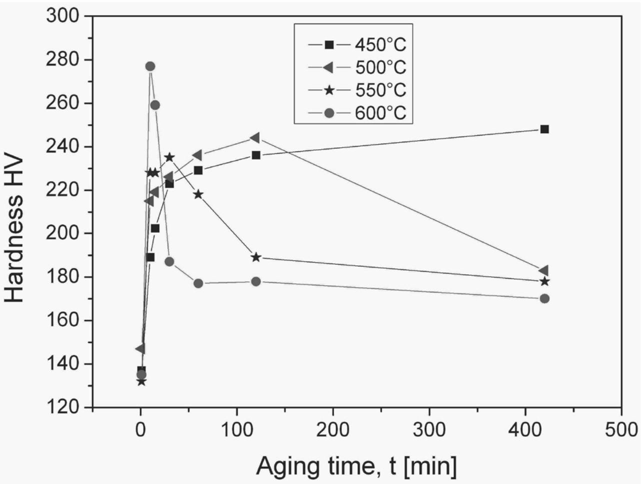

Fig. 1. Changes in hardness of the supersaturated alloy CuTi4 depending on temperature and aging time

The aging of the investigated alloy over 120 minutes in the temperature range 450°C results loss in partial of coherence of the second phase precipitates and reduction of dislocation density in the matrix in the deformed cold worked alloy before aging (Z=50%) which leads to a reduction in hardness. To undeformed alloy recorded the continuous growth of hardness in range investigated. The other hand the deformed alloy, aging at 500°C over 30 minutes result in the decrease of hardness while the for undeformed alloy after 120 min.

Then, with increasing aging temperature drop in hardness for the deformed alloy is, even after 30 minutes, but quite generally: the value of 265 HV to 150 HV (Fig. 1, Fig. 2).

The microstructure formed as a result of aging in the CuTi alloy, which consists of hardened grains and soft nucleus of recrystallization is unfavorable due to the mechanical properties. The best microstructure, which determines the favorable properties of the alloy obtained after aging at 400°C [11,31] because of the runs much slower recrystallization process compared to the process of precipitation. After aging, during which ensures maximum hardness was observed in the microstructure only single nucleuses of recrystallization. Recrystallization process is slow due to low mobility boundaries of subgrains.

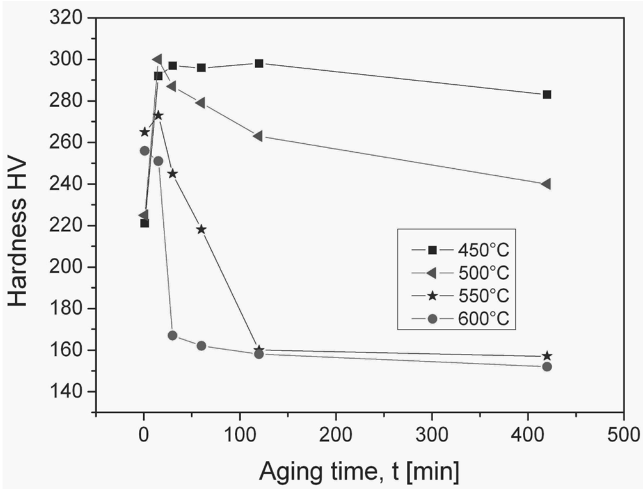

Fig. 2. Changes in hardness of supersaturated and deformed (50%) alloy CuTi4 depending on temperature and aging time

Similar result was obtained in [22,24,29]. The optimal structure ensuring the most favorable mechanical properties and especially the hardness was obtained after aging at 400°C. In addition, with increasing titanium additive increases hardness against aging, or in the initial stage of aging. With increasing concentration of titanium as one of the major copper alloy (Al, Be, Ni, Si, Sn, Zn), most strengthens the solid solution resulting, inter alia, increased fatigue strength. Later, however, with increasing aging time and temperature the hardness decreases after 32 hours of reaching the hardness of the alloy aged at 400°C. However, it should be noted that the alloy aged at 400°C is characterized by a more favorable structure in terms of mechanical properties.

