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# КОНДЕНСАЦИЯ ЛАНҒАН КҮЙДІҢ ФИЗИКАСЫ

## ФИЗИКА КОНДЕНСИРОВАННОГО СОСТОЯНИЯ

### PHYSICS OF THE CONDENSED MATTER

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### **Influence of a strong beam on structure and properties of plasma coatings**

The effect of electron irradiation on the structure and properties of plasma coatings is studied. The coatings were obtained by vacuum spraying of Fe-Al and 12X18H10T cathodes. The microstructure, microhardness and friction coefficients before and after irradiation by electrons were studied. After irradiation, all the characteristics of the coatings change. Taking into account the surface tension at the interface, leads to a significant change in the kinetics of the formation of coatings. The greater the surface tension, the lower the dissipation rate of the free energy. This means that most of the energy supplied goes to the formation of the coating. In this case, as a rule, cellular nanostructures with increased hardness are formed. Methods of determination are proposed and the melting and recrystallization temperatures of plasma coatings are estimated. It is established that the melting temperature of coatings obtained in argon is higher than for coatings obtained in a nitrogen medium. It was shown experimentally that 12X18H10T + Al, 12X18H10T + Fe-Al and 12X18H10T + Cu coatings obtained in argon medium have higher melting and recrystallization temperatures. This means that the latter can be used as a heat-resistant coating on steel parts. It is shown that the surface tension of coatings obtained in a nitrogen medium is lower than for coatings obtained in argon. Hence, the formation of nitride phases leads to a decrease in the surface energy of the coatings. It is shown that the greater the surface tension of the coating, the higher its corrosion resistance. It is established that the corrosion resistance of most of the coatings studied is not inferior to the most corrosion-resistant steels.

*Keywords:* plasma coating, electron irradiation, surface energy, microhardness, friction, wear resistance, destruction, heat resistance.

#### *Introduction*

Recent years are characterized by a heightened interest in multi-element coatings. They already form the basis for obtaining functional materials for mechanical engineering, aviation equipment and much more. A feature of these materials is the possibility of changing their properties over a wide range by changing technological parameters [1-3]. You can change the composition of the coating, the parameters of coatings, the parameters of external influences.

More recently, binary (TiN, ZrN, etc.) systems have been used for coating. Then there was a transition to ternary systems and then to multi-element systems.

Among the multielement alloys and coatings, high-entropy alloys (VES) and coatings are distinguished as a separate class [4-7]. VES are alloys which contain not less than 5 elements, and the quantity of each of them should not exceed 35 at.%. For such alloys, the values of  $S_{\text{mix}}$  mixing entropy are higher than in traditional multicomponent alloys. A high value of entropy leads to the formation of single-phase structures of bcc or fcc type. For VEC, each element has an equal opportunity to occupy the crystal lattice site, if chemical ordering is not taken into account. Since the dimensions of the atoms can be very different, the lattice is highly distorted, which leads to high elastic stresses and to the braking of dislocations. This effect is confirmed by the superhigh strength of the bcc windfarm.

In this paper we investigate the effect of electron irradiation on the structure and properties of a multielement plasma coating.

### Experimental method

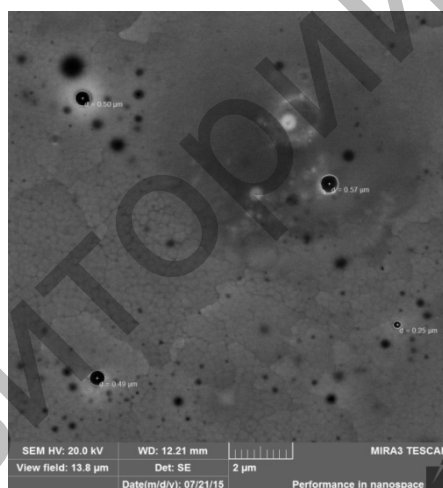
In the present work we used cathodes of the composition Fe-Al, which were obtained by induction melting, and cathodes made of steel 12Cr18N10T. Coatings were applied to steel samples by the ion-plasma method on a vacuum installation of NNV-6.6II while simultaneously spraying the cathodes mentioned above. The obtained samples were irradiated by electrons on a vacuum pulsed electron-beam installation of the CO-LO ISE SB RAS (Tomsk) using the procedure detailed in [8].

