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Original Empirical Research

Morphology and Properties of the Laser-Irradiated Composition "Chrome Coating — Copper Substrate"

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Abstract

Introduction. During pulsed laser processing and modification of the surface of non-ferrous alloys and coatings based on them, several still unresolved issues arise. In particular, the extreme thermal deformation conditions of laser processing are not linked to the peculiarities of structure formation and formation of properties in irradiated "coating — copper substrate" compositions. A metal physical analysis of the possibility and reasons for increasing the adhesion strength of coatings to a metal (copper) substrate during high-speed laser processing is insufficiently substantiated and evidence-based. To make a reasonable choice of technological parameters for the surface hardening mode of non-ferrous alloy products, as well as for obtaining high-quality workable composite layers on their surface, it is necessary to solve the above issues and tasks. The aim of this article is to determine the possibility and conditions for increasing the adhesion strength of a chrome coating to a copper substrate under laser irradiation of the composition.

Materials and Methods. Metal physical studies in the work were carried out on samples of non-ferrous alloys of the Cu–Zn system with a chrome electrochemical coating with a thickness of 20 µm. The "copper substrate — chrome coating" composition was irradiated at a Kvant-16 installation with a radiation power density of 70–250 MW/m^2. Metallographic structural analysis, scanning probe microscopy, and durometric studies were used in the work.

Results. It has been calculated that the dynamic and thermal stresses arising in the laser-irradiated compositions "chrome coating — copper substrate" were about 320 MPa. Metal physical studies revealed that, in extreme thermal deformation conditions of laser treatment, the effect of contact melting was manifested at the boundary of the coating with the copper base. Dynamic recrystallization occurred in the surface layers of the irradiated L62 copper alloy, resulting in the formation of grains with a size of 4.5–5.0 µm on the surface of the alloy with an initial grain size of 25 µm.

Discussion and Conclusion. It has been found that the adhesion strength of a chrome coating to a copper alloy substrate increased laser irradiation at a radiation power density of 150 MW/m^2. This was due to the formation of a transition region 2–4 µm deep in the contact zone with a structure consisting of sections of mutually insoluble solid solutions based on chromium and copper. Based on the analysis of the copper —chromium state diagram and the model of the temperature field under laser irradiation of the chromium coating, it was suggested that contact melting occurred in the transition zone from the coating to the copper substrate. It was shown that thermostrictive stresses, the calculated quantitative values of which were about 320 MPa, had an initiating effect on the observed processes of structure formation in the laser irradiation zones. It was found that such a level of stresses arising in copper alloys under laser irradiation was sufficient for plastic deformation and dynamic recrystallization of the metal and contributed to the formation of a fine-grained structure (4.5–5.0 µm) with an initial grain size of 25 µm. An analysis of the results of studies of irradiated compositions "coating — copper substrate" allowed us to conclude that they expanded the technological capabilities of the laser method of hardening materials and ensure guaranteed high performance of irradiated products with coatings.

Keywords: copper alloys, coatings, laser irradiation, structure, properties

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Оригинальное эмпирическое исследование

Морфология и свойства лазернооблученной композиции «хромовое покрытие — медная подложка»

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Аннотация

Введение. При проведении импульсной лазерной обработки и модифицирования поверхности цветных сплавов и покрытий на их основе возникает ряд до сих пор не решенных проблем. В частности, не увязаны экстремальные термодеформационные условия лазерной обработки с особенностями структурообразования и формирования свойств в облученных композициях «покрытие — медная подложка». Недостаточно аргументированно обоснован и доказательно проведен металлофизический анализ возможности и причин повышения прочности сцепления покрытий с металлической (медной) подложкой при высокоскоростной лазерной обработке. Для обоснованного выбора технологических параметров режима поверхностного упрочнения изделий из цветных сплавов, а также для получения на их поверхности качественных работоспособных композиционных слоев требуется решение приведенных выше вопросов и задач. Целью данной статьи явилось определение возможности и условий повышения прочности сцепления хромового покрытия с медной подложкой при лазерном облучении композиции.

