

V.Ch.Laurinas, S.A.Guchenko, O.N.Zavatskaya

*Ye.A.Buketov Karaganda State University  
(E-mail: vitas-laurinas@rambler.ru)*

## Structure and properties of multiphase ionic-plasma coverings

In work results of research of process of formation of ionic-plasma multiphase coverings are discussed. It is shown, that types of structures arising thus are not so diverse, as, for example, at crystallisation of alloys. Basically the globular structure and, less often, not closed and cellular structures is observed. It is shown, that physical properties can essentially differ depending on element structure of coverings. Properties of coverings Cr–Mn–Si–Cu–Fe–Al +Ti, received in the environment of argon and nitrogen, after a laser irradiation change in the various image. It is connected with features of behaviour of nitrides of the titan and chrome at influence of laser radiation. Properties of coverings Fe–Al after a laser irradiation practically do not change. It is connected with features of structure of these coverings which represents not closed structure.

*Key words:* plasma, a covering, microhardness, a friction, a microstructure, ionic irradiation, a laser irradiation.

### *Introduction*

The first cutting plates with a covering have appeared in the foreign market in 1968 when the Swedish firm «Sandvik Koromant» had been developed and introduced in manufacture a method of sedimentation of carbide of the titan on the tool from firm alloys. In 1971 (USA) have been received by firm Teledyne Firth Sterlig of a covering from nitride of the titan. At the Moscow institute of a steel and alloys in 1971 the method of sedimentation of a covering from niobium carbide has been developed. The next years researchers of many countries (the USA, France, Japan, the Great Britain, Germany, etc.) were developed technologies of drawing of various coverings of various appointment. There was a big number of the works devoted to various aspects of technology of drawing of coverings [1–11].

It has appeared, that properties of coverings are influenced by a considerable quantity of factors: structure of sprayed cathodes and their physical and chemical properties; properties of a substrate on which there is a material sedimentation; technology factors — an arch current, pressure of reactionary gas, substrate temperature, size of basic pressure etc. [1, 12–15].

A variety of influencing factors has generated a considerable quantity of processing methods of formation of coverings [16–19]. Last years the greatest popularity was received by methods of sedimentation of multiphase materials with high entropy [20].

In the present work generalisation of the experimental results received by us is spent, the part from which is published in works [21–30].

### *Role of a superficial tension in formation of ionic-plasma coverings*

Experimental definition of a superficial tension of solid states is complicated by that their molecules (atoms) are deprived possibility freely to move. The exception makes a plastic current of metals at the temperatures close to a melting point when mobility of atoms on a surface becomes appreciable.

In works [31–32] new methods of experimental definition of a superficial tension of solid states are offered. These methods are based on measurement of the dimensional effects connected with change of some physical properties of a small particle or a thin film at reduction of their size. Methods of definition of a superficial tension of the besieged coverings are based on dimensional effects, offered by us in work [33] also.

In works [26–28, 34–38] we in detail consider results of measurement of a superficial tension of nitrides of various metals, composite coverings. Here we will stop for roles of a superficial tension in formation of structure of ionic-plasma coverings.

This role is especially visually shown at formation of coverings in multiphase streams. In drawings 1 and 2 it is shown, how the covering structure changes at simultaneous dispersion of the multiphase cathode and the titan in the environment of nitrogen.

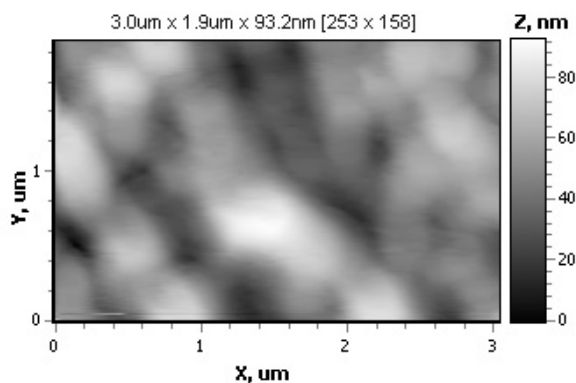


Figure 1. The image of globular structures of covering Cr–Mn–Si–Cu–Fe–Al, received on a nuclear power microscope