Electron microscopy was performed on a scanning electron microscope MIRA 3 from TESCAN. The investigations were carried out at an accelerating voltage of 20 kV and an operating distance of about 15 mm. For each sample, 4 images were taken from 4 surface points at different magnifications: 245 times, 1060 times, 4500 times and 14600 times. And also an energy-dispersive analysis was carried out at 4 points of the surface of each sample.

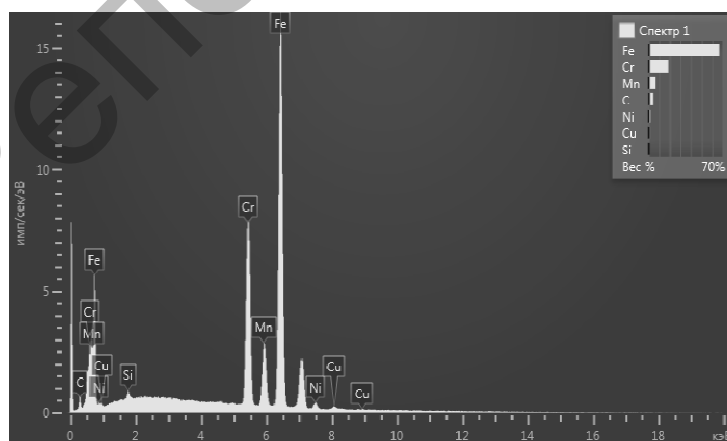
The optical microstructure was studied with the Epikvant metallographic microscope, and at the nanoscale with the NT-206 atomic force microscope. Investigation of microhardness of coatings was carried out on a microhardness meter HVS-1000A. Tribological studies were carried out on the installation described in [9].

### Results of the experiment

The 45-layer substrate was coated with Fe-Al + 12Cr18N10T coating in argon gas for 40 minutes. In Figure 1 shows the REM image of the coating, XPS and elemental composition before irradiation by electrons, and in Figure 2 - after irradiation with electrons. In Figure 3 shows an AFM image before and after irradiation by electrons.



a)



b)

Element	The weight, %
C	4.07
Si	0.36
Cr	25.35
Mn	5.09
Fe	62.37
Ni	2.00
Cu	0.75
Sum	100.00

c)

Figure 1. SEM image of the coating (a), XPS (b) and elemental composition (c) before irradiation with electrons