Материалы и методы. Металлофизические исследования в работе проводились на образцах цветных сплавов системы Cu–Zn с хромовым электрохимическим покрытием толщиной 20 мкм. Композиция «медная подложка — хромовое покрытие» облучалась на установке «Квант-16» с плотностью мощности излучения 70–250 МВт/м^2. В работе использовались металлографический структурный анализ, сканирующая зондовая микроскопия, дюрометрические исследования.

Результаты исследования. Расчетным путем установлено, что возникающие в лазернооблученных композициях «хромовое покрытие — медная подложка» динамические и термические напряжения составляют около 320 МПа. Металлофизическими исследованиями обнаружено, что в экстремальных термодеформационных условиях лазерной обработки на границе покрытия с медной основой проявляется эффект контактного плавления. В поверхностных облученных слоях медного сплава Л62 обнаружен эффект динамической рекристаллизации. Это выражается в формировании на поверхности сплава с исходным размером зерна 25 мкм мелких зерен размером 4,5–5,0 мкм.

Обсуждение и заключение. Установлено, что прочность сцепления хромового покрытия с подложкой из медных сплавов повышает лазерное облучение с плотностью мощности излучения 150 МВт/м^2. Это происходит за счет формирования в зоне контакта переходной области глубиной 2–4 мкм со структурой, состоящей из участков взаимно нерастворимых твердых растворов на основе хрома и меди. На основании анализа диаграммы состояния «медь — хром» и модели температурного поля при лазерном облучении хромового покрытия высказано предположение о протекании в переходной зоне от покрытия к медной подложке контактного плавления. Показано, что инициирующее влияние на наблюдаемые процессы структурообразования в зонах лазерного облучения оказывают термострикционные напряжения, расчетные количественные значения которых составили около 320 МПа. Установлено, что такой уровень возникающих в медных сплавах при лазерном облучении напряжений достаточен для пластической деформации и динамической рекристаллизации металла и способствует формированию мелкозернистой структуры (4,5–5,0 мкм) при исходном размере зерен 25 мкм. Анализ результатов исследований облученных композиций «покрытие — медная подложка» позволил сделать вывод, что они расширяют технологические возможности лазерного метода упрочнения материалов и позволяют гарантированно обеспечивать высокую работоспособность облученных изделий с покрытиями.

Ключевые слова: медные сплавы, покрытия, лазерное облучение, структура, свойства

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Introduction. In the work of G.V. Lomaev and E.V. Kharanzhevskii, "Hardening Surface Treatment Using High-Speed Laser Overcrystallization" [1], and in some other studies [2, 3], it has been shown that during laser irradiation, high temperature gradients, thermostrictive stresses of various origins appear in the surface layers of the hardened material, the relaxation of which leads to local plastic deformation, as well as the phenomena of dynamic return, polygonization, and recrystallization of metal in irradiated zones. The possibility to achieve a sufficiently high defect density in a crystalline structure and the desired level of mechanical properties in the surface layers is of practical significance [4].

It should be noted that during pulsed laser treatment [5], hardening [6] and modification [7] of the surface of non-ferrous alloys, in particular, copper alloys, several problems arise [8]. For example, the extreme thermal deformation conditions of pulsed laser treatment are not linked to the peculiarities of structure formation and formation of properties in irradiated "coating — copper substrate" compositions [8]. The reasons for increasing the adhesion strength of coatings to a metal (copper) substrate during high-speed laser processing have not been sufficiently substantiated [9].

The solution of the above-mentioned problems is of significant importance, as it allows for the reasonable assignment of technological modes of surface hardening and microalloying for copper alloy products. This, in turn, contributes to the creation of high-quality, workable composite surface layers on copper parts, thus creating conditions for trouble-free operation of irradiated components in mechanisms located in difficult-to-repair areas. In this regard, the aim of this article is to obtain, analyze, and quantify the results of metal physical studies, as well as to assess the degree of influence of pulsed laser treatment on the formation of structure and properties of the irradiated "chrome coating — copper substrate" composition.