With the strengthening of supersaturated CuTi alloys and cold deformed corresponds to metastable, coherent with the matrix phase Cu_4Ti [38,39] and the strengthening due to alternating heat treatment and cold plastic deformation [22]. Strengthening during aging causes an increase in wavelength and amplitude modulation. In alloys subjected to alternating heat treatment and plastic surgery wavelength modulation increases faster than the heat treated alloys. This results in a greater increase in hardness of the deformed alloys during aging.

3.2. Electrical conductivity

On the basis of data drawn the graphics dependences, which are contained in Figure 3 & 4. Effect of cold plastic deformation and subsequent aging on the electrical conductivity of the alloy CuTi4 shows on Figure 4. It is clear that with increasing aging time and with increasing aging temperature electrical conductivity increases. However, at 600°C after 100 minutes of aging with increasing aging time electrical conductivity decreases.

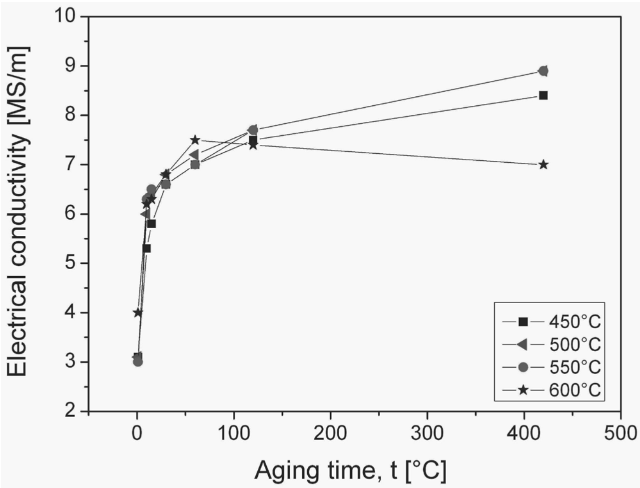

Fig. 3. Changes in electrical conductivity of the supersaturated alloy CuTi4 depending on temperature and aging time

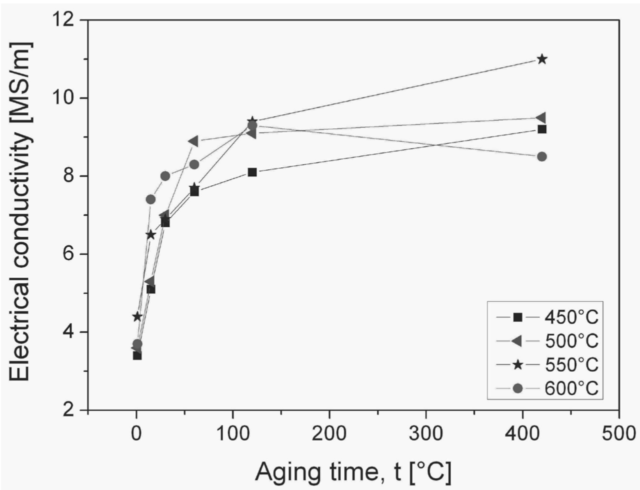

Fig. 4. Changes in electrical conductivity supersaturated and deformed (50%) alloy CuTi4 depending on temperature and aging time

3.3. Microstructure

Figure 5 shows the microstructure of the alloy after solution CuTi4 and subsequent cold rolling with a draft degree Z=50%. The bright field image (Fig. 5a) clearly shows deformation twins in addition also shown in the picture in the dark field (Fig. 5c), which comes from the Cu matrix reflexes. Solution diffraction has identified the second phase - Cu_3Ti.

In the Cu4Ti alloy saturated and cold rolled with a draft degree Z=50% can be seen in micro recrystallized subgrains (A) at the grain boundary (Fig. 6a). A subgrains border on the right, downwards as dislocations are visible (indicated by an arrow in the figure).