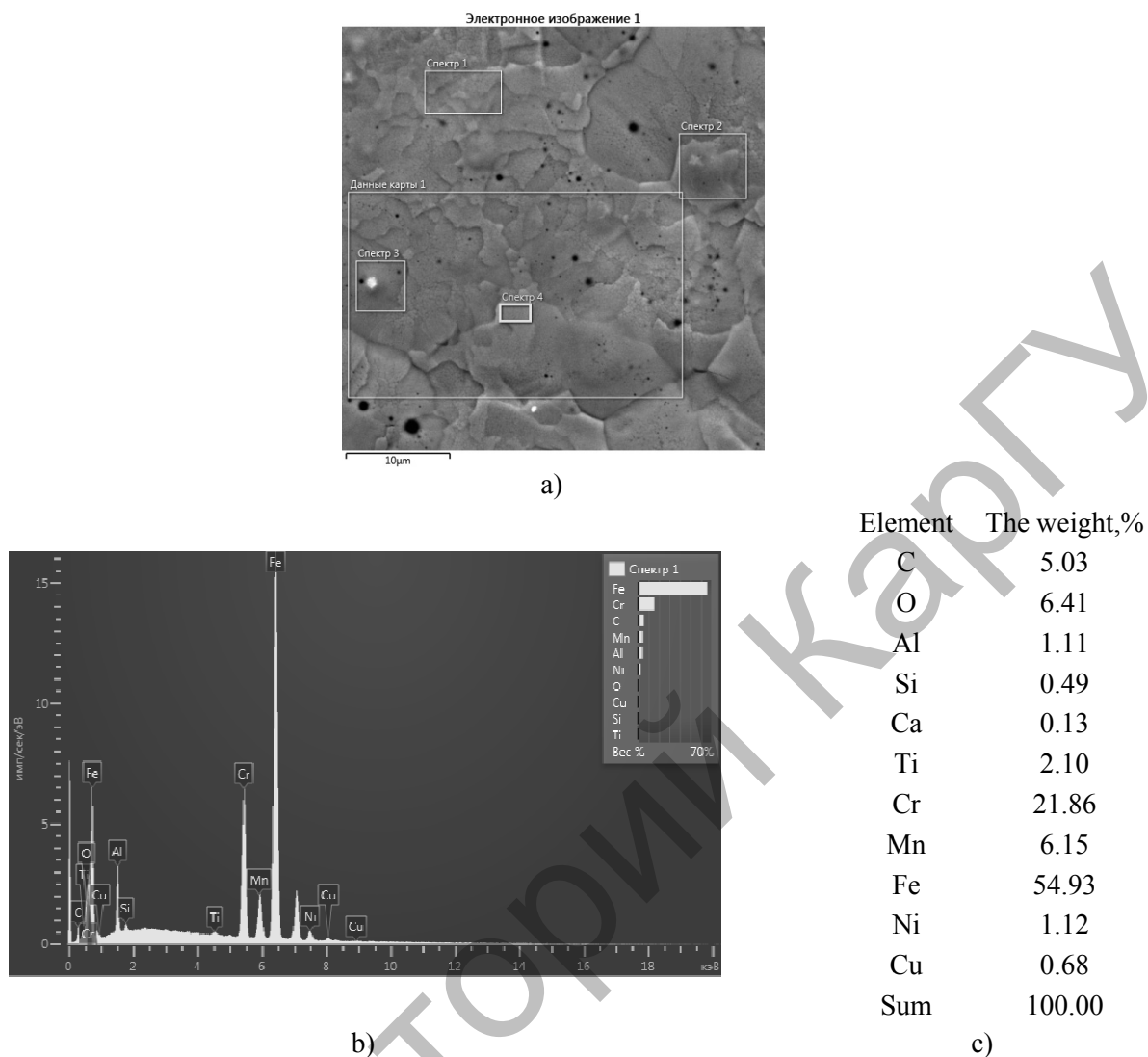


Figure 2. SEM image of the coating (a), XPS (b) and elemental composition (c) after irradiation with electrons

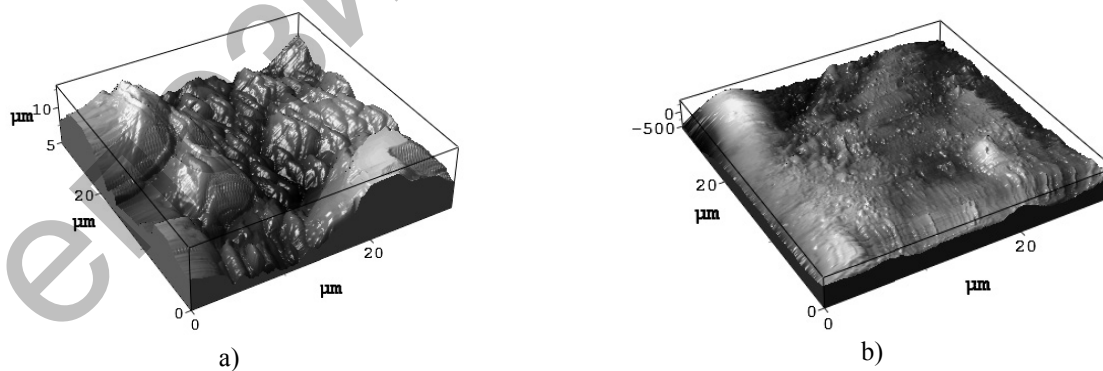


Figure 3. AFM-image of the coating before irradiation (a) and after irradiation with electrons (b)

Table 1 presents the microhardness values of the coating, and Table 2 shows the friction coefficients before and after electron irradiation.

Table 1

**Microhardness of the 12Cr18N10T + coating (Fe-Al) in argon medium at different deposition times (before and after irradiation)**

Microhardness, MPa	10 min	20 min	30 min	40 min
Before irradiation	416,2	428,6	464,2	273,5
After irradiation	502,4	534,6	542,4	564,7

