

# Study of the Formation of the Alloyed Surface Layer During Plasma Heating of Mixtures of Cu-Sn/CrXCY Alloys

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Cu-Sn  
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Plasma heating  
Tin-bronze  
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Microhardness  
Alloyed coating

## ABSTRACT

*In this work, to assess the possibility of obtaining an alloyed layer by plasma heating of the mixture of the PRV-BrO10 alloy and the coating of the welding electrode OK 84.78. The study of the structure is given using the optical microscope MET-2 and the measurement of microhardness - hardness testers of the HMV-G series. It is established that with a thin layer of pre-coating, the coatings are completely saturated, and with a thick layer - the formed zones are different in microstructure and microhardness. In the case of 0.25 mm pre-coating thickness, the addition of chromium carbide leads the strong increasing of the hardness compared to tin-bronze alone. In the case of 0.50 mm pre-coating thickness, for the upper soft zone rich in copper, the average microhardness value is below 250 HV, and for hard particles in this zone in the range of 500-700 HV, the lower saturated zones have high hardness in a wide range (400- 700 HV). The slow scanning speed of the torch and a thick layer of pre-coating are the reason for incomplete saturation and the appearance of defects on the surface of St3 steel.*

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## 1. INTRODUCTION

Under operating conditions, the surface of metal parts is influenced by several mechanical factors such as wear, erosion, shock, etc. For reducing the influence of these factors, it is often required to increase the hardness of materials by surface alloying or strengthening metal coatings [1,2]. It is known that types of coatings based on tin-bronze (Cu-Sn) have been used to protect many types of parts and various materials from corrosion in various environments [1,3]. Despite the high corrosion resistance, the coatings of the

Cu-Sn system alloy have a low hardness (below 300 HV) [3,4]; even thin bronze layers from electrodeposition limit their use under conditions of strong mechanical stress. The addition of a solid component can increase the hardness, and at least retain the original properties, but at the same time increase the likelihood of defects (porosity, cracks, heterogeneity). In recent years, highly concentrated heat sources have begun to be used to obtain hardening coatings in various methods, such as thermal spraying, cladding, chemical-heat treatment [1, 2].

This is due to several advantages: the first is the localization of the processing of the surface layer of the metal, the second is to reduce the time and consumption of materials and the third is to replace the technology of electroplating in order to protect it from the environment [5]. To overcome the low hardness of the Cu-Sn alloy coating, additives containing wear-resistant elements such as diamond, SiC, TiN, WC were used [6-8]. The combination of copper (Cu) and chromium (Cr) is found in studies of Cu-Cr coating on copper alloy substrate [9]. The study of the microstructure of the laser coating of carbon steel showed that saturation of the Fe-8.95Cr alloy and pure copper Cu results in several types of zones: particles rich in copper in a layer rich in iron; particles rich in iron in a layer rich in copper; and others [10]. The same paper noted that an increase in the laser scanning speed can increase the capture of dissolved copper in  $\alpha$ -Fe dendrites, improve the microstructure in the Fe-rich layer, and reduce the size of the Cu and Fe-rich spherical particles. Alloying the surface and creating a coating using a paste, coating, or a preliminary layer is much more detailed in traditional chemical thermal treatment (CHT) [11,12]. In modern research, the type of coating, paste is still used for carburizing [12], chromium-nickel plating [13], Fe-TiB<sub>2</sub> [14] with plasma heating by a non-consumable electrode system in an inert gas (TIG). It is known that chromium carbides (Cr<sub>x</sub>C<sub>y</sub>) are repeatedly added to matrix compositions to improve the hardened property [15-17]. The combination of the hard Cr<sub>x</sub>C<sub>y</sub> component in the tin-bronze alloy during plasma heating to high temperatures can increase the solubility of chromium in the soft Cu matrix, thereby improving the properties of the resulting coating as compared to a pure Cu-Sn coating.

The aim of this work is to assess the possibility of obtaining a wear-resistant composite surface layer by plasma heating of various thicknesses of the pre-coating of Cu-Sn + 20% Cr<sub>x</sub>C<sub>y</sub> mixtures. The results are presented by the appearance and study of the microhardness of the cross section of the obtained coatings.

## 2. EXPERIMENT

### 2.1 Equipment for surface carburizing

As a surface coating, we used a mixture of powder alloys PRV-BrO10, a Cu-Sn mixture (manufactured by JSC Polema, Russia) and a coating of the welding

electrode OK 84.78 (ESA, Sweden). Welding surfacing electrodes OK 84.78 (ESAB) according to the chemical composition of the coating contain: 4.5 C%, 1.2 Mn%, 1.2 Si, 33 Cr%. For preparing the powder from the coating of the OK 78.84 electrode, pliers were used when removing the coating, then it was rolled between two rolls until grinding - this is the first fraction. Then one part of the first fraction and additionally ground it in a porcelain cup using a pestle - the second fraction was obtained. The particle size distribution of the investigated powder alloys was determined by an ANALYSETTE 22 laser analyzer. The resulting powders were mixed with a binder (using stationery glue) to form a paste, which was then manually applied to the samples, and dried at a temperature of 373 K. Steel samples were rectangular plates of size 70×25×10 mm. The surface of the samples was ground to a roughness R<sub>a</sub> = 0,2 μm.

The device described in detail in [2,11,12] was used as a source of plasma heating. Plasma heating mode: current strength 140 A; the speed of movement of the samples is 1.9-2.7 mm/s; gas (argon) feed rate 10 l/min, the gap between the coating layer and the electrode 4-6 mm. After cutting, polishing, and etching the obtained samples, their microstructures were studied on a MET-2 metallographic microscope. Before studying the microstructure, grass samples in nitric acid 5%. The microhardness of the coatings was measured using an HMV-G21 microhardness tester; for reflowing, a paste containing a powder tin-bronze alloy PRV-BrO4 (Cu-Sn), a coating of the welding electrode OK 84.78 (ESAB), and silicate stationery glue were used. The particle size is shown in Figure 1. Plasma heating of the prepared samples was carried out with the compositions and modes given in Table 1.

