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STUDY ON SOME COPPER - MOLYBDENUM BASED COMPOSITE MATERIALS CONDENSED FROM THE VAPOUR PHASE

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ABSTRACT

Design features of equipment are given for realizing high-speed process of electron beam evaporation and condensation. Peculiarities of phase composition and structure of a crystallized pool (melt) on copper base are studied. Assumption was made about interrelation of structure-phase state of the melt and rate of evaporation-condensation. Specifics of surface structure and volume of condensate in different sections (before and after etching) are investigated. Technological defects, their influence on strength and ductility of the condensate at tensile test are considered. Interrelation of temperature dependence of energy of deformation activation, specifics of condensate fracture and technological defects of structure, different by origin, are studied.

KEYWORDS: installation for vacuum condensation, technology of electron beam evaporation, condensate, structure, properties.

1. Introduction

This paper presents the basis for obtaining the Cu-Mo based –alloy using electron technology and the study of structure and characteristics.

In order to obtain this alloy, condensed from the vapour phase, the UE-189 electron beam industrial installation has been used, which was modernized by „Gekont”. The image of this installation is presented in figure 1. It has a technological chamber *1* and another chamber, *2,* laterally, with the electron guns *3-6*.

The vacuum system is designed for creating dynamic vacuum in chambers of the UE-189 installation for the purpose of ensuring the process of evaporation and condensation of initial materials. The technological chamber and the gun chamber have separate vacuum pump-out systems, which ensure stability of EB heater operation.

The vacuum system consists of two roughing-down pumps, two booster pumps, and two high-vacuum units (one per the gun chamber and the technological chamber). On upper flange of the technological chamber *1* substrate *14* rotation mechanism *15,* designed and manufactured in «Gekont», it is installed [1].

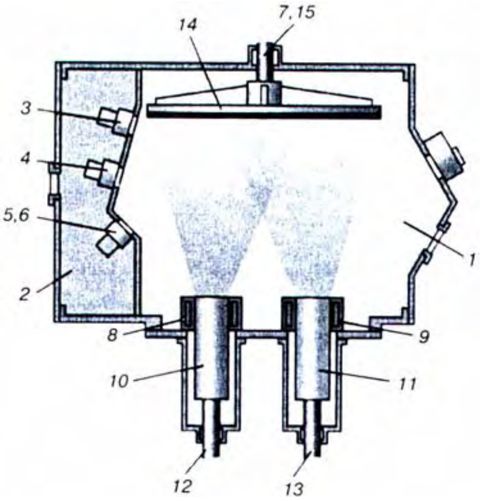

***Figure 1**. Principal scheme of EB installation: 1 — working chamber; 2 — gun chamber; 3-6 — EB guns; 7 — substrate rotation rod; 8, 9 — crucibles for evaporation of copper and molybdenum, respectively; 10, 11 — ingots for evaporation; 12, 13 — mechanisms for feeding ingots into evaporation zone; 14 — substrate; 15 — substrate rotation mechanism.*

A peculiar feature of the design of this mechanism is the possibility of its long operation (10-12 months) without violation of vacuum when rotating a substrate of up to 50 kg mass at speed up to 40 rpm at the temperature (600 ± 50) °C. Heating of the substrate (fixed on the rotation rod 7) up to the mentioned temperature [2] is performed by EB guns *5, 6* of 40 kW power. Heating of the initial material for evaporation is performed by means of EB guns *3*, *4* of 100 kW power. All guns have independent filament of cathodes and autonomous control of EB. The evaporation unit consists of two crucibles of 100 and 70 mm in diameter *(8* and *9*, respectively), designed for evaporation of copper *10* and molybdenum *11*, and mechanisms *12* and *13*, which ensure their feeding into the evaporation zone.

Initial (being evaporated) materials. For producing of condensed electric contact materials the copper, molybdenum, and alloying elements (yttrium and zirconium) were used as initial elements. Calcium fluoride was used for creation of a separating layer on the substrate. Substrates for deposition of 800-1000 mm in diameter and (20 ± 2) mm thick were manufactured from steel OL-37. Surface of the substrate, on which deposition was performed, was subjected to milling and grinding till roughness not less than $R_{(l}$ = 0.63 (GOST 2769-73).

Preparation of ingots for evaporation. Copper ingots were turned and drilled for placement of alloying element weights. These ingots and turned ingots of molybdenum were subjected to cleaning, de-greasing, and weighing. Alloying elements were used in the form of degreased and dried chips of zirconium and yttrium. Weights of mentioned metals at the ratio 7:3 in amount (100 ± 3) g per 10 kg of copper were placed into special channels, drilled in copper ingot being evaporated, and on the surface. For creation of a separating layer a weight of fluoric calcium (2.0 ± 0.3 g) was placed on the surface heated of the molybdenum ingot.