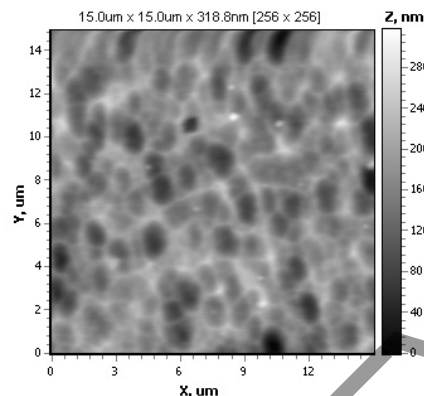


Figure 2. The image of cellular structure of a covering Cr–Mn–Si–Cu–Fe–Al + Ti in the environment of the nitrogen, received on a nuclear power microscope

*Influence of technological parametres on structure of ionic-plasma coverings*

Direct influence on structure and physical properties of the coverings received by a method of ionic-plasma sedimentation, render following parametres: pressure of reactionary gas in the working chamber; basis potential; a current of the category of an arch; properties of a material of the cathode; substrate temperature. In figures 3, as an example, electron-microscopic images of composite coverings are shown at temperatures of a substrate 350 and 450 °C. From figure 3 it is visible, that at temperature 450 °C there is a coagulation of a zinc phase in larger fragments, and other (most part) becomes more uniform.

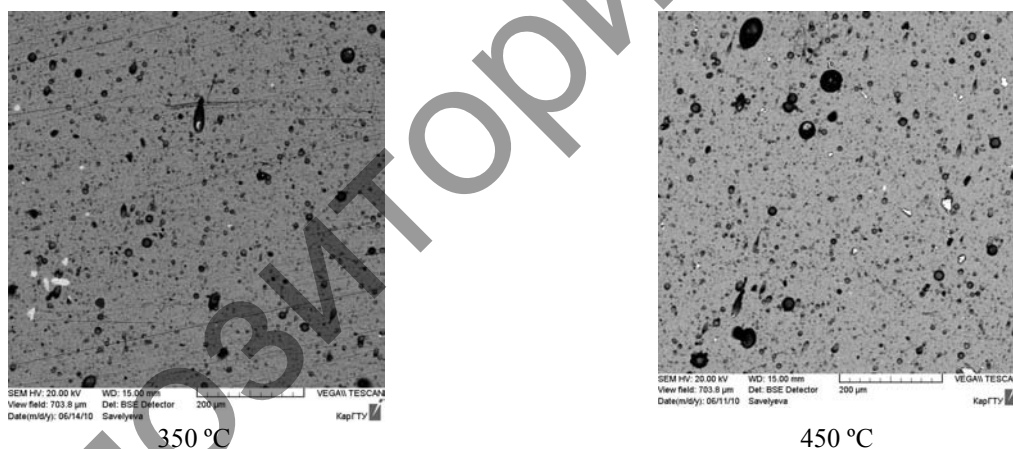


Figure 3. Electron-microscopic image of a covering Zn–Al

The optimum temperature of a substrate for all composite coverings has appeared equal nearby 400 °C.

The increase in a current of the category of an arch leads to increase in a thickness of a covering, however at current increase from above 130 A perfection of structure decreases and sharply the quantity of a drop phase which is the reason of decrease in durability of coupling of a substrate with a covering raises. At low power of the category (an arch current <20–30 A) because of reduction of factor of ionisation of plasma in a film neutral particles of reactionary gas and the cathode «are immured», that promotes increase of concentration of defects of a covering.

We had been investigated dependence of properties of composite coverings on pressure of nitrogen in the working chamber; thus force of a current, basic pressure, a material of the cathode, a fastening and heat-conducting path condition, time of processes of clearing and a dusting remained constants. In table 1 results on microhardness are presented.