Table 2

**Coefficients of friction before and after irradiation**

	Coefficients of friction
Before irradiation	0,426
After irradiation	0,130

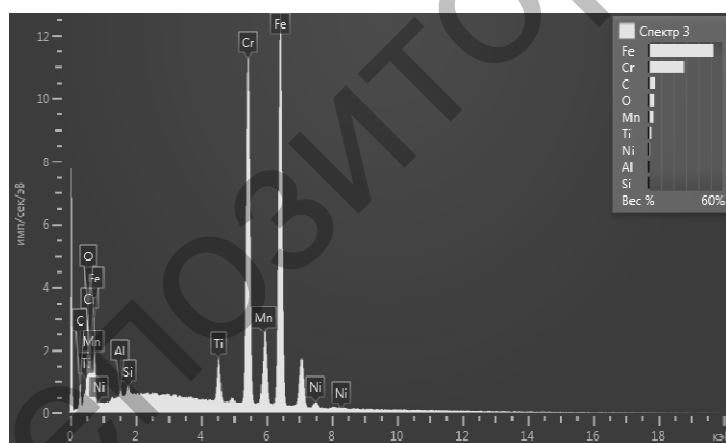
### Discussion

The electron microscopic images in Figure 1 and 2 show the presence of large inclusions of chromium, which are slightly dispersed after irradiation with electrons (Fig. 2, 4).

This is clearly seen from the analysis of spectrum 3 (Fig. 2a). The mapping of the elements along the surface of the coating showed that, with a step of 30  $\mu\text{m}$ , the distribution of all the elements in the samples is uniform, with the exception of chromium. The brightness, contrast and number of displayed points correspond to the mass content of the element in the specified area of the study.

Irradiation of the coating with electrons leads to fragmentation and dispersion of its structure. This is particularly clearly seen from the AFM measurements (Fig. 3), which shows a sharp decrease in the roughness of the coating. This effect was also observed for other coatings [8, 10, 11].

This leads to an increase in the microhardness of the coating (Table 1) and to a significant (more than 3-fold) decrease in the coefficient of friction (Table 2).



b)

Element	The weight, %
C	5.66
O	4.85
Al	0.32
Si	0.30
Ca	2.53
Ti	28.60
Cr	4.30
Mn	52.16
Fe	1.29
Ni	5.66
Cu	4.85
Sum	100.00

c)

Figure 4. Of the XPS (b) and elemental composition (c)

In the process of electronic treatment of coatings and during cooling, tensile states are formed in the latter, which can be sources of multiplication of dislocations throughout the volume of the deposited coating. The increase in the microhardness of the formed film observed in this process is a consequence of the dislocation hardening of the coating material.

Let us consider this question with the use of a model of a YaDC dislocation structure. Plastic deformation of crystals (and coatings) is accompanied by the formation of a deformation relief on their surface that reflects the process of localization of deformations in the crystal at the meso-, micro- and nanoscale lev-

els. The YaDC begins to form in the deformable crystal at the end of the second beginning of the third stage of the strain hardening curve of metals and ends at the end of the third stage (see [12] and the bibliography therein).

With further deformation, a fragmented dislocation structure-FDC (in the fourth and fifth stages of the strain hardening curve) is formed in the material. It is believed that YaDC is a process of self-organization of dislocations under conditions of multiple slip. For its occurrence, it is necessary to fulfill a certain criterion linking the coefficients of multiplication, immobilization, and annihilation of dislocations. The YaDC modeled in [12] is shown in Figure 5.

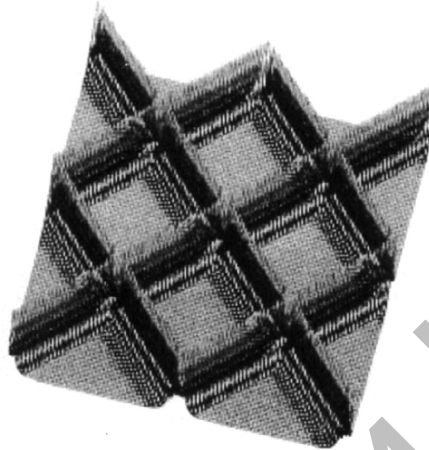


Figure 5. Cellular dislocation structure in the axonometric projection [12]

Let us consider qualitatively the micromechanism of friction of metals with coatings. It consists mainly of their two mechanisms: the destruction of contacts of contacting bodies and the intermolecular interaction of bodies. The sliding friction in the case of a molecular mechanism can be represented in the form [13]:

$$F_{mp} = \int_L \sigma dl \approx \sigma L, \quad (1)$$

where  $\sigma$  is the surface tension of a solid;  $L$  is the length of the path traversed by the slip.