After charging of the substrate and initial materials the installation was vacuumized**.** When vacuum 1-10 Pa was achieved, EB guns were switched on and technological process of vacuum deposition was started.

Deposition of the separating layer CaF2 was performed in vacuum not less than 1-10 Pa after heating of the rotating substrate up to the temperature (873 ± 50) K. Duration of the separating layer application was 120-180 s.

After termination of the separating layer deposition process, alloyed pool was formed on the copper ingot face. At the same time molybdenum ingot was heated. The conditions were considered achieved when evaporation currents of copper equaled 3.0-3.4 A, molybdenum 1.2-16 A at acceleration voltage of 20 kV. Change of the beam power in evaporation of molybdenum in different experiments allowed producing composite materials Cu-Mo-Zr-Y of three grades with molybdenum concentration equal to 2.5-5.0, 5.1-8.0, 8.0-12.0 wt.%, respectively [3].

Evaporator, rate of evaporation, composition, structure. The selecting of the methods for variation of the copper evaporation intensity is based on known data. Most frequently, the evaporation rate is increased by increasing size of the evaporator and power of EB [4-6]. However, when 50 kW power and specific rate of copper evaporation from the water-cooled crucible $4 \cdot 10^3$ g/(cm$^2 \cdot$s) is exceeded, stability of the pool is violated: splattering of metal from the crucible takes place. Outburst of the metal may be partially avoided by introduction of alloying additives and arrangement of a multi-component pool on surface of the melt being evaporated (an intermediate pool) [8, 9]. In [9] the intermediate pool is called melt of the additive-metal, which restrictedly interacts with the base metal, occupies ≈ 10 % of the whole melt volume, and preserves on its surface. The main requirement to the additive-metal is lower density than in the metal-base, and vapors pressure within technological range of temperatures should be lower than that of the metal-base. From chosen by us alloying elements, zirconium completely and yttrium partially met these requirements. Density values of zirconium − $6.49 \cdot 10^{-3}$ and yttrium − $4.55 \cdot 10^{-3}$ are lower than those of copper − $8.92 \cdot 10^{-3}$ kg/m^3. As far as vapors pressure is concerned, for copper and zirconium required difference is preserved, while for copper and yttrium it is not so significant (Figure 2) [10]. However, according to [9], zirconium and yttrium increase specific rate of copper evaporation. From analysis of Figure 3, presented in [9], one can see that alloying elements in copper melt accelerate evaporation of copper: zirconium − 2 times, vanadium − almost 6 times. There is no satisfactory explanation of evaporation rate increase in case of using an intermediate pool. It is assumed that increase of evaporation rate may be caused by surface tension and heat diffusivity of the melt being evaporated, which change during alloying [11].

However, peculiarities of phase equilibriums in binary systems Cu-Zr, Cu-Y, Cu-Nb, Cu-Fe and Cu-V may influence specific rate of the melt evaporation. Four intermetallic compounds (YCu_6, YCu_4, YCu_2, YCu) were detected in binary system Cu-Y, three of which melt congruently within temperature range of (935 ± 15)–(985 ± 15) °C. In the system Cu-Y also four eutectic transformations were registered, which proceeded within temperature range (890 ± 10)–(760 ± 15) °C and yttrium concentrations 12.5 **-** 74 wt.% [12]. There are six intermetallic compounds in binary system Cu-Zr, four of which melt congruently. All compounds participate in eutectic reactions within concentration range of zirconium content 8.85-55 wt.% and temperature range 971-885 °C [12].

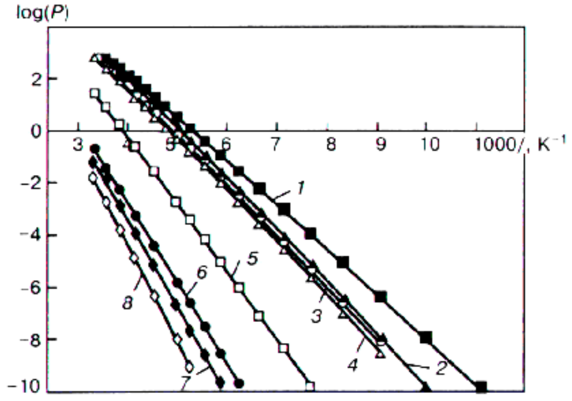

Figure 2. *Dependence of vapor pressure of some metals on inverse temperature [10]:*
1 – molybdenum; 2 – zirconium;
3 — niobium; 4 — vanadium; 5 — yttrium;
6 — iron; 7 - copper; 8 — chromium.