Table 1

Dependence of microhardness of a covering on pressure of gas in the chamber

Residual pressure of gas in the chamber	Viker's microhardness, HV			
	Al-Fe	Zn-Cu-Al	Zn-Al	Al-Fe
$10^{-8}$	0,662			0509
$10^{-7}$	0,66			0,512
$10^{-6}$	0,60	0,573	0,569	0,514
$10^{-5}$	0,61	0,600	0,520	0,470

At pressure of nitrogen  $P = 0,058-0,81$  Pa the small dense structure close to стехиометрическому to structure which is characterised optimum, from the point of view of metal properties, is formed by a parity of metal and ionic components of communication. Thus the maintenance of a drop phase decreases, and the quantity of defects increases. At the further increase of pressure the big number of free ions leads sharp defects.

Having analysed results of research, it is possible to draw a conclusion that the samples received at pressure of nitrogen  $P = 0,081-0,81$  Pa, have in regular most intervals distributed small dense structure, the minimum maintenance of a drop phase and the greatest values of microhardness.

#### *Influence of an ionic irradiation on structure and property of multiphase coverings*

The basic influence of ionic bombardment on properties of coverings is carried out at a stage of their origin owing to a relaxation of pressure in the field of ionic blow and reorganisation of crystal structure. Thus on a surface dot defects which are the active centres of adsorption are formed. The important role also plays, mobility of atoms on a surface (superficial diffusion) which amplifies at low-energy bombardment of a growing film by ions of inert gas. To increase quantity of formed dot defects it is possible or increase in energy of a stream of ions, or increase in density of a current of ions. Simultaneously with formation of defects there is also their return process of recombination which reduces concentration of defects. As a result of these two processes the equilibrium quantity of the centres of formation of germs which it is possible to influence is established, changing parametres of an ionic irradiation.

In figure 4–9 are shown AFM images of a surface of composite coverings before and after an irradiation by argon ions.

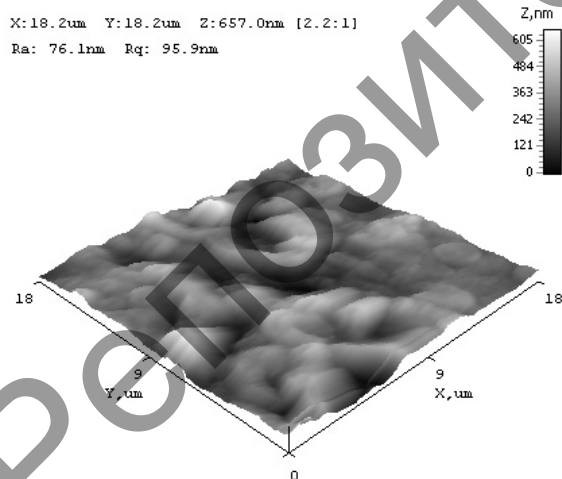


Figure 4. AFM the image of a surface of covering Fe-Al (to an irradiation)

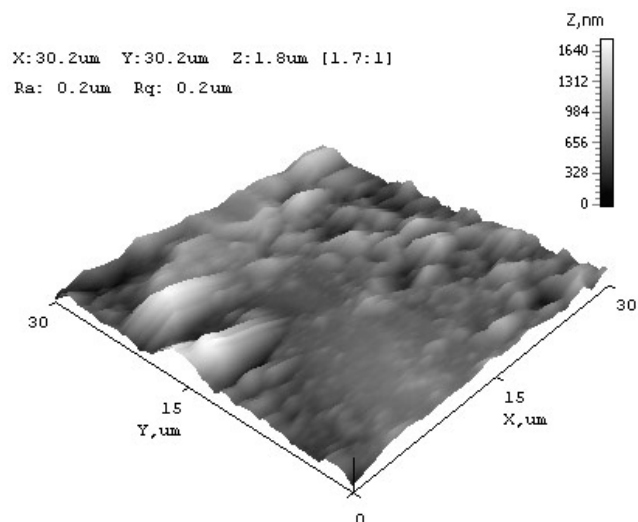


Figure 5. AFM the image of a surface of covering Fe-Al (after an irradiation)