Materials and Methods. Metal physical studies were conducted on samples of non-ferrous alloys of the Cu–Zn system with a chrome electrochemical coating with a thickness of 20 µm. The "copper substrate — chrome coating" composition was irradiated at a Kvant-16 installation with a radiation power density of 70–250 MW/m^2.

Metallographic structural analysis, scanning probe microscopy, and durometric studies were performed. MIM-7 and Neophot-21 microscopes were used for the metallographic analysis. Microhardness measurements were conducted on the PMT-3 device with an indenter load of 0.49 N.

The adhesive properties of the coatings were determined based on indirect experiments to measure the microhardness of the coatings under different indentation loads. Traces of destruction or peeling of the coatings from the surface of the copper substrate, as observed on the cross-section of the coating–substrate composition, were considered a criterion for insufficient adhesion strength.

Research Results. As a result of metallographic and durometric studies, we found that laser treatment improved the adhesion strength between the chrome coating and the copper substrate (Fig. 1). This conclusion was based on the fact that there was no chipping of the coating when the microhardness tester indenter was pressed into it, that is, when a load was applied.

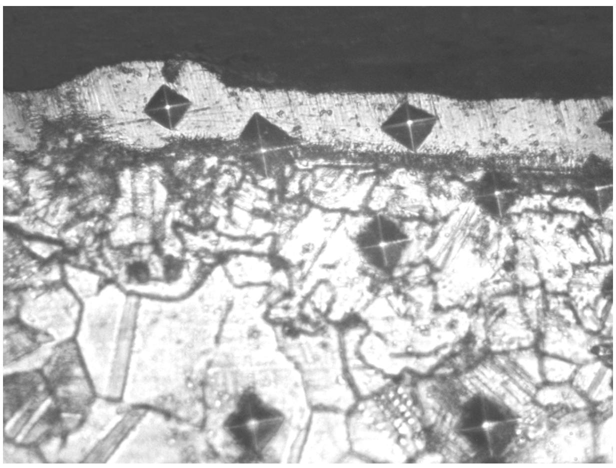

Fig. 1. Structure of the chrome coating on the copper alloy (brass L62) with impressions from the microhardness tester indenter

At the same time, as can be seen in Figure 2, in the transition zone at the boundary between the chrome coating and the copper substrate, that is, at a depth of 20 µm, after laser treatment, there were clearly visible local areas of solid solutions, apparently based on chromium and copper, penetrating each other at a distance of 2–4 µm.

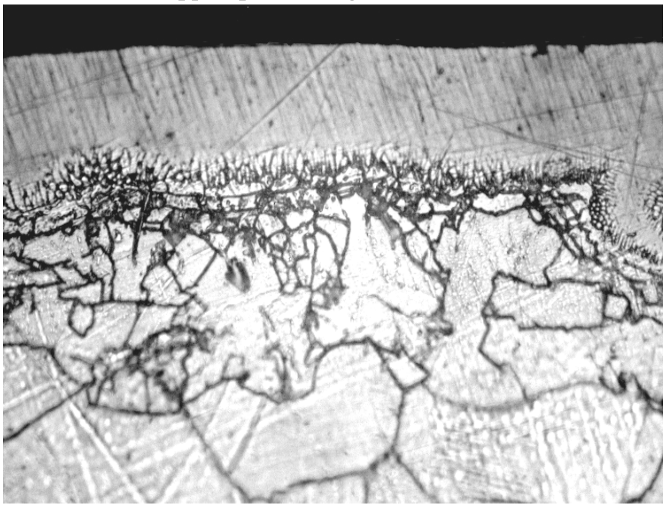

Fig. 2. Microstructure of the contact zone of the chrome coating with brass after laser irradiation

To study the features of the structure formation at the boundary of the coating and the substrate, an analysis of the state diagram of the Cr–Cu system was conducted [10]. It was noted that at a temperature of 1767°C, monotectic equilibrium was observed; the liquid phase was stratified to form two liquids of different chemical composition. It could be assumed that the observed in Figure 2 structure of the contact zone of the coating and the substrate was also formed from a state melted by laser exposure, that is, an immiscibility region of copper and chromium in the liquid state was formed in the transition zone, in which two solid solutions based on Cu and Cr were fixed after high-speed crystallization. But the quantitative analysis of the temperature field carried out using the Mathcad software package during laser irradiation of the chrome coating [11] excluded the possibility of reaching melting temperatures at the depth of the coating, that is, at a depth of 20 µm (Fig. 3).