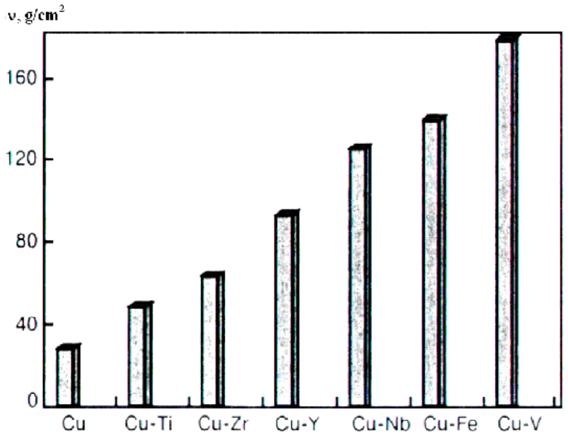

Figure 3. *Change of specific copper evaporation rate v during alloying depending upon melt composition*

So, the interaction of elements in each mentioned binary system exists within wide range of concentrations probability of formation of eutectic type melts with significantly lower melting point than that of initial metals. For binary systems Cu-V, Cu-Nb and Cu-Fe presence of areas of immiscibility and lamination (with the maximum, approximately, in equimolar area) is characteristic, and monotectic reaction, for example, in system Cu-V at temperature 1530 °C, and in system Cu-Nb at temperature 1550 °C [12].

This allows assuming possibility of morphology change of structural components of the system and properties of its capillarity and increase of evaporation rate at the temperature, which significantly exceeds copper melting temperature, which does not contradict data of [9] that prove increase of copper melt evaporation rate in presence of vanadium, niobium and iron additives.

In the process of development of technology for manufacturing massive Cu-Mo-Zr-Y condensed materials, samples of three alloyed pools were investigated, which ensured three rates of condensation that made up 16-20, 8-10, 4-5 μm/min, respectively. For investigation of pool materials optic and electron scanning microscopy, X-ray spectral microanalysis, and method of micro hardness were used.

Despite different values of density of alloying elements of yttrium and base metal (copper), lamination of components in the pool was not detected. Active interaction of these elements with copper, each other, and interstitial impurities was noted.

Phase composition of the pools, after solidification, corresponded mainly to binary diagram Cu-Zr, according to which in equilibrium with copper-base solid solution exists intermetallide $ZrCu_5$, which forms due to peritectic reaction, and intermetallide $ZrCu_4$, which represents a congruently melting compound.

Different mass shares of zirconium in these phases (in $ZrCu_5$ approximately 22 %, in $ZrCu_4$ – 26 %) and morphology of the latter (Figure 4) allow assuming that crystals of intermetallide $ZrCu_4$ of various shape form during solidification of the pool.

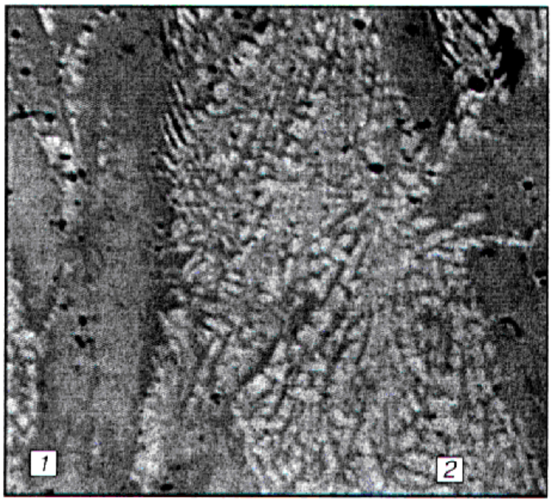

Figure 4. *Morphology peculiarities of phases $ZrCu_4$ (1) and $ZrCu_5$ (2), × 200.*

Intermetallide $ZrCu_5$ in the form of thin fibers precipitates in solid solution at the temperature below solidus, i.e. as a result of aging.

In solidified pool 1, which ensures maximum rate of copper evaporation, big anisotropic particles prevailed (Figure 5, *a)* with sizes sometimes equal to the pool depth (approximately 12 mm). It proves the fact that alloyed melt occupies the whole volume of the pool.