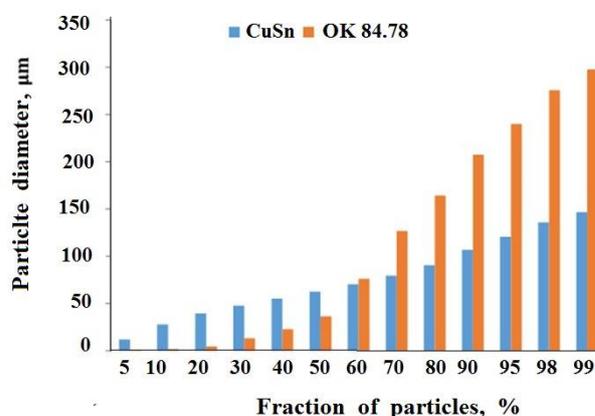
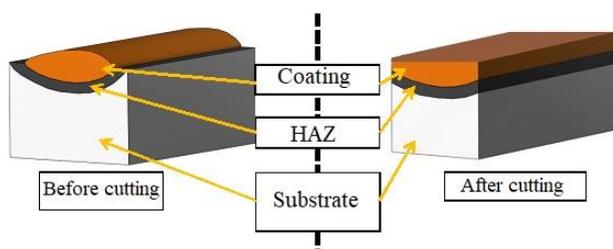


Fig. 1. Granulometric composition of particles of Cu-Sn alloy and OK 84.78.

**Table 1.** Compositions of pastes and plasma heating mode.

Alloy mixture composition	Thickness of the pre-coating; torch scan speed	Plasma treatment parameters
1) CuSn	0,25 mm; 2,7 mm/s	Current: 140 A Plasmatron travel speed: 1.9-2.7 mm/s Gap between tungsten electrode and substrate surface: 4-6 mm. Argon gas feed rate: 10 L/min
2) CuSn+20%OK 84.78	0,25 mm; 2,7 mm/s	
3) OK 84.78	0,25 mm; 2,7 mm/s	
4) CuSn	0,50 mm; 1,9 mm/s	
5) CuSn+20% OK 84.78	0,50 mm; 1,9 mm/s	
6) OK 84.78	0,50 mm; 1,9 mm/s	

Tests of the investigated materials under sliding friction conditions were carried out on an SMT-1 friction machine according to the "disk - plane" scheme. The essence of the method lies in the wear of a sample from the material under study by a rotating disk (indenter), followed by determination of the volume of the worn out material. As samples, we used plates made of the materials under study with a size of 20x10x10 mm. Before testing, the samples were processed on a surface grinding machine and polished to a roughness Ra of 0.32. The indenter was a disc made of hardened 45 steel with an outer diameter of 50 mm and a width of 10 mm. The sample was pressed against the indenter with a constant force  $P = 1000$  N. The disk rotation frequency  $n$  was 200 rpm. Mineral oil M8B was used as a lubricant. The wear resistance of the surface layers was evaluated based on the volume of the worn out material, which was calculated from the results of measurements of the geometry of the wear hole using a Brinell magnifier.



**Fig. 2.** Sample preparation for wear test against fixed abrasive.

For tests for abrasive wear on a fixed abrasive [18, 19], the preparation of the test sample is given. After plasma heating and cooling, the samples were cut (Fig. 2) to obtain them in the form of a parallelepiped with dimensions of 12x5x10 mm. The working surface of the coating in contact with

the abrasive was 100%, and the other was filled with acrylic resin in the form of a cylinder for subsequent fixation to the holder of the grinding and polishing machine shown in Figure 3.



**Fig. 3.** Grinding-polishing machine Struers Tegramin-25 for testing wear on fixed abrasive.

The fixed abrasive wear test was carried out on a grinding and polishing machine shown in Fig. 3. Wear resistance was determined for samples of coatings before and after plasma hardening, by rotary movements on the surface of grinding paper based on silicon carbide. The rotation speed of the aluminum plate was 500 rpm. / min, holder speed 150 rpm. / min with reverse rotation, water cooling. Normal load on the sample ranged from 5 to 50N. The wear resistance index of coatings, equal to the ratio of changes in the mass of the standard and the sample, was determined based on the results of three tests. Samples of St3 steel without plasma hardening were taken as a standard.

### 3. RESULTS AND DISCUSSION

Reflow of the Cu-Sn alloy with a pre-coating thickness of 0.25 and 0.50 mm in the mode: power - 140 A, sample movement speed - 2.7 mm/s. The surfaces of the samples obtained were different, shown in Figure 4 (a), (b). It was seen that in the case of the thick pre-coating, the surface is smoother, and for the thickness of 0.25 mm, gas pores appear due to the strong pressure of the plasma arc column.

In the case of adding an additional coating of the welding electrode OK 84.78, which has a large particle size, for the complete saturation of the alloy with the same power of 140 A, the sample movement speed is suggested to be slower - 1.9 mm/s. After processing, the surface of the coating is not smooth; there is a strong convexity

and concavity, shown in Figure 4 (f). Due to the unevenness of the surface, the depth of the central section, concave below 1 mm, is lower than the coating with a thickness of pre-coating 0.25 mm (Fig. 4 (c)). The use of the same speed (1.9 mm/s) for pre-coating of the mixture of Cu-Sn alloy and 20% coating of the welding electrode OK 84.78 with a thickness of 0.50 mm also leads to the appearance of convexity and concavity, like a large wavy surface, but not stronger than in case of coating only OK 84.78. Further, it was be seen that unmelted sintered particles with a size of several hundredths of a micrometer appear at the edge of the coating (Fig. 4 (e)). Of course, in comparison with the coating of the 0.25 mm pre-coating (speed 2.7 mm/s), the surface is melted more evenly (Fig. 4 (b)). Thick layer of pre-coating, i.e. the large volume of the alloy mass strongly prevents heat transfer in all the melting zones, and the slow speed of the plasmatron movement causes the arc column to act on the surface of the molten metal, displacing the latter to the periphery of the melt pool. As a result, the movement of the solid solution is disordered, and even throws out undissolved particles, or deflects the arrangement of the phases in the molten bath.

