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STRUCTURE-PROPERTY CORRELATION OF COMPOSITE MATERIALS BASED ON Cu-Mo OBTAINED BY PVD METHOD

Lucica ORAC[1], Vera GRECHANYUC[2]
Olga MITOSERIU[1], Stela CONSTANTINESCU[1]
[1]University "Dunarea de Jos" of Galati, Romania
[2]University of Building and Architecture, Kiev, Ukrain
email: lucia_orac@yahoo.com

ABSTRACT

The paper describes reviews referring to determination of working parameters of the development processes of composites in copper matrix, by vapour-phase deposition method. The molybdenum was used as complementary phase. Structural characterization of coatings developed by optical microscopy and electronic scanning and effects of surface structure on their characteristics were presented. The specificity of the technology for obtaining Cu-Mo composites is a condition to formation a special laminated structure. The mechanical characteristics of the condensate depending on the amount of molibden. An important role in forming the structure and characteristics is played by the defects which were formed in the condensate during the transfer in the liquid phase as drops. Electrochemical tests have shown that the corrosion resistance of the composites obtained by PVD method is higher than pure copper.

KEYWORDS: PVD, coatings, defects, electrochemical corrosion

1. Introduction

One of the main causes of the limitation to the use of materials for electrical contacts free of Ag or other nobel matals, is the fast corrosive damage when operating in wet environment.

Cu-Mo (12% max. Mo) composite materials are produced by simultaneous evaporation from separate Cu and Mo crucibles with subsequent condensation of the vapor flow on OL-37 steel layer of 15 to 20mm thickness and 800 mm diameter. The surface of the disk-support on which condensation of the vapor flow takes place was machined until a roughness of R_a=0.63 was obtained.

The analysis of chemical composition and structure of composites based on copper and molybdenum content allowed to determine the variation of these elements from layer to layer (of up to 20-25% to 4-5 mas.) and the distribution gredient of these elements in the layers.

The mechanical properties of the Cu-Mo composite materials, depend on molibden content.

The condensate obtained as plates of 0.7-1.2mm thickness has been used for the study of corrosion resistance [1].

2. Results and experimental research

To obtain composite materials based on copper and molybdenum, copper, molybdenum and calcium fluoride powder have been used; their characteristics are presented in Table 1. For evaporation, use is made of copper ingots of 100mm diameter and 70mm diameter molybdenum. The ingots were blanked to 98.5, respectively 68.5mm size to avoid their jam in the evaporation process.

Table1. *Material marks that were used to obtain composite materials based on copper and molybdenum*

Materials	Marck	Standard
Cu	M0b, M00, M0, M1, M2	GOST 859-78
Mo	MCVP	TU 48-19-247-87
CaF_2	c	GOST 7167-77

Calcium fluoride powder was pressed with a press-type P-457 at a pressure of 240 to 300MPa, in a number of pills of 30 mm diameter and 15 to 20mm thickness. The CaF_2 pills were used to form a separate layer of the disc-holder, where vapor flow condensation takes place. Due to the separation layer the condense separates easily from the holder/support.

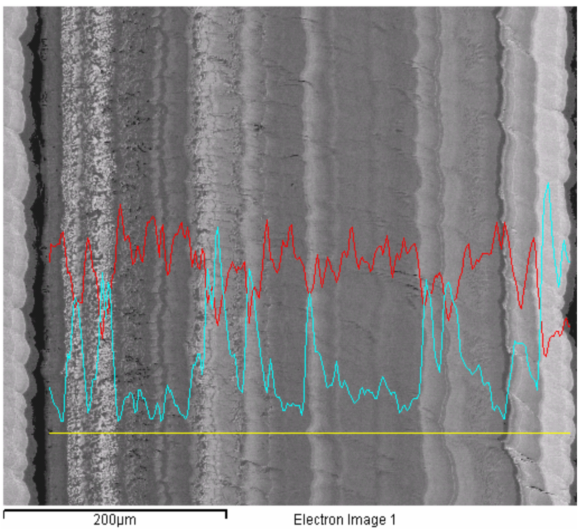
The chemical composition of the condensates was determined by chemico-analytical methods [2]. The chemical composition was determined at the Electrotechnical Research Institute in Bucharest, according to STAS STAS 1706/1-85 and 1706/17-71 using spectrometric methods.