Work  $A$  ( $J$ ), spent on the destruction of contacts (particles) is proportional to the newly formed surface of contacts (particles):

$$A = \gamma \Delta S = K_R D^2, \quad (2)$$

where  $\gamma$  time resistance to compression ( $Nm/m^2$ );  $\Delta S$  — area of newly formed surface ( $m^2$ );  $K_R$  — coefficient of proportionality ( $Nm/m^2$ );  $D$  is the characteristic dimension of the piece ( $m$ ).

Equation (2) corresponds to the Rittinger hypothesis (1867) [14]. If the energy is expended mainly on the deformation of the volume when the cubic particle is destroyed, then the work done is directly proportional to the change in its initial volume and is determined by the Kirpichev-Kick formula:

$$A = K \Delta V = K_K D^3, \quad (3)$$

where  $K$  and  $K_K$  are the proportionality coefficients ( $Nm/m^3$ );  $\Delta V$  — deformed volume ( $m^3$ ).

P.A. Rebinder (1941) combined both hypotheses and in this case the total work of destruction is equal to:

$$A = K_R D^2 + K_K D^3. \quad (4)$$

According to the Bond hypothesis (1950), the total work of destruction is proportional to the geometric mean between the volume and surface area of the particle:

$$A = K_B \sqrt{D^2 D^3} = K_B D^{2.5}. \quad (5)$$

All formulas (2)–(5) differ by the proportionality coefficients and the exponents of the particle diameter. However, it is rather difficult to determine experimentally these coefficients.

The work to break the contact in a pair of friction can be represented as the energy of dispersion [13]:

$$A = \sigma \cdot S = F_{mp} \cdot L. \quad (6)$$

All formulas (2)–(5) differ by the proportionality of the coefficients and the exponents of the particle diameter. However, it is rather difficult to determine experimentally these coefficients.

The work to break the contact in a pair of frictions can be represented as the energy of dispersion [13]:

$$A = \sigma \cdot L^2 + \sigma \cdot S = \sigma(L^2 + S). \quad (7)$$

From equation (7) it follows that the work of friction forces is determined, basically, by the surface tension of a solid body.

Surface tension is the main characteristic of processes associated with the participation of surfaces or interfaces. Surface tension determines the free energy (work) that must be expended to form a unit of surface area or phase separation.

The presence of surface tension is due to the fact that atoms on the surface of a liquid or solid have a greater potential energy than the atoms or ions inside them, therefore the surface energy is usually treated as an excess of energy per unit area.

Taking into account the surface tension at the phase interface leads to a significant change in the kinetics of the formation of coatings.

Taking into account that the formation of coatings is well described by the theory of dislocations, it is possible to choose two control parameters—the dislocation density and surface tension. In this case, the process of coating formation can be considered from the point of view of catastrophe theory. Catastrophes are called spasmodic changes in the form of a sudden response of the system to a smooth change in external conditions [15]. The theory of catastrophes can be used in technical problems for mathematical modeling of processes in which catastrophes can be observed. The growth of the coating film (a first-order phase transition) is also a catastrophe. For mathematical modeling, it is possible to consider three objects:

- purpose of functioning;
- one or two coordinates of the process of functioning;
- one or several control parameters, changing which we control the progress of the process.

In our consideration, the purpose of the functioning is the formation of the coating, and by the coordinate of this process we take the rate of dissipation of the free energy  $F$ , due to the formation of a film of  $N \gg 1$  clusters or islands. As control parameters, we take the dislocation density  $\rho$  and the surface tension  $\sigma$ .

For one coordinate and two control parameters in catastrophe theory, there is only one standard, canonical dependence for recording the dependence of the goal function:

$$\dot{F}(N) = 0,25N^4 - 0,5\rho N^2 - \sigma N, \quad (8)$$

where  $F(N)$  is the potential function, which is the energy of formation of  $N$  clusters.

A catastrophe having such a potential function is called a catastrophe of the «assembly» type [15]. The assembly has in the subcritical region one stable equilibrium state (one pit of the potential function), and in the supercritical region there are two stable and one unstable equilibrium states (ie, two pits separated by a hill).

The equation (8) specifies a static model for the formation of the coating. For us, the dependence of the rate of dissipation of free energy on the surface tension  $\sigma$  is important. It follows from equation (8) that the greater the surface tension, the lower the dissipation rate of the free energy. This means that most of the energy supplied goes to the formation of the coating. In this case, as a rule, cellular nanostructures with increased hardness are formed.