The study of the cross-section of the Cu-Sn alloy coating samples showed that the pre-coating thickness of 0.25 mm at the current of 140 A, and cooling the sample with water leads to the appearance of cracks in the alloyed layer in Fig. 5 (a). In this case, the large difference in the coefficient of thermal expansion between the soft and hard phases during rapid cooling is the main reason for the formation of defects. With a pre-coating thickness of 0.50 mm, the backing metal is diluted in a large bronze matrix (compared to a thin layer), therefore it is not enough to form a large hard zone, but only small hard and soft zones alternate. Figure 5 (b) shows that the soft zones rich in copper occupy a large cross-sectional area, therefore, several places with low hardness are likely to appear (134, 229, 310 HV). With a pre-coating thickness of 0.25 mm, corresponding to the number in Table 1, coatings No. 2, 3 have no defects. It can be seen that the microhardness values of the cross section of coating No. 2 are in the range 460-670 HV (Fig. 6 (a)), for coating No. 3 - in the range 700-840. HV (Fig. 6 (d, e)). The presence of chromium carbide in the Cu-Sn matrix significantly increases the hardness of the surface alloyed layer.

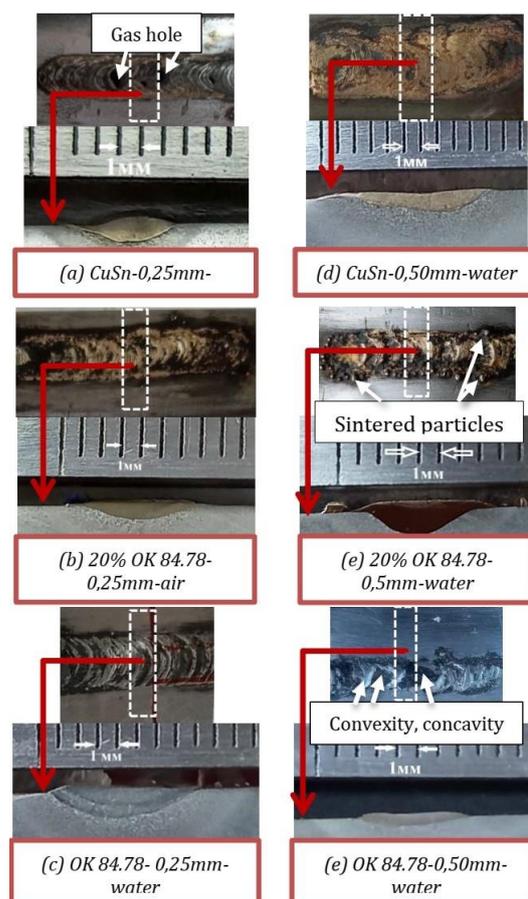
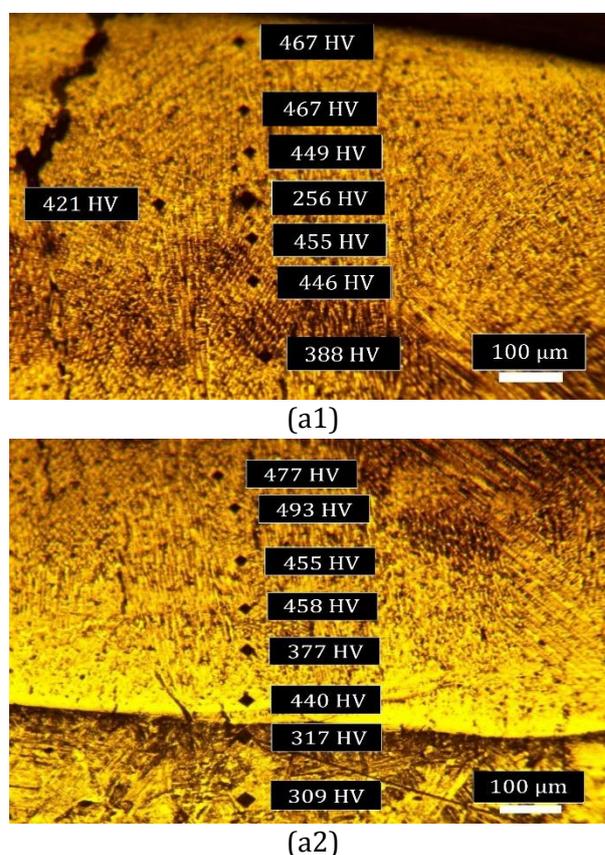
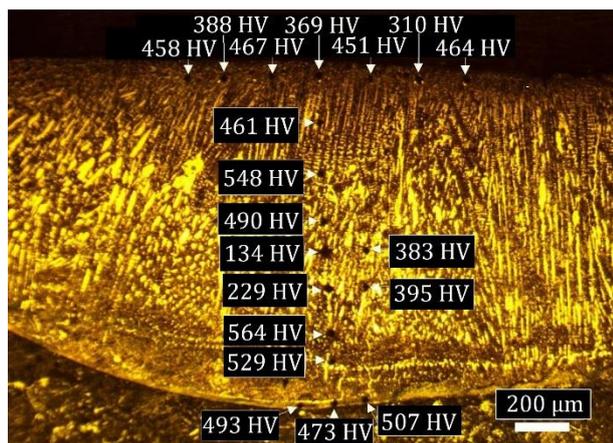


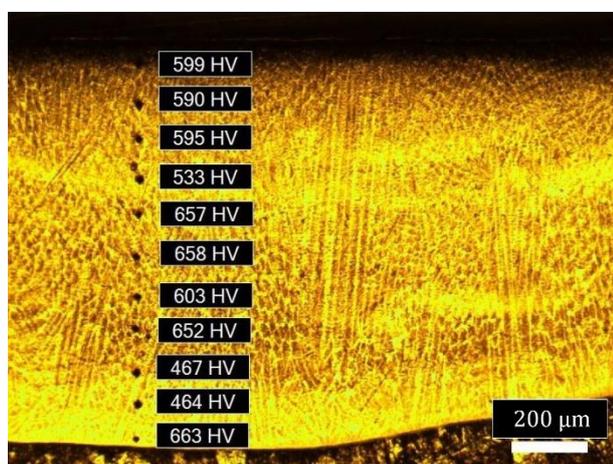
Fig. 4. Appearance of the surface layers and their cross-section of the central section.