The "chrome coating — copper substrate" composition was exposed to radiation with a power density of 150 MW/m^2. These energy conditions did not lead to evaporation of the coating and to the formation of "craters" on the surface. Thermal calculations, which are shown in Figure 3, allowed us to determine that a temperature of 900–1000°C was achieved in the transition zone. However, this temperature was not high enough for melting, and no formation of two solid solutions based on the components of the "chrome coating – copper substrate" composition was observed during the study. The possible cause of the described effect requires a separate discussion.

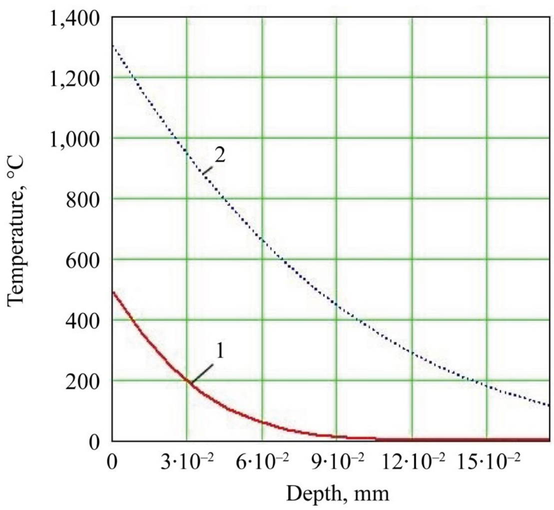

Fig. 3. Temperature distribution over the depth of the chrome coating at the heating stage
(1 — the beginning of the laser pulse; 2 — he end of the pulse)

Layers of copper alloy at a depth of 5–10 µm were subjected to melting and subsequent high-speed crystallization. A feature of the structure of the fused surface layer of brass, as shown by the results of studies on a scanning probe microscope (SPM), was a homogeneous dispersed structure consisting of solid solution dendrites with a cross section of up to 25 nm (Fig. 4) and with a sufficiently high hardness of about 1 GPa.

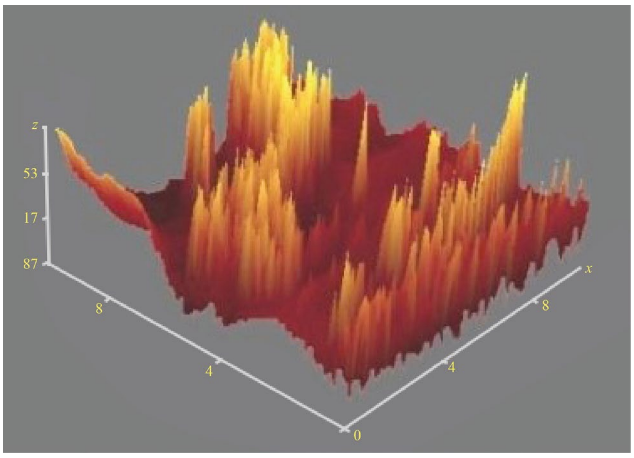

Fig. 4. Scanned image of the surface of the L62 copper alloy (SPM)

Under the influence of heating alone, the recorded structural transformations could not occur during the laser pulse action (10^{-3} s). However, thermostrictive stresses appeared to have made a significant contribution to the acceleration of structure formation processes.