We shall use the general thermodynamic approach described in [15]. In this paper, on the basis of nonequilibrium quantum thermodynamics, an expression is obtained for the function  $\Phi$  of the response of the subsystem to an external action, which has the form:

$$\Phi = \frac{kT}{C} \cdot \frac{A}{G^0} \cdot \bar{N}, \quad (9)$$

where  $A$  is the work (energy) of the external field;  $T$  is the temperature;  $k$  is the Boltzmann constant;  $G^0$  is the Gibbs potential of a massive sample;  $\bar{N}$  the average number of elementary excitation carriers;  $C$  is a constant.

In the case of metals, the Gibbs potential coincides with the chemical potential ( $G^0 = \mu$ ). If the friction coefficient is taken as the response function, then we get:

$$k_{mp} = C \cdot T \cdot \frac{A}{\mu} \cdot \bar{N}. \quad (10)$$

For pure metals, the chemical potential coincides with the Fermi energy.

In Table 3, these values are given for some metals [16].

Fermi energy of some metals [16]

Metall	Fermi energy, eV	Metall	Fermi energy, eV
Cu	7,00	Zn	9,39
Ag	5,48	Al	11,63
Au	5,51	Pb	9,37
Mg	7,13	Sn	10,03
Sr	3,95	Ca	4,68

From equation (10) it follows that the coefficient of dry friction is the smaller, the greater the chemical potential (the Fermi energy).

Another important property of coatings is their heat resistance.

Heat resistance is the ability of metals and alloys to resist gas corrosion at high temperatures for a long time.

If the product works in an oxidizing atmosphere at a temperature of (500-550) °C without heavy loads, it is sufficient that they are only heat resistant (for example, parts of heating furnaces).

To improve the heat resistance, steel elements are introduced into the steel composition, which form oxides with a dense structure of the crystal lattice (chromium, silicon, aluminum). High-temperature resistance is possessed by the silchromes, nickel-based alloys, nichrome, steel 08X17T, 36X18H25C2, 15X6CЮ.

The destruction of metals in the supply of thermal energy is accompanied by the accumulation of thermoelastic stresses, which lead to an increase in the density of dislocations, various defects (dilatonons, frustrons, etc.).

If we take the heat resistance as the response function in formula (9), then we get:

$$\zeta = C \cdot \sigma. \quad (11)$$

Since, as noted above, the surface tension of the metal is proportional to its melting point, it follows that the heat resistance, in the first place, depends on the melting point of the metal. The higher the melting point of a metal, the higher its recrystallization temperature. Between the named temperatures, there is the following relationship:

$$T_p = K \cdot T_{na}, \quad (12)$$

where  $K = 0.3 \dots 0.4$  for technically pure metals,  $K = 0.6 \dots 0.8$  for alloys.

The surface energy of the coatings studied, as measured by the method of [17], is:  $\sigma = 1.684 \text{ J / m}^2$  (before irradiation) and  $\sigma = 1.980 \text{ J / m}^2$  (after irradiation).

Since the work to destroy the coating  $A = \sigma \cdot S$  ( $S$  is the coverage area), the results presented above show an increase in wear resistance after irradiation with an electron beam.

The melting temperature of the coating can be estimated from the formula proposed in [18]:

$$\dot{O}_m = 1,4 \cdot 10^3 \cdot \sigma.$$

Then we get:  $T_m = 2537.6^\circ\text{C}$  (before irradiation) and  $T_m = 2772.0^\circ\text{C}$  (after irradiation). Since the melting point of the coating characterizes its heat resistance, it follows that irradiation with electrons leads to an increase in the heat resistance of the coating.

### Conclusion

Irradiation with electrons leads to dispersion of the structure of the coating and changes in physico-mechanical properties.

Taking into account the surface tension at the phase interface leads to a significant change in the kinetics of the formation of coatings. Since the formation of coatings is well described by the dislocation theory, it is possible to choose two control parameters—the dislocation density and the surface tension. In this case, the process of coating formation can be considered from the point of view of catastrophe theory.

The greater the surface tension, the lower the dissipation rate of the free energy. This means that most of the energy supplied goes to the formation of the coating. In this case, as a rule, cellular nanostructures with increased hardness are formed.