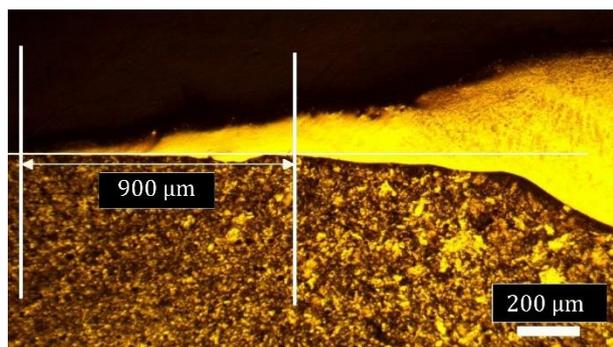


(b)

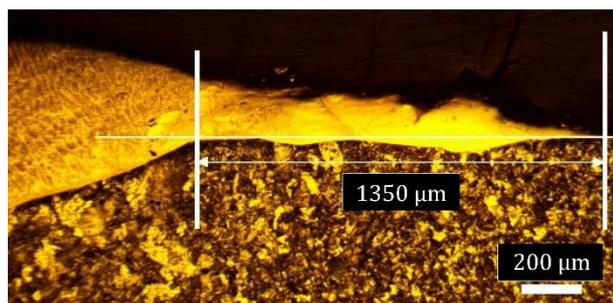
Fig. 5. Distribution of microhardness of the cross-section of the alloyed coating with a thick pre-coating of 0.25 (a1, 2) and 0.50 mm (b) of the Cu-Sn mixtures.



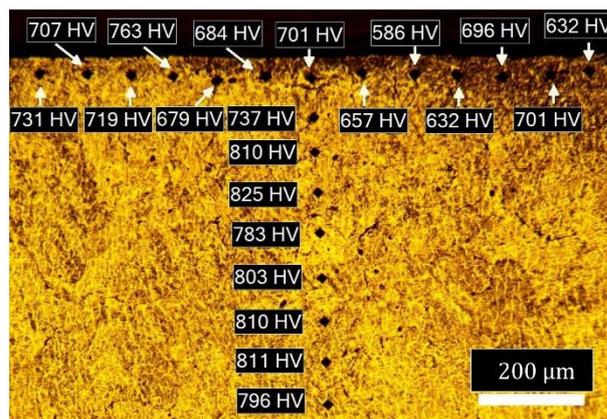
(a)



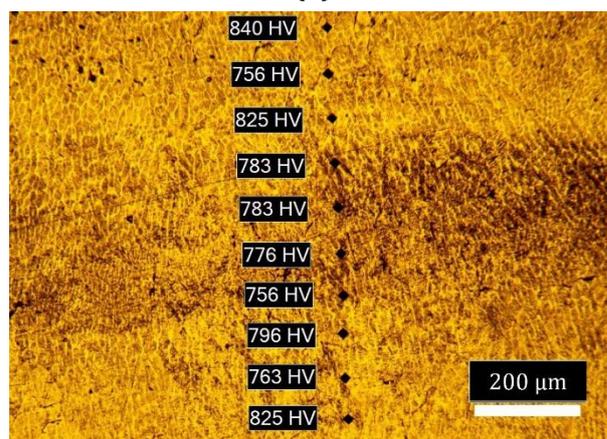
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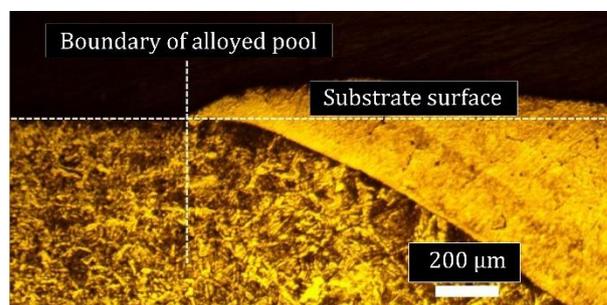
(c)



(d)



(e)



(f)

Fig. 6. Distribution of microhardness of the cross-section of coatings OK 84.78 and CuSn + 20% OK 84.78.

It is known that chromium is a good diffusion element for steel. This is indicated by the formation of a layer edge for pure OK 84.78 and 20% OK 84.78 with tin bronze as shown in Figure 6 (d, e, f). It can be seen that in the edge of the OK 84.78 coating the alloyed section remains at the boundary of the surfacing pool, and in the edge of the Cu-Sn + 20% OK 84.78 coating the alloyed section continues beyond the boundary of the surfacing, a weld puddle of more than a millimeter on the surface of unalloyed steel, shown in Fig. 6 (b). The dispersion of the values of the microhardness of the cross-section of the coatings is shown in Fig. 7. It can be seen that their concentration in the bronze

coatings is lower than in the bronze-carbide coating. This is explained by the fact that due to the presence of defects and incomplete saturation (dissolution) of bronze in the iron matrix, as a result, large zones of soft bronze phases remain, on which the indenters fall. Due to the high saturation for a thin pre-coating layer (0.25 mm), therefore, the concentration of the hardness values of the surface layer is higher than that of a thick pre-coating layer (0.50 mm). 75% of the hardness values of the coating No 3 (containing chromium carbide) is higher than the value of 100% of the coating No 1 and 75% of the No 2. In addition, the average value of the coating No. 3 is higher than the average value of the coatings No1, 2 about 150 HV.