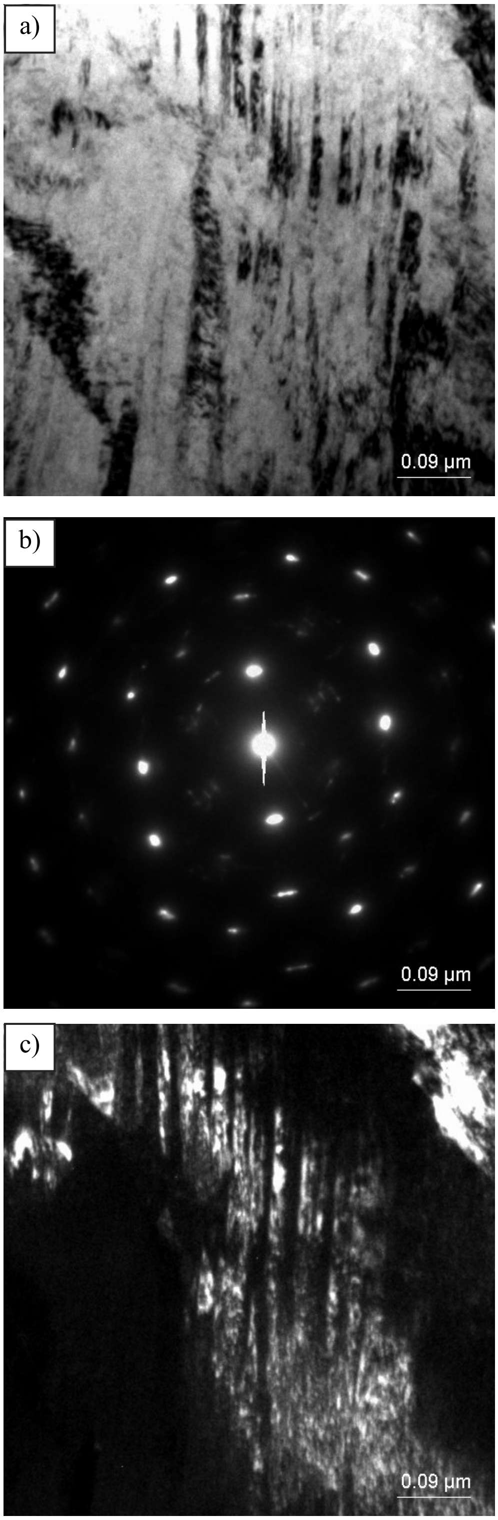

Fig. 5. The structure of the alloy CuTi4 a) bright field image, b) diffraction pattern from the area as shown in a); with the solution of the diffraction pattern, c) dark field image of the reflexes of Cu matrix lattice (space group Fm-3m)

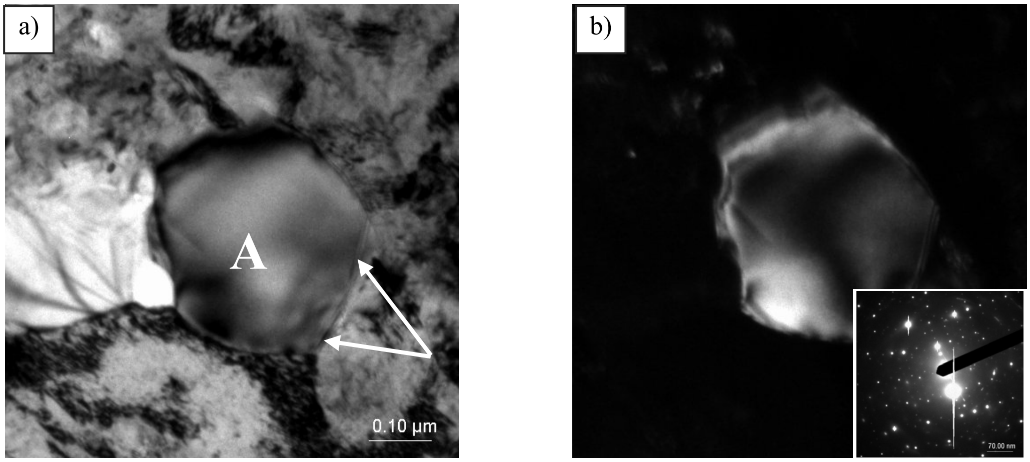

Fig. 6. Microstructure of CuTi4 alloy supersaturated at 930°C then cold rolled with 50% draft degree a) bright field image, b) dark field image phase with reflexes $2\bar{2}\bar{2}$ CuTi2 crystallization in the cubic lattice (space group Fd-3m) with diffraction pattern from the area as shown in a)