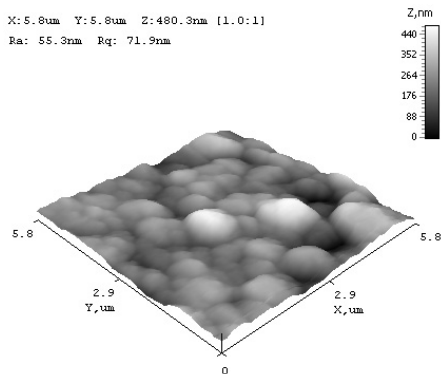


Figure 6. AFM the image of a surface of covering Zn–Al (to an irradiation)

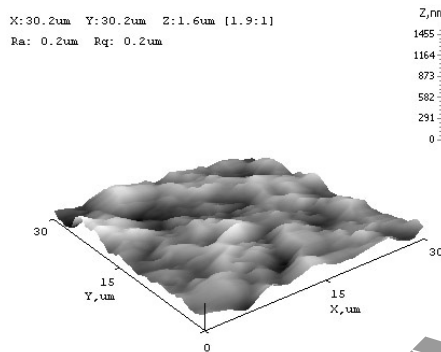


Figure 7. AFM the image of a surface of covering Zn–Al (after an irradiation)

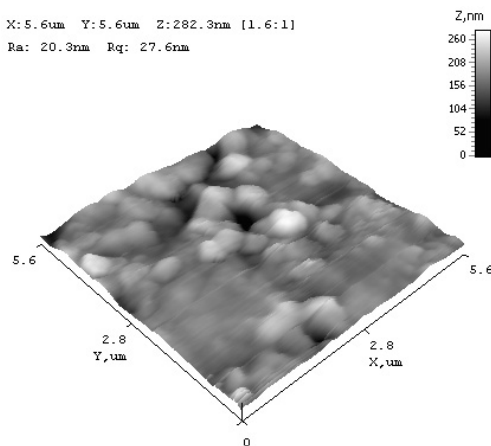


Figure 8. AFM the image of a surface of covering Mn–Fe–Cu–Al (to an irradiation)

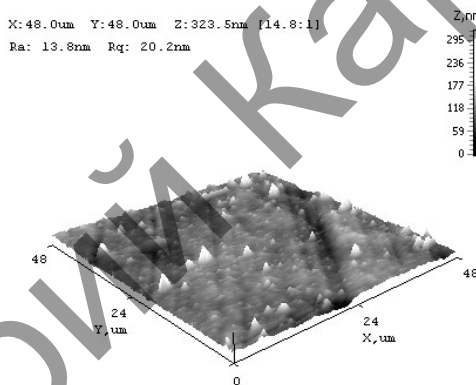


Figure 9. AFM the image of a surface of covering Mn–Fe–Cu–Al (after an irradiation)

From figures 4–9 follows, that the ionic irradiation influences structure of coverings. The exception makes covering Zn–Al which has appeared proof to an irradiation. With the received result correlates also value of the Jung’s module measured before and after an irradiation. Such behaviour of covering Zn–Al communicates us with its strongly pronounced of globular structure. Presence of such system of «spheres» leads to elastic dispersion of ions of argon so local deformation appears insignificant. It finds the reflexion in behaviour of the Jung’s module whom (tab. 2) and parametre of a roughness (tab. 3) does not change in the course of an irradiation The structure of covering Fe–Al sharply differs from structure of covering Zn–Al. In this case we observe not closed structures (fig. 4). The roughness of this covering after an irradiation increases almost three times while the Jung’s module decreases in as much time. The same behaviour of the Jung’s module is observed and for covering Mn–Fe–Cu–Al.

Table 2

Value of the Jung’s module

Covering	After an irradiation	To an irradiation
	$E$ , GPa	$E$ , GPa
Cr–Mn–Si–Cu–Fe–Al	1,0	0,5
Zn–Al	0,6	0,6
Mn–Fe–Cu–Al	0,1	0,3
Fe–Al	0,2	0,7

Table 3

The Roughness of covering  $R_a$ 

Covering	After an irradiation	To an irradiation
	$R_a$ , nm	$R_a$ , nm
Cr-Mn-Si-Cu-Fe-Al	22,26	13,34
Zn-Al	76,39	78,0
Mn-Fe-Cu-Al	23,08	29,89
Fe-Al	147,06	56,14