In pool 2, where average rate of copper evaporation was 8-10 μm/min, mainly anisotropic particles of intermetallide $ZrCu_4$ were registered having a shape of somewhat elongated hexahedron (Figure 5, *b).*

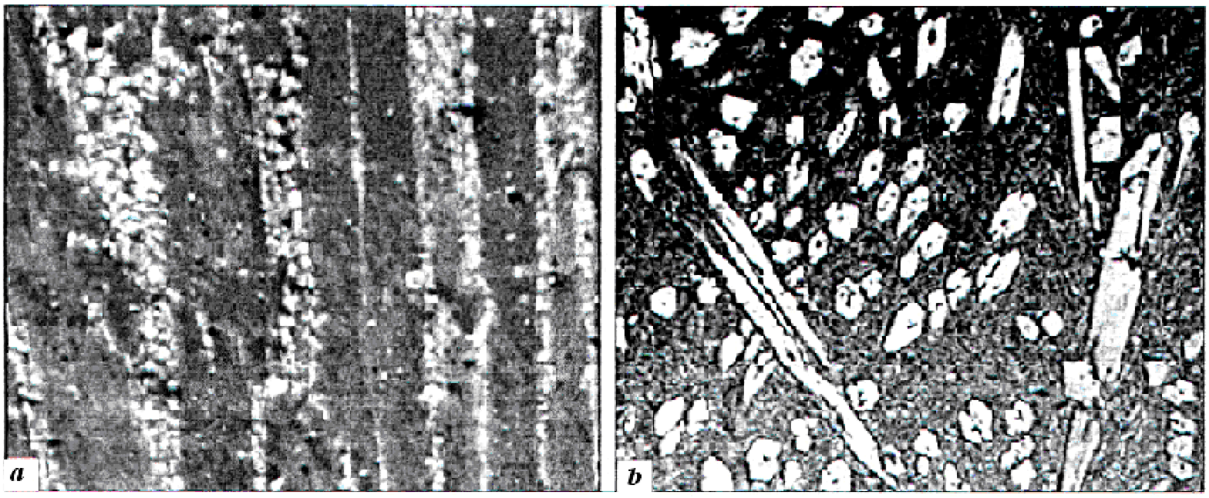

Figure 5. *Melt structure of pools 1 (a – ×100) and 2 (b– ×50) after cooling.*

In pool 3, for which minimum rate of copper evaporation is characteristic (4 – 5 μm/min), size of isotropic particles in dominant areas sharply reduced (Figure 6, *a).*

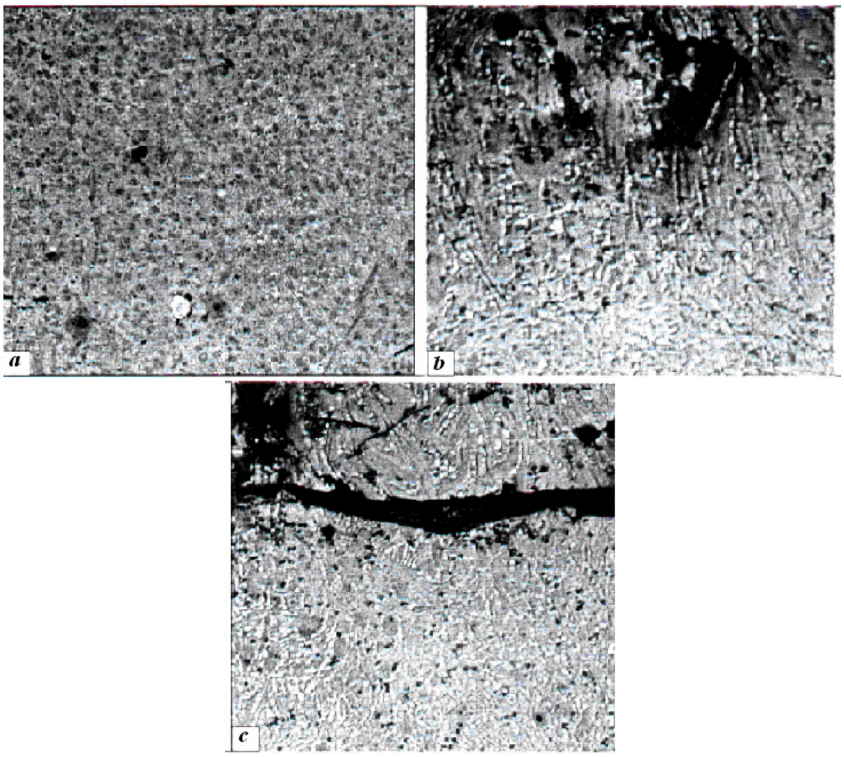

Figure 6. *Pool 3 (a), structure in homogeneity (b) and slag inclusions (c) near its surface (× 200).*

Structure of the pool was inhomogeneous. Near surface of the pool accumulation of anisotropic particles $ZrCu_4$ was detected, as well as porosity (Figure 6, *b)* and slag inclusions in system Zr-Y-O (Figure 6, *c*). Disperse (anisotropic or isotropic) phase $ZrCu_5$ (Figure 4) was present in investigated pools between particles of intermetallide $ZrCu_4$. In the near-bottom zone this very phase formed branched structure (Figure 7).