In order to determine the level of stresses and local plastic deformations in the irradiated zones of alloys, quantitative estimates were made using “model” single-component copper samples. As shown above, the melting point was not reached in the contact zone of the coating and the substrate, therefore, the irradiation of uncoated copper samples was carried out in a mode that did not lead to melting of the surface (with a radiation power density of 100 MW/m^2). After laser irradiation, as can be seen in Figure 5 *a*, sliding lines appear on the pre-polished surfaces of copper samples, that is, traces of local plastic deformation of the metal.

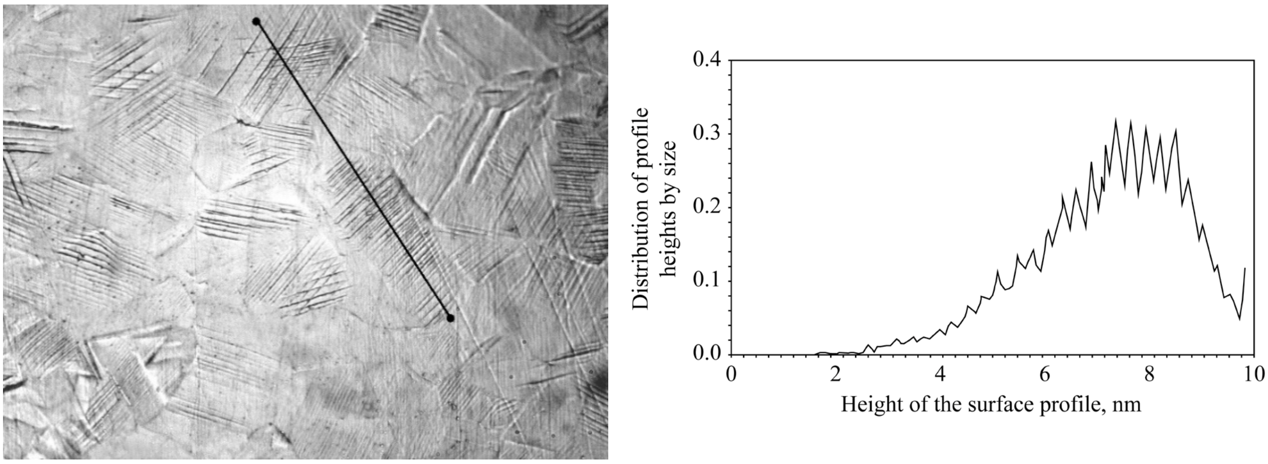

Fig. 5. Microstructure and height distribution of the emerging surface relief on copper after laser irradiation:
a — sliding lines; *b* — height distribution of the surface relief

It should be noted that the sliding lines are formed due to the movement of dislocations in the areas of the metal that have been irradiated [12]. Due to the appearance of different numbers of dislocations on the irradiated surface, a relief with varying heights of the surface profile was created [13] (shear steps h in Fig. 6).

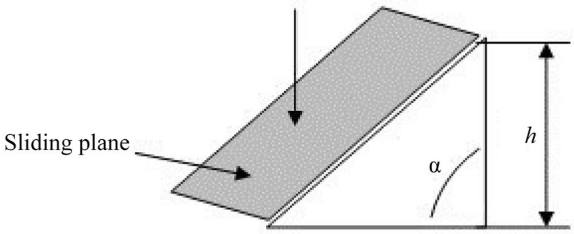

Fig. 6. Diagram of the shear step on sliding lines [14]

The analysis of the pattern of the sliding lines and the relief of the irradiated surface was carried out using the computer image processing program Gwiddion. As can be seen in Figure 5 *b*, the average height of the profile of the irradiated surface of the copper sample, and therefore the height of shear site *h*, was 6.5–8.5 nm. Based on the results of measurements of this height, it was possible to judge the degree of plastic deformation achieved during metal processing and the level of stresses causing it.

Considering that during plastic deformation, the height of step *h* was proportional to value *nb*, where *n* — number of dislocations that have come to the surface, and *b* — Burgers vector of the irradiated material, we can write:

$$nb = h. \tag{1}$$

To simplify the calculation of shear stresses, it was assumed that the elastic deformation preceding slid in the crystal could be defined as *t*/*G*, where t — applied stress; *G* — shear modulus of the material. It was also taken into account that elastic deformation during sliding could relax in an area with a diameter of 2*L*, where *L* — length of the dislocation slip lines.