*Properties of coverings at simultaneous dispersion of various cathodes*

The basic idea used in the present work consists in the following: for generation of multicomponent streams of ions of the various metals besieged on a substrate, we use the multiphase composite cathode on one gun of vacuum installation and the single-phase cathode from the titan on other gun. In the course of simultaneous dispersion of various cathodes ions of metals mix up in plasma and, after sedimentation, form a covering. Such approach is «compelled» as reception of multiphase composite cathodes with the set number the component represents a challenge connected with reception of multicomponent alloys, thermodynamic properties which component can differ considerably. Results of measurements of microhardness of composite coverings are presented to tab. 4.

Table 4

## Microhardness of samples with the coverings in argon and nitrogen

Covering	Quantity of tests	Microhardness, HRC		
		The sample № 1 (15 min) in nitrogen	The sample № 2 (30 min) in nitrogen	The sample № 2 (30 min) in argon
Zn-Al + Ti	1	216,2	305,3	217,5
	2	232,0	361,7	226,8
	3	224,1	251,4	211,8
	4	285,9	206,0	211,6
	5	226,3	235,6	217,8
	Average value	236,9	272,0	217,1
Fe-Al + Ti	1	185,7	264,1	331,8
	2	206,2	257,2	334,7
	3	208,5	263,9	339,2
	4	226,6	278,4	355,9
	5	200,5	286,5	319,7
	Average value	205,5	270,0	336,2
Zn-Cu-Al + Ti	1	279,0	248,6	222,0
	2	207,6	252,3	217,7
	3	291,2	243,2	221,2
	4	185,7	252,5	221,8
	5	191,1	249,3	222,0
	Average value	230,9	249,2	220,9
Cr-Mn-Si- Cu-Fe-Al + Ti	1	298,4	257,8	227,8
	2	266,0	244,6	225,7
	3	286,4	246,0	229,6
	4	270,8	244,3	220,2
	5	249,6	253,6	232,4
	Average value	274,2	247,2	227,1

As microhardness in the environment of nitrogen more than in the inert environment of argon at the expense of formation of a phase of nitride of the titan follows from tables 4, in all cases.

For a long time it is known, that iron impurity make the big impact on quality of received aluminium, that is with increase in the maintenance of these impurity corrosion firmness worsens, plasticity decreases, durability of aluminium raises.

Clearing of aluminium of iron filtering and in other ways is labour-consuming and expensive procedure. Therefore as an alternative variant of use of technical aluminium with the raised maintenance of iron drawing of coverings from it is at simultaneous introduction of the titan in the environment of nitrogen. Thus negative influence of iron sharply decreases for properties of aluminium.

Advantage of the offered approach is that high adaptability to manufacture of process which is difficult for carrying out traditional methods is thus reached, at good technical and consumer properties of coverings.

*Influence of laser radiation on properties of multiphase coverings*

Laser radiation possesses high energy, monochromaticity and a narrow orientation. It allows to concentrate energy with density of capacity from extremely small to  $10^{18}$  W/cm<sup>2</sup>. Laser radiation at influence on a surface of a processed material allows quickly and is dosed out to transfer this energy. Possibility of such intensive supply of energy to a material causes locality of other physical processes thermal and connected with them. Updating of properties of materials by laser radiation on a physical essence is reduced to local thermal influence. Therefore it is defined thermal by material parametres, density of capacity and time of influence of radiation, as a whole quantity of the specific energy absorbed by a material, and speed of its dispersion. The structure of the layer strengthened by laser radiation, is characterised by the big hardness lowered by chemical activity, mechanical characteristics, thermal stability, size and character of distribution of residual pressure [39–42] vary.

On figures 10–11 the microstructure of coverings before laser processing is shown.