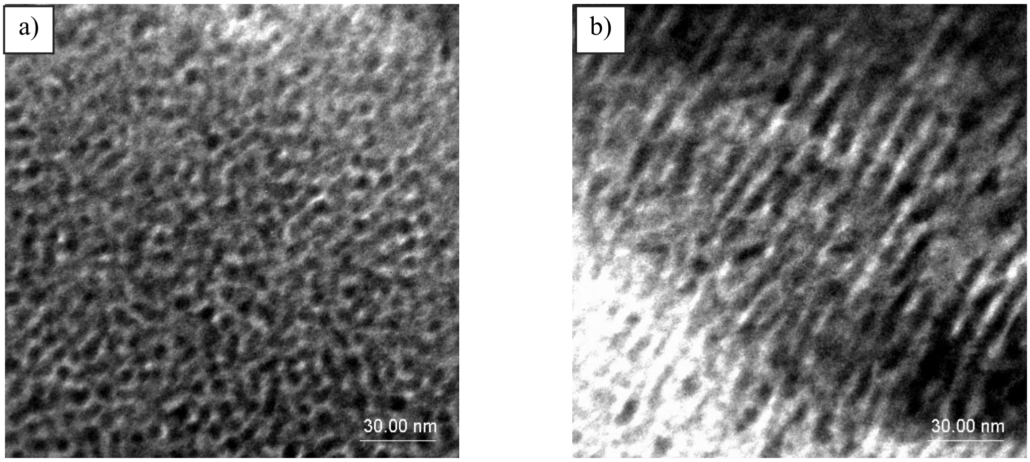

Fig. 7. Area of a continuous precipitation a), and discontinuous in the alloy CuTi4 aged for 1 minute at 550°C b)

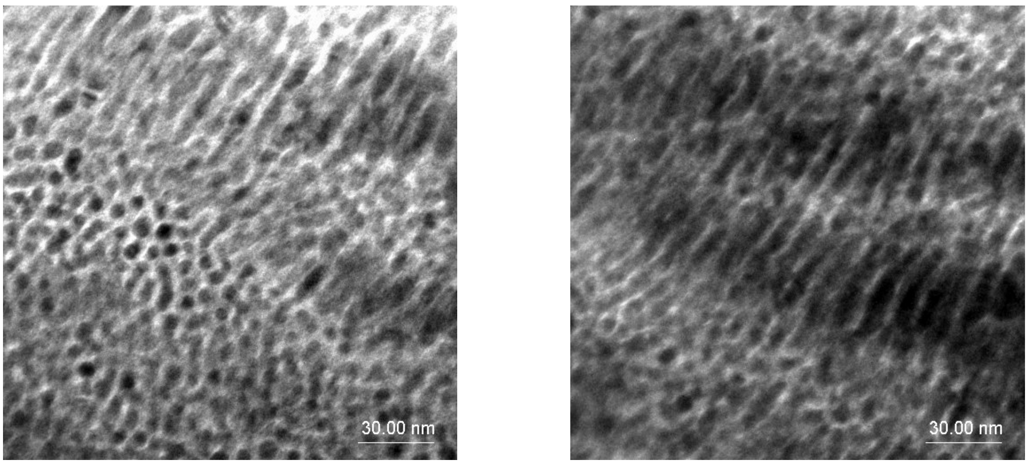

Fig. 8. The border area of discontinuous and continuous precipitation in the alloy CuTi4 aged for 1 minute at 550°C