Assuming the complete transition of elastic deformation ($2L\tau/G$) to plastic deformation equal to the value *nb*, we obtain [14]:

$$\tau = h\left(\frac{G}{2L}\right).$$

By calculation, we determine shear stress (τ), causing plastic deformation using expressions (1), (2) and, taking $L = 10^{-3}$ mm, from expression [15]:

where *h* — height of the shear site, during laser processing was 6.5–8.5 nm; *G* — shear modulus, for copper $G = 4.3 \times 10^4$ MPa; *L* — length of the dislocation slip lines, we assume $L = 10^{-3}$ mm.

Shear stresses amounted to approximately 320 MPa, which exceeded the conditional yield strength of copper (40–80 MPa) and led to plastic deformation of the surface layers of the irradiated metal.

The work also assessed the degree of local residual plastic deformation, which ranged from 5 to 9% in the case of laser treatment of copper.

Possible structural processes in the laser irradiation zones of the "coating – metal substrate" composition, in particular, in a copper substrate, were studied in detail using the Mathcad program. A temperature field was constructed that spread along the depth after irradiation of the surface of the brass sample (Fig. 7).

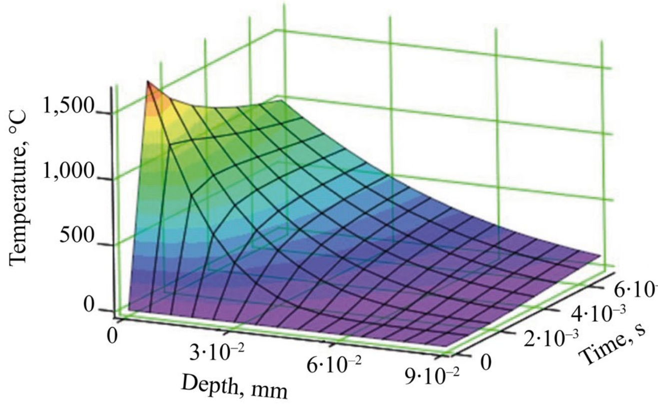

Fig. 7. Temperature field in a copper alloy during laser irradiation of the surface

As can be seen in Figure 7, at a depth of 15 μm, the temperature dropped to 800°C. At this temperature, in the thermal deformation conditions of pulsed processing, despite the extremely short time of thermal exposure to laser radiation, there was a possibility of dynamic stress relaxation effects accompanying the process of high-speed laser irradiation, that is, processes of polygonization and recrystallization in irradiated zones on copper alloys are possible. The consequences of recrystallization of irradiated brass L62 are shown in Figure 8 *a* in the form of grinding of grains of solid solution.

A similar recrystallization process was observed in a brass substrate under a chrome coating after laser irradiation. It was found that in the presence of a coating, the metal grain was larger than on an uncoated copper sample. Apparently, recrystallization in this case reached the stage of collective recrystallization. It can be concluded that there was a lower level of residual stresses and a decrease in the risk of cracking in the composition during laser irradiation.