The results, which re the arithmetic average of tens of tests on samples with different thicknesses, are presented in Table 2.

Table 2. *Chemical composition of the composites*

Sample code	Chemical composition, %	
	Cu	**Mo**
1	98.8	1.2
2	96.09	3.91
3	94.9	5.1
4	93.2	6.8
5	91.6	8.4
6	87.7	12.3

By electronic microscopy in cross –section and from the spot analyses EDX it could be noticed the stratified distribution of both molybden and copper (Figure 1).



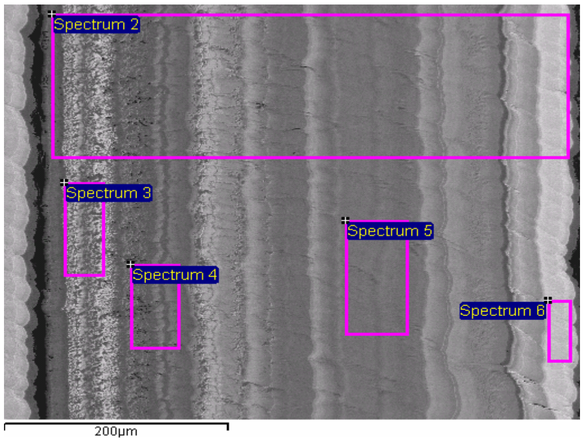


Fig. 1. *Analyses EDX in cross section and spots for the stratified composite, Cu - Mo (8.4% Mo)*

Processing option: All elements analysed (Normalised)

All results in weight %

Spectrum	Cu	Mo	Total
Spectrum 2	91.85	8.15	100
Spectrum 3	91.80	8.20	100
Spectrum 4	92.02	7.98	100
Spectrum 5	92.14	7.86	100
Spectrum 6	91.6	8.4	100

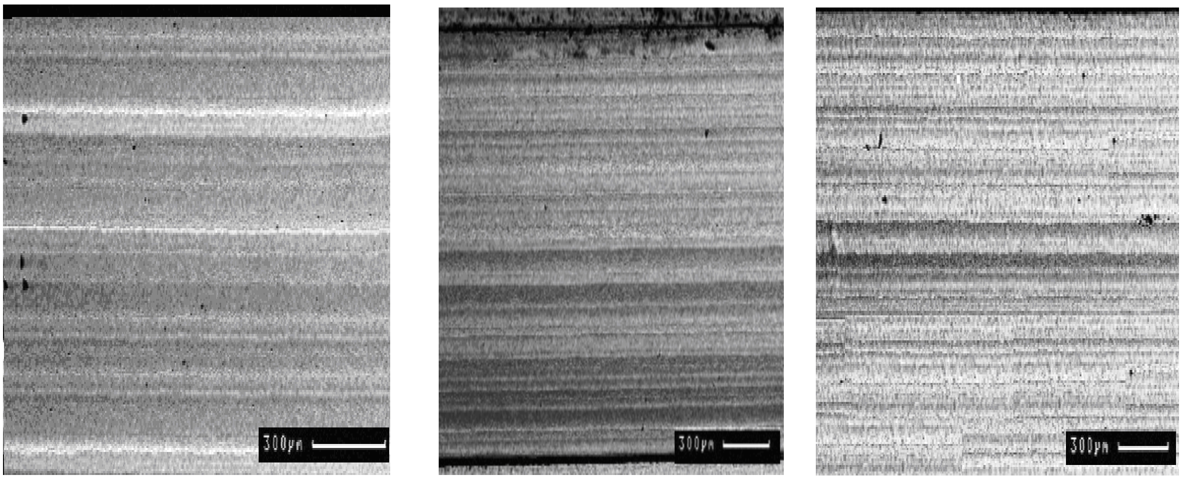
The structure of Cu-Mo composites is in the form of blocks. For each block it is characteristic an arbitrary periodic striped structure. The period of stripes repetition is 150 ±3µm.

Composites were obtained in Cu-Mo micro layers with a structure sufficiently balanced by condensation at temperatures above the melting temperature to 0.3 than the melting temperature of the lowest fusible component (°C).

The mechanical characteristics were determined from the tensile test results of flat samples in vacuum with 15 mm length on a 1248-R plant as per Standard of Ukraine 9651-84.