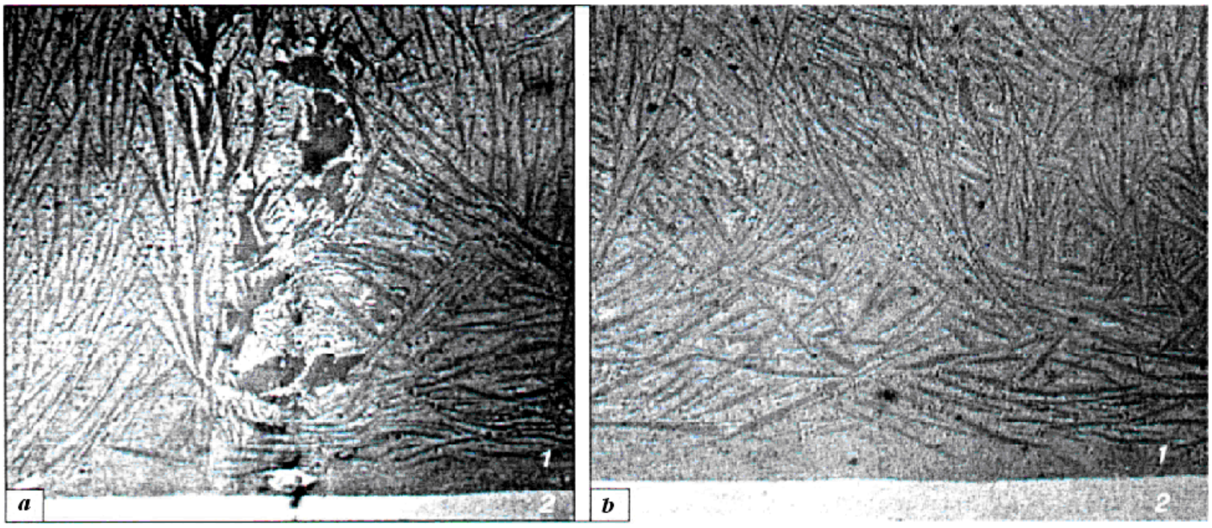

Figure 7. *Structure of solidified melt (1) near boundary with copper ingot (2) irrespective of pool structure (×100): a – high; b – medium rate of evaporation*

So, it was experimentally established that content and distribution of elements in the alloy Cu-Zr-Y with high rate of evaporation during solidification ensure formation of anisotropic structure, which is stipulated by directed growth of intermetallide $ZrCu_5$. Absence of sufficient knowledge about mechanism of melting and solidification does not allow authentic judging of the nature of detected interrelation. However, data of [13, 14] prove that the melt has to be considered as a crystal dispersed into clusters of 0.5-3.0 nm size. That's why one may assume existence of hereditary connection between structures of copper-base alloys in liquid and solid state and influence of ordering on the melt evaporation rate.

On the basis of established peculiarities of the structure, phase, and chemical compositions of the pools one may draw conclusion that phase equilibriums in copper-, zirconium- and yttrium-base systems, complicated by presence of impurities in commercially clean materials, have to be considered as some of the main factors which control rate of evaporation-condensation.

Efficiency of alloying elements is, probably, stipulated by their ability to change structure of the melt and its capillary properties. This assumption fits established in [9] two-fold increase of the copper-base melt evaporation rate in presence of zirconium and yttrium, which form intermetallic compounds and low-melting eutectics in correspondence with similar equilibrium diagrams.

Evidently the same analogy of equilibrium diagrams in systems Cu-Fe and Cu-V (presence of areas of immiscibility in liquid state and possibility of monotectic reaction proceeding at reduced temperature of the ingot, which determine structure of the melt) allows achieving even higher (4-6–fold) increase of evaporation rate in the presence of additives of iron and vanadium.

Condensate, its composition, structure and properties. Ratio of values of metal vapors pressure at various temperatures, composition and structure of the pool melt determine peculiarities of the condensate mass transfer and chemical composition.