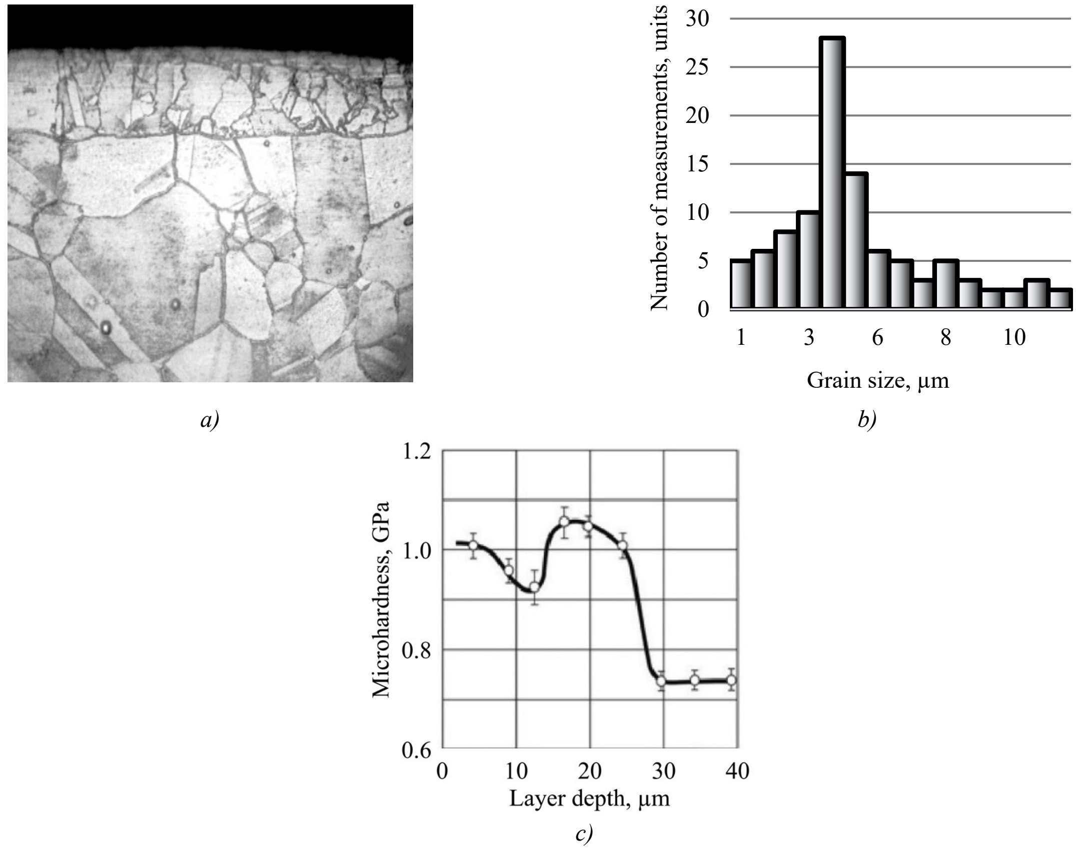

Fig. 8. Microstructure, histogram of size distribution of the subgrains and hardness distribution over the depth of the irradiated zone on the L62 copper alloy: *a* — microstructure; *b* — size distribution of the subgrains; *c* — hardness distribution over the depth of the alloy L62

The average size of the recrystallized grains, as shown in Figure 8 *b*, was 4–5 µm with an initial grain size of 25 µm. At the same time, the highest hardness was achieved in the considered area of the irradiated spot on brass (Fig. 8 *c*).

Discussion and Conclusion. The increase in the adhesion properties of the "chrome coating — copper substrate" composition after laser treatment can be indirectly assessed through the results of durometric tests. It is notable that when the microhardness tester indenter was introduced into the coating, its peeling from the copper matrix was not observed, whereas immediately after application the coating had insufficiently high adhesion strength to the metal (copper) substrate.

The adhesion effects in the irradiated composition are positively influenced by grain grinding, i.e. recrystallization, in a copper substrate, as well as local structural ensembles formed in the transition zone at the boundary of the coating and the substrate from sections of solid solutions based on chromium and copper.

The observed processes of structure formation under the action of an "instantaneous" heat source are possible only under conditions of joint action of high temperatures and stresses on the irradiated composition.