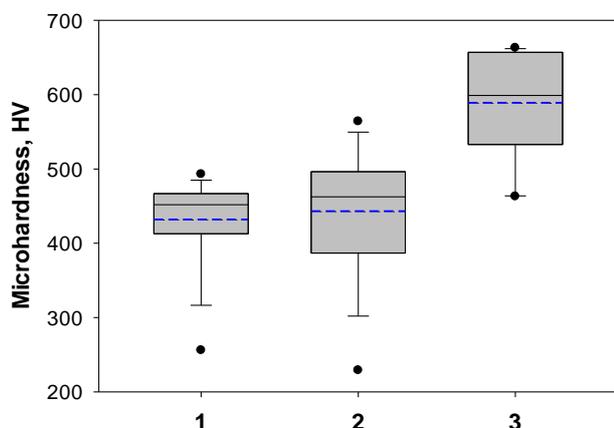
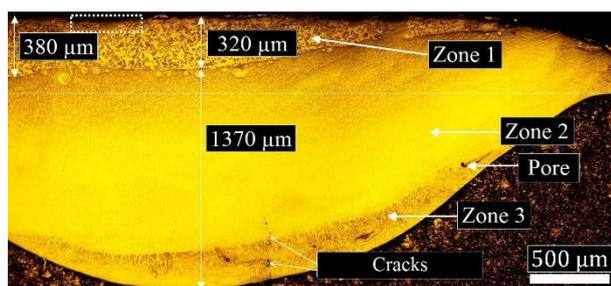
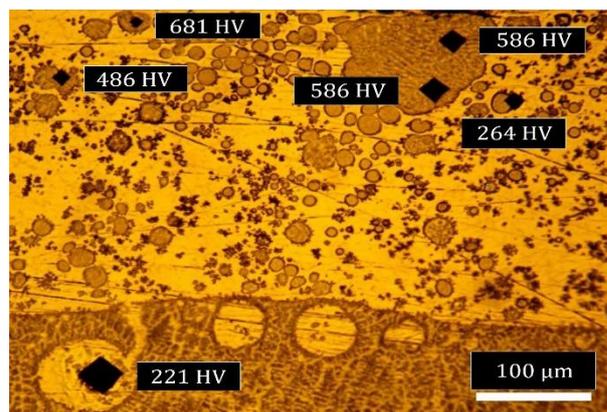


Fig. 7. Dispersion of microhardness values of the cross-section of coatings; 1-CuSn (0,25 mm); 2-CuSn(0,50 mm); 3-CuSn+20%OK 84.78 (0,25 mm).

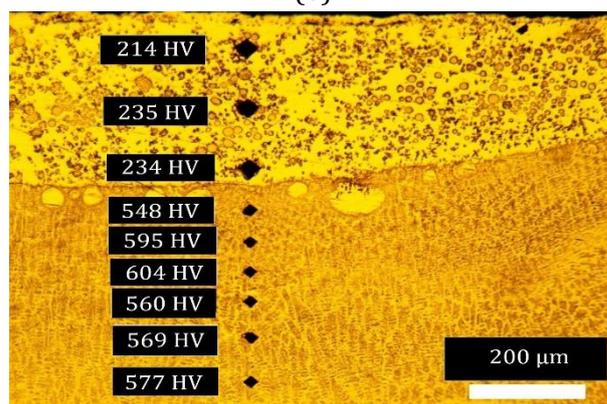
The cross-section of a coating made of the pre-coating (0.50 mm) of the mixture of Cu-Sn and 20% OK 84.78 with the distribution of microhardness values is shown in Figure 6. It can be seen that the depth of the alloyed layer is ~ 1700 μm, which is significantly higher than that of coating 0.25 mm of this composition. The resulting surface layer is not completely saturated; there are three different zones in it, shown in Figure 8 (a) and defects (pores and cracks). The upper zone (zone 1) has a maximum depth of 380 μm from the surface with a mixture of rich bronze and saturated particles of iron and Chromium carbide.



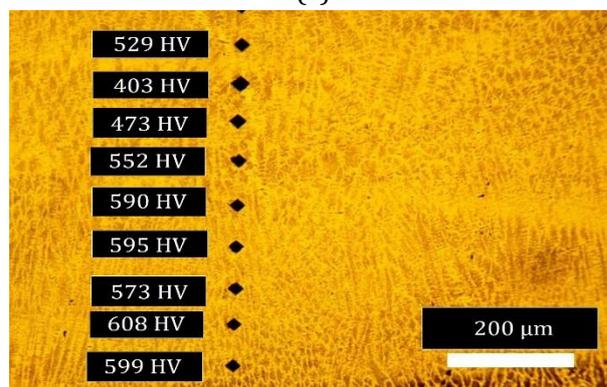
(a)



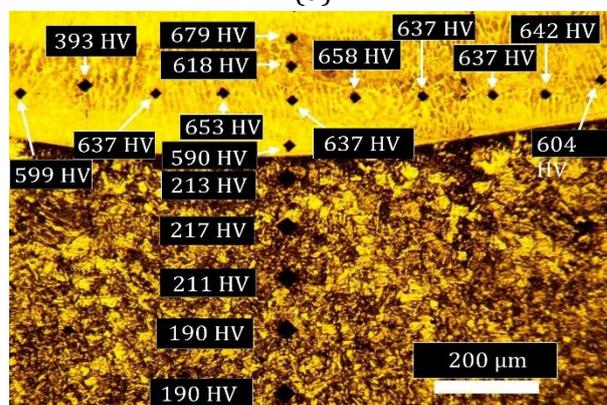
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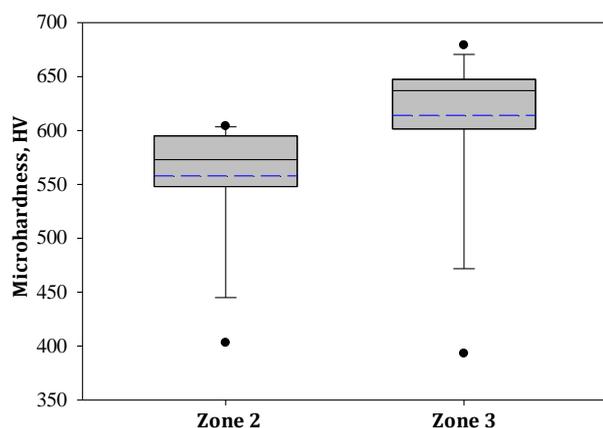