Samples were cut from the composite with thickness of 0.9 - 1.4mm in the initial state and after annealing in vacuum to 1170K for 3h.

The experiments were done at room temperature in open air and vacuum, of max. 0.1Pa at a temperature of 370-1070K from 100 to 100degrees. At each temperature three to six samples were studied. The deformation rate of the sample during the experiment was 2 mm/min, which corresponds to the relative speed of deformation ~ $2.2 \cdot 10^{-3}\ s^{-1}$ [3].



a b c

Fig. 2. Microstructures a) Cu -5.1% Mo, b) Cu -8.4% Mo, c) Cu -12.3% Mo magnification x 200

***Table 3.** The mechanical characteristics of the condensate depending on the amount of molibden*

Composition	Mechanical characteristics					
	Before annealing			After annealing		
	R_{p02}	R_m	δ	R_{p02}	R_m	δ
	[MPa]		[%]	[MPa]		[%]
Cu-2.5-5.0Mo	210-370	300-430	10.3-7.3	200-360	295-420	17.6 – 9.5
Cu-5.1-8.0Mo	380-530	440-630	7.25-3.4	365-510	425-600	9.45-4.9
Cu-8.1-12Mo	550-750	635-785	3.25-1.8	520-695	605-730	4.85-3.9

From the charts made, the conventional yielding limit $R_{p0,2}$, resistance limit R_m, relative elongation δ were calculated.

The mechanical characteristics of Cu-Mo composites as to the content of molybdenum are shown in Table 3.

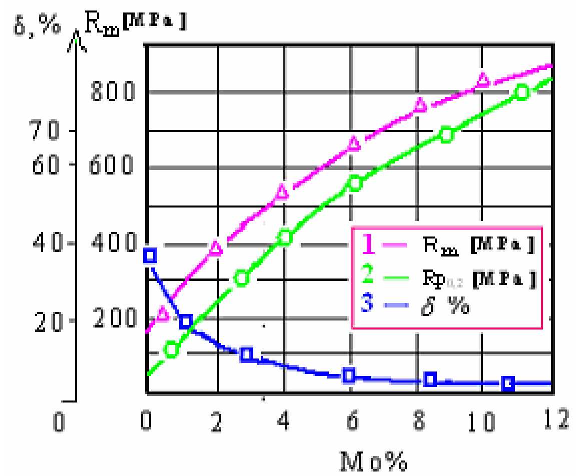


***Fig. 3.** Variation of the mechanical strength R_m, yielding point and relative elongation depending on the concentration of Mo*

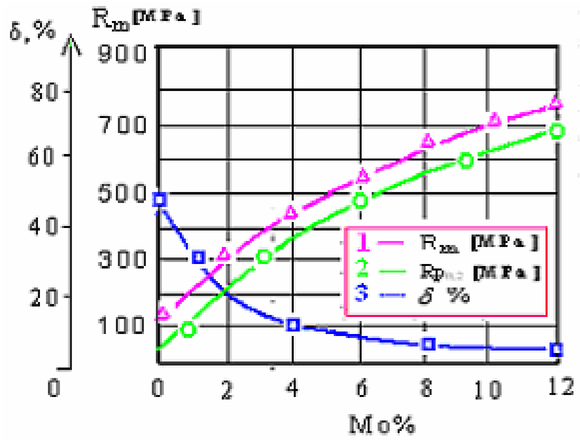


***Fig. 4.** Variation of the mechanical strength R_m, yielding point and relative elongation depending on the concentration of Mo. (after vacuum annealing at 1170K)*

The results of tensile vacuum elongation checks showed that with increasing molybdenum content values $R_m, R_{p0,2}$ increased and δ decreased almost to „zero” values at Mo concentration ~ 14%.

The variation of the mechanical strength, yielding point and relative elongation depending on the concentration of Mo is given in Figure 3.

Similar values of the mechanical characteristics of these materials were obtained after vacuum annealing of the samples at a temperature of 1170K for three hours (Figure 4).