To evaluate the parameters of these thermal power factors and determine the degree of their influence on the formation of structure and properties of irradiated materials, first of all, an analysis of the state diagram of the Cr–Cu system has been conducted. It showed that the observed structural transformations can occur if the contact zone between the chrome coating and copper substrate melts, that is, if the temperature is heated to 1767°C. At this temperature, according to the diagram, the formation of two liquids is observed, that is, in the transition zone between the coating and the substrate, an immiscibility region of copper and chromium in the liquid state should be formed, and after high-speed crystallization, two solid solutions based on Cu and Cr should result. The quantitative analysis of the temperature field, conducted using the Mathcad software package, during laser irradiation of the chrome coating, excluded the possibility of reaching melting temperatures at a depth of 20–30 µm, which is the depth of the contact zone studied in the composition. On the other hand, an analysis of structural processes in the laser irradiation zone of a copper brass sample has showed that melting and subsequent rapid crystallization of copper occur at a depth of 5–10 µm. Scanning probe microscope studies have confirmed that the surface fused layer of the copper sample has a dispersed structure and a sufficiently high hardness of approximately 1 GPa.

The most significant aspect is that the structure of the thin fused layer on the copper alloy is similar to the structure of sections of a copper-based solid solution in the contact area between the copper substrate and the chrome coating. This indirectly confirms that in the transition zone from the coating to the copper substrate, despite heating to temperatures below the melting point, a liquid phase was still present. It can be assumed that contact melting occurs at the studied boundary under extreme thermal deformation conditions of laser treatment, leading to the described structure formation and, as a result, to an increase in the adhesive properties of the "chrome coating — copper substrate" composition.

It should be noted that the results obtained are primarily related to the extreme temperature and force conditions of laser irradiation. Significant temperature gradients combined with high laser processing speeds lead to the formation of high levels of thermostrictive stresses and local plastic deformations in the irradiated areas of alloys. To confirm their existence and perform a quantitative assessment of the values they achieve, as well as for the purity of the experiment, research was conducted not only on copper alloys, but also on single-component copper samples [11]. After laser irradiation, sliding lines appeared on the pre-polished surfaces of the copper samples, indicating traces of local plastic deformation of the metal. The analysis of patterns of sliding lines and relief of the irradiated surface made it possible to determine that the average height of the surface profile of the irradiated copper sample, and, consequently, the height of the shear site was 6.5–8.5 nm.

Based on the measurements of this height and the calculations made, it was found that the shear stresses were approximately 320 MPa. These values were higher than the conditional yield strength of copper (40–80 MPa), which led to plastic deformation of the surface layers of the irradiated metal, which ranged from 5 to 9% [3].

It should be noted that the actual values of stresses and plastic deformation that arise during the action of a powerful thermal shock of a laser pulse on local metal sites will undoubtedly have much higher values.

Thermal deformation effects appearing in the areas of laser irradiation of alloys, despite the extremely short laser pulse time (10^{-3} s), lead to stress relaxation not only to local plastic deformation, but also to dynamic polygonization and recrystallization of the structure in the surface layers of metals.

As shown by the results of quantitative assessment of temperatures in the irradiated copper substrate, at a layer depth of 15 µm, the temperature drops to 800°C, that is, the zone of laser treatment of a copper sample in a solid state begins. It can be concluded that at this temperature, under the thermal deformation conditions of laser irradiation, despite the extremely short time of thermal exposure, it becomes possible to manifest dynamic polygonization and recrystallization in irradiated areas on copper. As a result, the copper grain is crushed from the initial size of 25 µm to 4–5 µm in the recrystallized metal. The evidence of dispersion of the structure of the surface irradiated layers of the alloy is the results of durometric analysis, which indicate that the highest hardness is achieved in the recrystallized zone.

The described recrystallization process leads to an increase in the crack resistance of the irradiated "chrome coating – copper substrate" compositions. This is due to the removal of stresses that appear in the coating during its application and high-speed laser processing, which reduces the likelihood of brittle destruction of the composition.

Thus, it was found that laser treatment with a radiation power density of 150 MW/m2 contributes to an increase in the adhesion strength of the chrome coating to a metal (copper) substrate due to the formation of a transition layer of specific morphology at the boundary of the coating and the base material, as well as due to recrystallization processes in the surface layers of the copper alloy.

An analysis of the results of studies of irradiated "coating – copper substrate" compositions has allowed us to conclude that they expand the technological capabilities of laser method of hardening materials and ensure guaranteed high performance of products with irradiated coatings.

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