(e)

Fig. 8. Distribution of microhardness of the cross-section of an alloyed coating with a thick pre-coating of 0.50 mm of the Cu-Sn + 20% OK 84.78 mixture.

Especially, on the left side of the coating, there is a small zone containing saturated phases of iron and chromium, represented by a dashed elliptical region. The presence of these particles can occur due to the movement of the solid solution convection jet at the moment of incomplete saturated mixing in the Cu matrix. This zone has the microhardness in the range of 200-250 HV (Fig. 8 (c)). However, the hardness of the small iron saturated particles is slightly higher than that of the basic copper matrix, shown in Figure 8 (b) approximately for a dashed rectangular region. Almost all values of the microhardness of the lower zone (zone 3 - fig. 8 (e)) are from 393 to 680 HV, and for the central zone (zone 2 - fig. 8 (d)) from 400 to 610 HV.

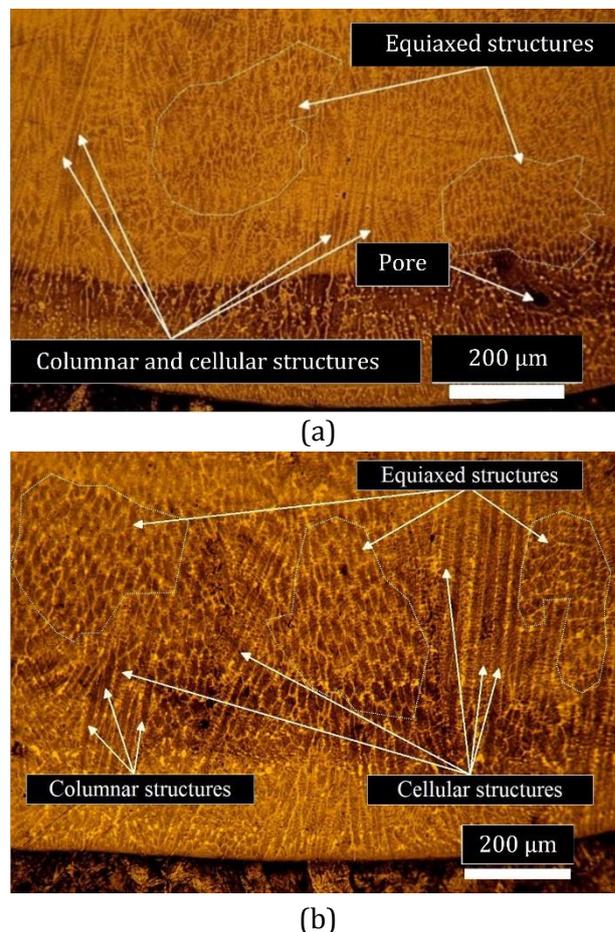
The high hardness of the lower zone can be caused by the high saturation of chromium carbide due to the close radius of chromium to iron. Despite the wide distribution of hardness, values of zone 3, but 75% of the values are higher than 75% of the values of zone 2. This is, respectively, with the distribution of soft Cu phases in depth and the ability to diffuse chromium and carbon in the interior of the base steel. Also seen in fig. 9, the dispersion of 75% of the high hardness values of the two zones found that chromium carbide strongly influences their high concentration. The bottom ejection values are represented by uneven distribution of hard phases and Cu in places where Cu phases remain more.



**Fig. 9.** Dispersion of microhardness values of zones 2, 3 of coating No. 5.

The main zones of alloyed coatings from a mixture of Cu-Sn and 20% OK 84.78 also have similar phase structures shown in Figure 8. Independent of the coating thickness, the main structures are equiaxed, columnar and cellular. However, in

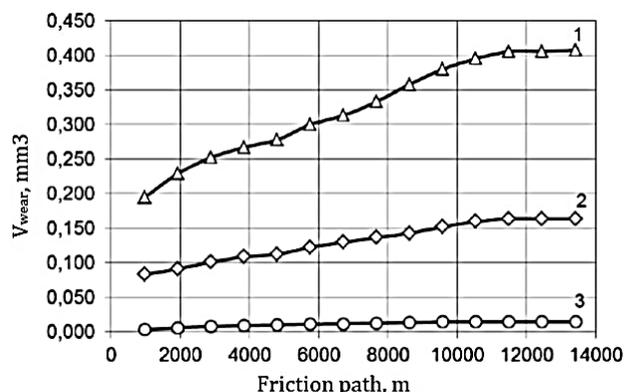
coating No. 2 - 0.25 mm of pre-coating (Fig. 10 (a)), the structures are more distinct and larger than in coating No. 5 - 0.50 mm. Only for the lower coverage zone No. 5, the structures are already more distinct than those of the upper zone (Fig. 10 (b)). This may be due to a decrease in the diffusion content of bronze, which causes a decrease in hard phases that form large grains.



**Fig. 10.** Typical structures of Cu-Sn + 20% OK 84.78 coatings with pre-coating thickness: (a) - 0.25 mm; (b) - 0.50 mm.