As follows from presented data and results of mass-spectral analysis of copper condensate, containing alloying elements zirconium and yttrium used during its production, zirconium is mainly accumulated in the melt, while yttrium participates more actively in mass transfer. According to the results of this analysis, weight share of alloying elements varies in zirconium within 0.01-0.08 and in yttrium within 0.02 - 0.012 %.

The parameter, which allows qualitative estimating possibility of increasing intensity of the alloying element mass transfer, may be the temperature, at which pressure of saturated vapor, 133.3 Pa, is achieved [15]. For example, for iron, yttrium and zirconium it increases at 1923, 2128, and 2883 K, respectively. In the same sequence the content of elements in condensates Cu-Fe, Cu-Y, Cu-

Zr (6.08-9.43; 0.13-0.18; 0.02-0.08 wt.%, respectively) reduces [9]. These data don't contradict peculiarities of temperature dependence of vapor pressure of iron, yttrium, and zirconium (see Figure 2) [10].

The alloying elements into condensate, notably affect its mechanical properties (Table 1) [9].

Table 1. *Influence of alloying elements on mechanical properties of copper-base condensate*

Material	Mechanical properties of condensates at T, °C			
	20		300	
	δ_t, MPa	δ, %	δ_t, MPa	δ, %
Cu	188	62	98	33
Cu-Y	213	53	79	34
Cu-Zr	192	56	115	50
Cu-Fe	318	33	118	25

The morphology of additive and its content affect properties of the initial condensate and intensity of its strength at the temperature 573 K. Maximum worsening of properties was registered when copper condensate was alloyed with iron. Tensile strength of the condensate reduces by 63 % and relative elongation δ – by 24 %. The lower reduction of property parameters by 40 and 11 % – takes place, when the condensate is alloyed by zirconium, whereby it should be noted that tensile strength and ductility of pure copper reduce at 573 K by 48%, in comparison with room temperature. Taking into account structural sensitivity of mechanical properties, one may assume that strength reduction is stipulated by the processes of recovery and ability of alloying elements to hinder these processes by means of action on phase composition, morphology, and dispensability of new phases. However, mechanical properties are affected, in addition to mentioned factors, by other factors as well. They are stipulated by application in technological processes of commercially pure initial materials and residual mediums. In this work, the influence of impurities on structure and properties was investigated on samples of composite material Cu-Mo-Zr-Y proceeding from the need of increasing the level, reproducibility of the condensate properties, and optimization of technological conditions of their manufacturing. As test specimens, samples of copper and molybdenum based materials have been taken from different lots, containing up to 12 mass % of Mo, 0,08 mass % of Zr and Y (for each of them) and for the rest, copper [3].

Samples were cut out from condensate sheets of 800 mm in diameter. Size of samples allowed sequential performing macro structural analysis of the surface, measuring thickness, density, electric resistance, hardness, and determining tensile mechanical properties. Surface and sections of the samples parallel and perpendicular to the vapor flow (before and after etching), and fractures were subjected to micro structural investigations [16]. Thickness measurement of the samples showed that condensate, formed during rotation of the substrate, may be represented in the form of a truncated toroid. Cuneiform shape is characteristic of the periphery. Here the thickness, in comparison with maximum for the toroid, reduces by (45 ± 3) %. Ion etching of section, perpendicular to the surface, allowed establishing that disperse-strengthened copper consists, as a gradient material, from micro-layers of different composition and morphology (Figure 8). For the layers, enriched with molybdenum, anisotropic (columnar) structure is characteristic (Figure 8, *a)*, which, according to [17], forms as a result of connection of atoms from a volumetric diffusion field (VDF) of the condensed flow with 2D insular layers by diffusion coalescence. Different thickness of the layers occurs in this case as a result of formation of more efficient VDF and increase of rate and time of condensation of the fed vapors. For layers enriched with copper mainly isotropic structure is characteristic, which consists either of disoriented polygonal grains (Figure 8, *b*), or from particles of spherical and (or) lenticular form dispersed in the matrix (Figures 8, *c* and 9, *a)*.

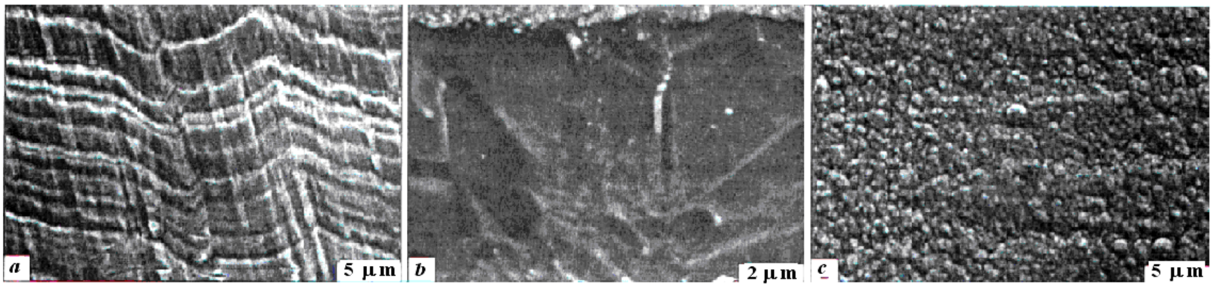

Figure 8. *Condensate layers with columnar (a), polygonal (b) and spherical (c) shape of particles.*