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С.А. Гученко, Е.Н. Еремин, В.М. Юров, В.Ч. Лауринас, С.С. Қасымов, О.Н. Завацкая

## Плазма жамылғыларының құрылымы мен қасиеттеріне жоғарығымды шоқтың әсері

Мақалада электрондық сәулеленудің плазмалық жабындардың құрылымы мен қасиеттеріне әсері зерттелді. Қаптамалар Fe-Al және 12X18H10T катодтарын вакуумдық бұрку арқылы алынды. Электрондардың сәулеленуіне дейінгі және кейінгі микроқұрылымы, микроқаттылығы және үйкеліс коэффициенттері зерттелді. Сәулеленгеннен кейін жабындардың барлық сипаттамалары өзгеріске ұшырады. Интерфейс бетіндегі кернеуді ескере отырып, қаптамалардың қалыптасуының кинетикасының айтарлықтай өзгерісі байқалғаны тіркелді. Беттік кернеу неғұрлым көп болса, соғұрлым еркін энергияның диссипация жылдамдығы төмендейді. Бұл жеткізілетін энергияның басым бөлігі жабынның пайда болуына байланысты. Осы жағдайда қаттылық дәрежесі жоғарыұялы наноқұрылымдар пайда болады. Анықтау және бағалау әдістері арқылы плазмалық жабындарды еруі және қайта кристалдану орындалды. Азот орта шеңберінде жабындар үшін жоғары аргон алынған, ТЭМ-температура балку жабындылар құрылды. Ол 12X18H10T+Fe-Al және аргон астында дайындалған 12X18H10T+Cu, -Lea BO балку температурасы жоғары және қайта кристалдану бар

жабын 12X18H10T+Al деп эксперименттік түрде көрсетілген. Бұл соңғы жасалған бөлшектер ыстыққа төзімді жабын ретінде қолданылуы мүмкін екенін білдіреді. Азот ортада алынған жабындардың беттік керілуі, аргондағы алынған жабындарға қарағанда, төмен екенін көрсетті. Ол беттік энергия жабындарды төмендеуіне нитридтік фазалар нәтижелерін қалыптастыруын дәлелдейді. Жабыны үлкен бетінің шиеленісі жоғары коррозияға төзімділігі деп көрсетілген. Зерттелген жабындардың көпшілігінің коррозияға төзімді, дат басуға төзімділігі болаттан кем емес екендігі анықталды.

*Кілт сөздер:* плазма жабыны, электронды сәулелену, бетінің энергиясы, микроқаттылық, үйкеліс, тозуға төзімділік, бұзылу, жылуға төзімділігі.

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## Влияние сильноточного пучка на структуру и свойства плазменных покрытий

В работе исследовано влияние облучения электронами на структуру и свойства плазменных покрытий. Покрытия получены при вакуумном распылении катодов Fe-Al и 12X18H10T. Исследованы микроструктура, микротвердость и коэффициенты трения до и после облучения электронами. После облучения изменяются все характеристики покрытий. Учет поверхностного натяжения на границе раздела фаз приводит к существенному изменению кинетики формирования покрытий. Чем больше поверхностное натяжение, тем меньше скорость диссипации свободной энергии. Это означает, что большая часть подводимой энергии идет на образование покрытия. При этом, как правило, формируются ячеистые наноструктуры, обладающие повышенной твердостью. Предложены методы определения и осуществлена оценка температур плавления и рекристаллизации плазменных покрытий. Установлено, что температура плавления покрытий, полученных в среде аргона, выше, чем для покрытий, полученных в среде азота. Экспериментально показано, что покрытия 12X18H10T+Al, 12X18H10T+Fe-Al и 12X18H10T+Cu, полученные в среде аргона, имеют более высокие температуры плавления и рекристаллизации. Это означает, что последние могут быть использованы как жаростойкие покрытия на стальные детали. Показано, что поверхностное натяжение покрытий, полученных в среде азота, меньше, чем для покрытий, полученных в среде аргона. Отсюда следует, что образование нитридных фаз приводит к уменьшению поверхностной энергии покрытий. Доказано, что чем больше поверхностное натяжение покрытия, тем выше его коррозионная стойкость. Установлено, что коррозионная стойкость большей части исследованных покрытий не уступает наиболее коррозионно-стойким сталям.

*Ключевые слова:* плазменное покрытие, облучение электронами, поверхностная энергия, микротвердость, трение, износостойкость, разрушение, жаростойкость.

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