The durability of the coatings under sliding friction was evaluated according to the "disk - plane" scheme. In the course of the research, the amount of worn out material was assessed (Fig. 11). Based on the test results, the dependences of the volume of the worn out material on the friction path were plotted. At the initial stage, for all samples, a significant increase in the size of the wear hole is not observed. Wear of coatings (Cu-Sn, Cu-Sn + 20% OK 84.78) formed by plasma surface melting is characterized by a gradual increase in the volume of worn material. At values of the friction path over 9000 m, the wear process is stabilized. The wear process is

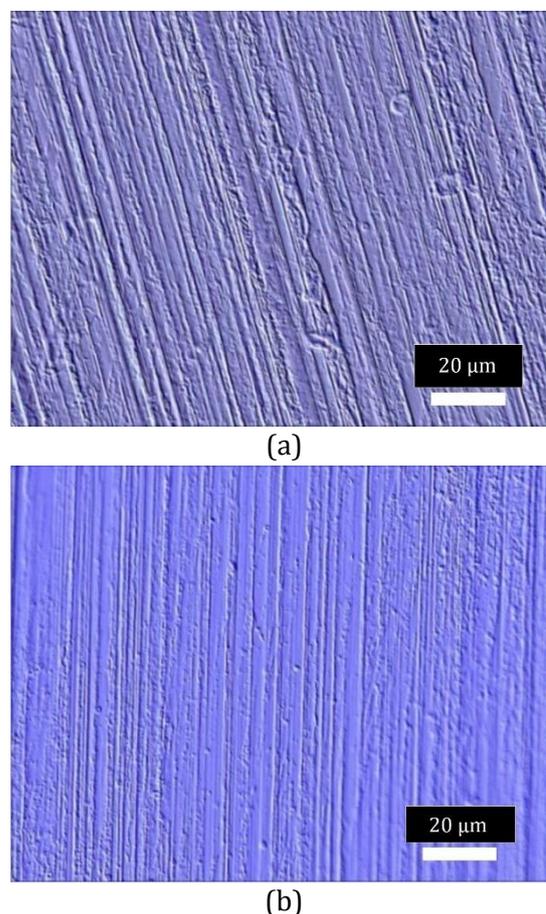
accompanied by an increase in the volume of worn material approximately every 4000 m. During the test, a transition to steady-state friction was not recorded. During wear, the surface of the Cu-Sn coating became rough with numerous sticky spots; there was clearly plastic deformation. The lamellar fragments were torn off from the surface of the Cu-Sn coating, which indicates strong adhesive wear. In contrast, the worn surface of the Cu-Sn + 20% OK 84.78 coating was relatively smooth, with little scratches and little adhesive wear characteristics Fig. 12(a). The worn surface of the specimen, corresponding to the Cu-Sn coating, is characterized by a large number of adhesion craters and fatigue pits, as well as rather long continuous grooves formed during wear tests (Fig. 12 (a)). This is due to the low strength and lower hardness of the Cu-Sn coating.



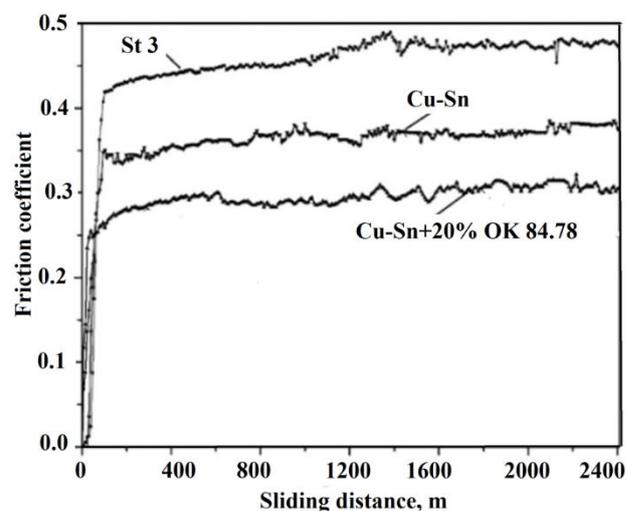
**Fig. 11.** Volumetric wear of materials during tests under sliding friction conditions: (1- steel sample witness; 2- Cu - Sn coating; 3- metal coating from a Cu-Sn mixture + 20% OK 84.78).

These observations indicate an improvement in adhesive wear, which is consistent with the results in Fig. 10. Plastic deformation of the coatings of the Cu-Sn + 20% OK 84.78 mixture during dry sliding is difficult due to their relatively high hardness. Thus, their excellent resistance to plastic deformation and abrasion leads to increased resistance to abrasion of plastic and adhesive wear (Fig.12(b)). The surface coating of the metal from a mixture of Cu-Sn + 20% OK 84.78 provides an increase in wear resistance and at the time of testing the volume of worn material did not exceed 0.010 mm<sup>3</sup>. Eutectic colonies present in the alloy effectively absorb plastic deformation, which prevents cracking and destruction. During the tests, a relief with a roughness Ra = 0.162 is formed. Analysis of the topography of the friction surface indicates the formation of separate

grooves of small depth. The results obtained indicate a low wear rate of the material when working in a friction pair. Tests of steel billets have shown that volumetric wear reaches 0.17 mm<sup>3</sup>. The coefficient of friction of the Cu - Sn coating and the coating based on CuSn + 20% OK 84.78 are shown in Fig. 13.