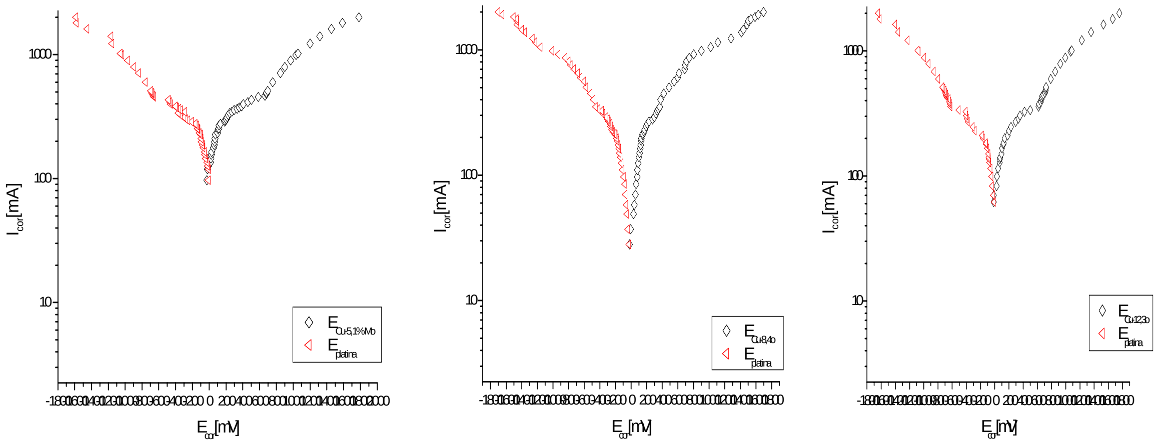
It is noted some decrease in strength characteristics by 8 - 10% and increased plasticity by 10 - 25% with increasing mean square deviation (3.3-5%) of the values of these parameters compared to the initial condition of the materials [4]. An important role in forming the structure and characteristics is played by the defects which were formed in the condensate during the transfer in the liquid phase as drops. For the most part these droplets grow, forming irregular spheres. These spheres, being in the previous layer, cause in the vapor flow disruption of crystallization of the next layers. The dislevel which is formed by such particles, is passed to each micro and macro-layer until the final surface of the composite (Figure 5), subject to the influence of throwing and melting condensation.

Fig. 5. *The structure of the sample surface without cutting*

Metallographic analysis of structural defects, subject to the transfer of the liquid phase allows us to conclude that there is a possibility of disturbance of the crystallization and the occurrence of such defects in the composite structure.

Electrochemical tests have shown that the corrosion resistance of the composites obtained by PVD method is higher than pure copper.

Figure 6 presents the Taefel curves for Cu-Mo composites produced by PVD method.



a) b) c)

Fig. 6*. Taefel curves for Cu-Mo composites produced by PVD method:*
a) Cu-5.1% Mo, b) Cu-8.4% Mo, c) Cu-12.3% Mo

In Figure 6 it is observed that the current density of corrosion of the blank sample is higher than the composite samples, leading to the conclusion of increased resistance to corrosion of the composite samples, the visual appearance of the sample surfaces being clean without oxide stains [5].

Table 4. *Behaviour of Cu-Mo composites produced by PVD method, when tesed for corrosion by galvanostatic method*

Cu	Mo	Current density j_{cor}	Corrosion rate v_{cor}	Penetration index p	Resistance group
[%]		$[A/m^2]$	$[g/m^2 \cdot h]$	[mm/an]	
94.9	5.1	17.7	0.0056	0.0054	highly resistant
91.6	8.4	15.6	0.0049	0.0048	highly resistant
81.5	12.3	14.2	0.0042	0.0041	highly resistant

The values of current density, corrosion rate and penetration indices when tested for corrosion by the galvanostatic method are given in table 4.

Corrosion resistance of composites obtained by the two methods - PVD, electrochemical - is better than that of pure copper.

3. Conclusions

-The composites obtained by physical vapor deposition increase, forming chains and conglomerates actually very important because they increase the resistance to high temperatures.

-The structure of Cu-Mo composites is in the form of blocks. For each block it is characteristic an arbitrary striped structure. The period of stripes repetition is 150 ± 3mm.

-The results of the vacum elongation tests shown that with higher amounts of molibden the values of $R_m, R_{p0,2}$, increase, while δ decreases down to „zero" at a concentration of Mo ~ 12%.

-An important role in forming the structure and characteristics is played by the defects which were formed in the condensate during the transfer in the liquid phase as drops.

-Electrochemical tests have showed that the corrosion resistance of the composites obtained by PVD method is higher than pure copper.

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