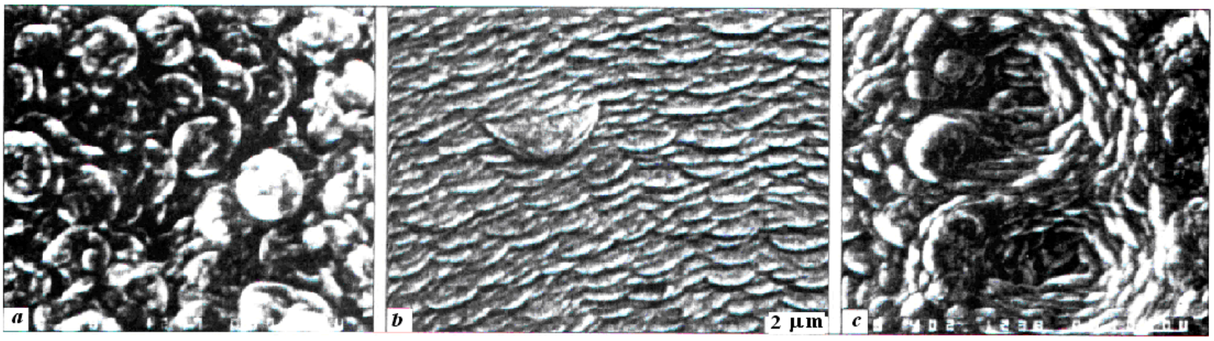

Figure 9. *Mixed (a) and lenticular (b) structures of condensate and* ***conglomerate*** *from these particles (c) of this shape*

Such analysis of sections of these condensate samples allows assuming that correct spherical shape of particles and respective morphology of layers are connected with aggregated transformation in copper in the direction vapor → liquid phase.

Lenticular shape of particles forms, evidently, as a result of coalescence of solid-liquid clusters of composite material Cu-Mo and origination of spherical particles during their approach to the substrate and deformation of these particles during their collision with the substrate and under action of the next portions of the «drop» vapor. This is proved by the shape of particles in Figure 9, *a* and *b*.

Turbulence of vapor flow may be the reason of formation of different forms of conglomerates during consolidation of particles in micro layers (Figure 9, *c*). Chemical etching of the section perpendicular to the condensate surface proves that in case of lower content of molybdenum, refractory component forms separated grains with $d << 1$ μm and conglomerates of these grains in the copper-base matrix (Figure 10). When content of molybdenum increases, chains of grains and anisotropic colonies form in the matrix.

Change of structure and chemical composition of micro layers fits peculiarities of the micro hardness change. Micro hardness increases together with increase of the share of columnar structure and molybdenum content in the layers. In the process of mechanical tensile tests of condensed composite material dependence of sample properties upon molybdenum content was also detected (Table 2).

In case of annealing of the samples (1173 K, 3h, vacuum), their strength reduces, ductility increases, but mean value of dispersion does not reduce. Ductility of samples, which correspond to periphery of the sheet, reduces because of lamination first of all along boundaries of macro layers enriched with carbon and other impurities.