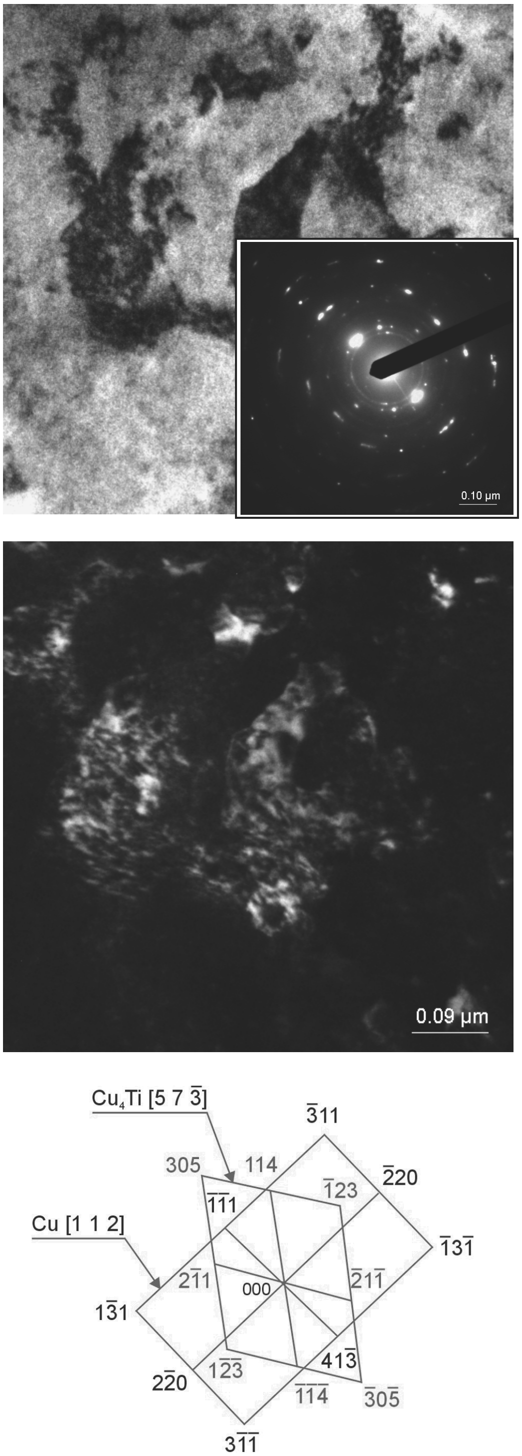

Fig. 9. CuTi4 alloy structure after aging at 550°C for 120 min: a) bright field image;b) diffraction pattern from the area as shown in a), d) the solution of the diffraction pattern Figure b, c) dark field image of the reflex phase crystallization in the lattice Cu_4Ti orthorhombic (space group Pnma), the diffraction pattern is also visible Cu matrix network - crystal zone [112]

The next stage of the experiment was to stop aging. After aging at 550°C per 1 minute the modulated microstructure was observed (which was not observed after 15, 30, 120 or 420 minutes of aging) - characteristic for spinodal transition (Fig. 7.a) and the plate, formed by nucleation and growth (Fig. 7.b). In Figure 8 shows the boundaries between areas of discontinuous and continuous precipitation. Based on the analysis of these microstructures were found in both the secretion process of continuous and discontinuous precipitation proceed at 550°C after 1 min of aging already. Similar results were obtained in [40].

Changes in properties of the alloy as a result of precipitation hardening is the result of ongoing structural changes [41-45]. Precipitation-hardened alloys are characterized by a continuous phase which is the significant part of the volume of the alloy and the occurrence of the second phase (separated particles) strengthening the alloy (Fig. 9). Enhancing particles should be hard, as well as small, high density, uniformly distributed in the volume of the alloy and at least partially coherent. Separation should not have sharp edges and should not form the matrix grain boundaries continuous film. This prevents the nucleation of cracks and their spread.

4. Conclusions

The study shows that the use of cold plastic deformation after solution of the following aging promotes the precipitation process, as well as contribute significantly to the value of the hardness and electrical conductivity. In this variant, technology in many ways overlapping processes of precipitation and recrystallization.

The process of precipitation and dispersion strengthening to a much lesser extent, decreases the electrical conductivity of copper. The reason is that the value of the mean free path in the presence of these precipitates is higher in comparison to the distance between the dissolved atoms.

Nucleation of particles of size much less than 1 minute of aging is undoubtedly related to the primary recrystallization process taking place, which occurs in the solid state without changing the composition of input and output phase transformation.

Obtained in this study results may provide a basis for further research on the mechanisms of recrystallization and precipitation in other species of copper alloy and copper alloys as well as the basis for searching the optimal bands of their functional properties.

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