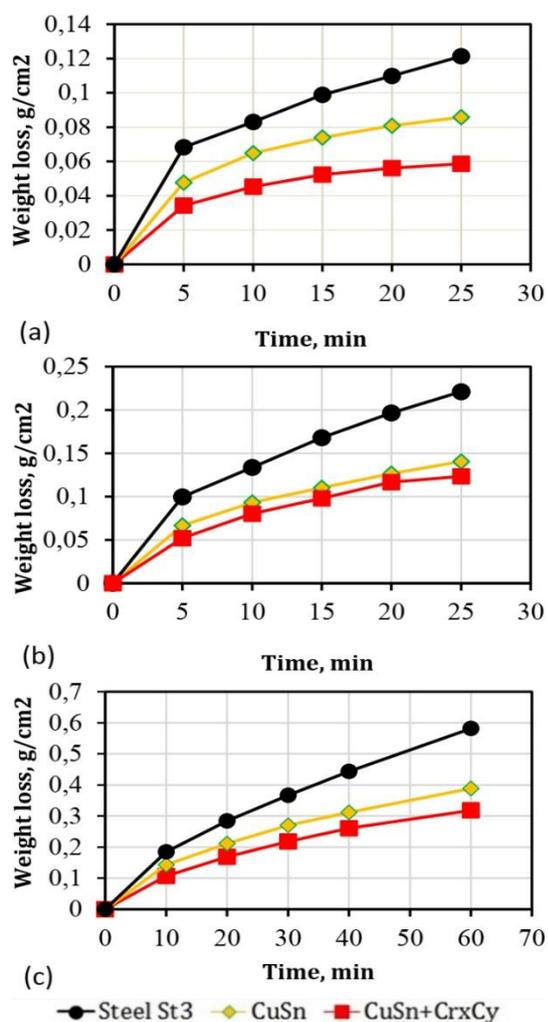


**Fig. 12.** Worn surfaces of specimens: (a) Cu-Sn, (b) Cu-Sn + 20% OK 84.78.



**Fig. 13.** Variations of friction coefficient with sliding distance of specimens.

The resistance of coatings under conditions of friction against the fixed abrasive will be determined from the structure of the main alloyed layer, the heat-affected zone (HAZ). Analysis of literature sources on wear tests of surface coatings showed that in order to achieve a complete wear process up to the base metal zone, several wear areas with different intensities will be observed. The state of the sample surface and, consequently, the resistance to abrasive wear will change along the depth of the hardened layer in the following order: alloyed layer → alloyed layer + heat-affected zone → heat-affected zone + base metal zone → base metal zone. In the course of the tests, we used two variants of rotation, the codirectional movement of the sample holder from the aluminum plate and the non-co-directional one. Let us consider the results in the case of the co-directional movement of the sample holder relative to the aluminum plate in Fig. 14.



**Fig. 14.** Weight loss of samples under loads: 5, 20, 50 N, (a) Sandpaper - 220 SiC, Load - 5 N, Speed - 100 revs/min, Water, (b) Sandpaper - 220 SiC, Load - 20 N, Speed - 100 revs/min, Water, (c) Sandpaper - 220 SiC, Load - 50 N, Speed - 100 revs/min, Water.

Of all the graphs shown in Fig. 14 it can be seen that all the coatings under study are more wear-resistant than the reference steel St3 sample (without coating). For Cu-Sn plating, surface cracking can be the reason for reduced wear resistance. In contrast, the Cu-Sn coating with the addition of chromium carbide with a higher hardness, has a high wear resistance regardless of defects. As the test time increases, the curvature of the time-mass loss curve increases; indicates that the inner sublayers of the surface layer are significantly saturated with a reinforcing component with high uniformity, and the number of defects in them is reduced. This is also consistent with many studies related to the permeability and solubility of copper in iron and diffusion of chromium in steel. To accelerate the wear process, the load was increased to 20 N. It can be seen that all coatings and even St3 steel exhibit a greater weight loss than in the case of a load of 5 N, shown in graph (Fig.14 (b)). At the same time, the CuSn-CrxCy coating not only continues to exhibit outstanding wear resistance, but also maintains this level over time, showing a curve that continues to bend more than other coatings. To test the same samples, the load was increased to 50 N, and the time between two measurements is 10 minutes. The results are shown in graph (Fig.14 (c)). It can be seen that the plasma coatings continue to show good wear resistance, with the CuSn-CrxCy coating being the best. After testing, the wear surfaces were examined using an optical microscope and the surface roughness was measured. It was found that on the surface of the worn out hardened zones, traces of microcutting are observed in the form of clear scratches of various lengths and depths. Microcutting processes are associated with the action of abrasive particles with the surface, which is confirmed by microcutting traces in the form of grooves and dumps with sharp edges, oriented in the direction of movement of abrasive grains. The interaction of the abrasive particle with the surface consists in the removal of metal microchips and the displacement of the material to the edge of the formed risks under the action of plastic deformation. In addition to deep scratches on the Cu-Sn coating, delamination of fragments of the coating is also observed, which indicates a low cohesive strength associated with the formation of oxide layers at the interlamellar boundaries. The development of the microcutting mechanism indicates that the hardness of the used abrasive is more than 1.3 times higher than the hardness of the structural components of the fused coating Cu-Sn coating.

#### 4. CONCLUSIONS

It was established that various coatings were obtained with plasma heating of the mixture PRV-BrO10 and the coating of the welding electrode OK 84.78 with different thicknesses: saturated with a thin-alloyed layer, In completely saturated with a thick-alloyed layer. Measurement of microhardness showed that the addition of chromium carbide leads to an increase in hardness compared to a coating based on Cu-Sn alloy; its values are in the range of 460-670 HV. In the case of a thick pre-coating layer (0.50 mm), the coating has different zones: soft, rich in copper with mixing of hard particles (200-250 HV), and the rest, saturated with chromium carbides (400-700 HV). Uneven surface, heterogeneity and defects cause difficulties in controlling technological parameters in the case of a thick layer of alloy coating to obtain a high-quality alloyed coating.

The results of tribotechnical tests under sliding friction conditions of friction against a fixed abrasive indicate the high efficiency of the obtained coatings using plasma hardening. The highest results were achieved with the content of chromium carbides in the CuSn-CrxCy coating. The wear resistance of the coatings obtained in this case is significantly higher in comparison with the samples based on the Cu-Sn alloy. The coefficient of friction of the coatings based on Cu - Sn - CrxCy was higher than that of the Cu - Sn coating.

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