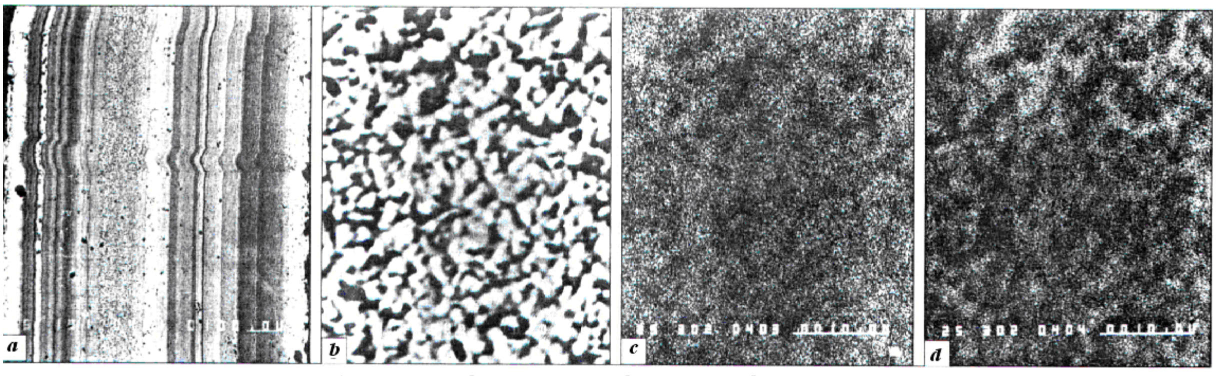

Figure 10. *The grains in the copper-base matrix.*

Table 2. *Influence of annealing on mechanical properties of condensates*

Composite materials with Mo concentration, wt. %	Initial state of composite material		Annealing of composite material at 900 °C for 3 h	
	δ_t, MPa	δ, %	δ_t, MPa	δ, %
2,5 … 5,0 8,0 … 12,0	518 ± 118 659 ± 106	0,69 ± 0,53 0	543,0 ± 60,9 629,3 ± 61,3	2,4 ± 1,1 0,79 ± 0,57

The mechanical characteristics of the condensed composite material with 8,0... 12,0 Mo content have been studied function of temperature.

Processing of the test results according to methodology of [18], which was carried out by us with determination of gross errors (blunders) in small samplings, allowed establishing step-wise character of strain activation energy change (Figure 11).

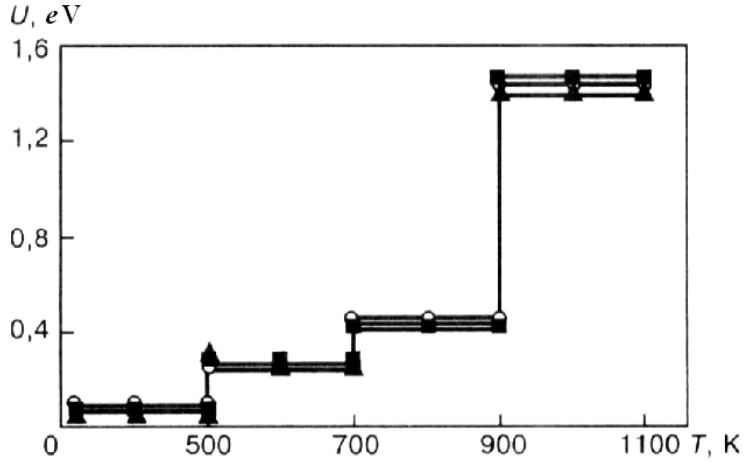

***Figure 11**. Temperature dependence of strain activation energy of composite material with Mo concentration 8,0 … 12,0 wt. % samples in tensile tests.*

Comparative analysis of these data and peculiarities of fracture of samples of two lots allow assuming that strain activation energy is minimal and makes up 0.061-0.062 eV within temperature range 300-500 K and corresponds to strain localization near the notches on the surface. Maximum strain activation energy 1.467-1.462 eV within temperature range 900-1100 K for the same lots is connected with fracture toughness of the material with isotropic and columnar structure.

The results obtained don't contradict assumption made in [18] about change of strain mechanisms (dislocation slippage for dislocation creep age) during tensile tests of copper within the same temperature range. Data of fractographic studies expand our idea about peculiarities and role of structure defects of condensed composite materials as places of localization of plastic strain, origination and development of cracks, and failure.

Conclusions

Main technological factors (combination of conditions – preparation of a substrate, its rotation, alloying of the pool, conditions of the pool heating, etc.) which affect process of evaporation and structure formation of condensed composite materials are established.

It is shown that in case of copper alloying by the elements, which may affect rate of the melt evaporation, peculiarities of phase transformations in binary and more complex systems and possibility of morphology control of structural components and capillarity of a system in liquid-solid state have to be taken into account.

It is established that condensate surface at the condensation front is of block character with periodic and arbitrary striation according to roughness of the substrate. Behind condensation front lamellar structure with hierarchy of layers was detected, which had columnar, polygonal disoriented, composite (with spherical, lenticular or mixed shape) structure, varieties of which corresponded to the condensation mechanisms.

It is shown that in tensile tests of copper- and molybdenum-base condensates, which contained defects of structure, reduction and instability of mechanical properties were registered stipulated by combined or separate influence of defects on peculiarities of deformation and failure. In case of the tensile test temperature increase and change of the nature of structure defects, which determine localization of plastic strain, origination, development of cracks and failure match well stepwise change of strain activation energy.

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