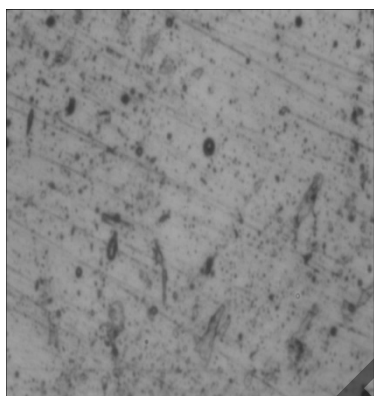


Figure 10. Covering microstructure Cr–Mn–Si–Cu–Fe–Al +Ti in the gas environment argon to a laser irradiation

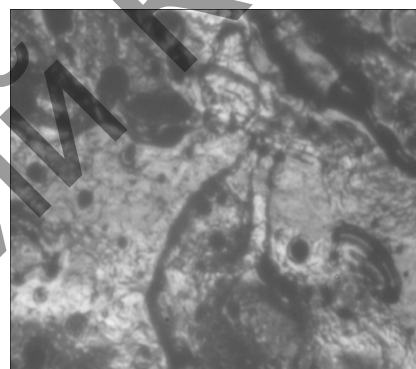


Figure 11. Covering microstructure Cr–Mn–Si–Cu–Fe–Al +Ti in gas to the environment of argon after a laser irradiation

In table 5 factors of a friction of samples Cr–Mn–Si–Cu–Fe–Al + Ti in the environment of argon without laser processing and after laser processing are resulted. Definition of factors of a friction was made in steam: a covering — a covering; a covering — aluminium; a covering — copper.

Table 5

**Factors of a friction of covering Cr–Mn–Si–Cu–Fe–Al + Ti, received in the environment of argon**

The sample	Factors of a friction		
	a covering — a covering	a covering — aluminium	a covering — copper
Cr–Mn–Si–Cu–Fe–Al + Ti to a laser irradiation	0,413	0,302	0,269
Cr–Mn–Si–Cu–Fe–Al + Ti after a laser irradiation	0,274	0,265	0,264

In table 6 values of microhardness on Vickers (HV) samples Cr–Mn–Si–Cu–Fe–Al + Ti in the environment of argon without laser processing and after laser processing are resulted.

Table 6

**Microhardness of covering Cr–Mn–Si–Cu–Fe–Al + Ti, received in the environment of argon**

The sample	Test loading, kg	Microhardness, HV
Cr–Mn–Si–Cu–Fe–Al + Ti to a laser irradiation	0,01	190,5
Cr–Mn–Si–Cu–Fe–Al + Ti after a laser irradiation	0,01	328,0

On figures 12–13 the microstructure of covering Cr–Mn–Si–Cu–Fe–Al + Ti, the nitrogen received in atmosphere is shown.

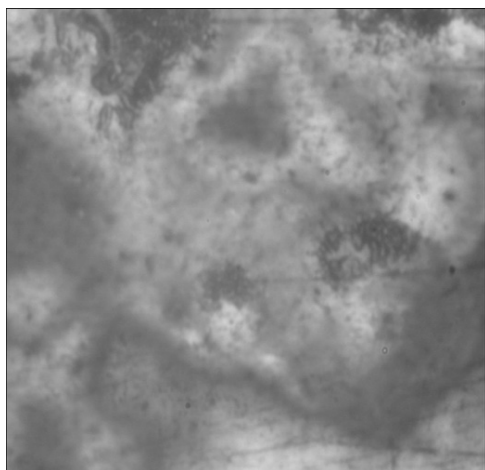


Figure 12. The microstructure of covering Cr–Mn–Si–Cu–Fe–Al + Ti in the gas environment of nitrogen to a laser irradiation

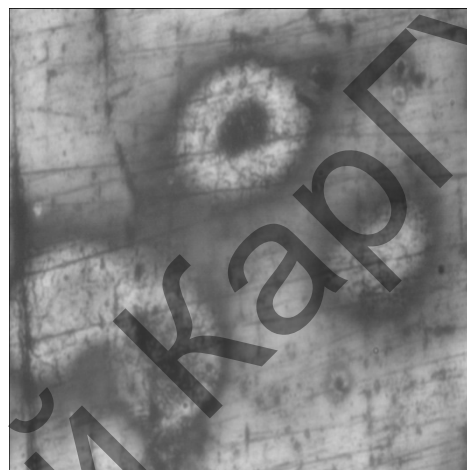


Figure 13. The microstructure of covering Cr–Mn–Si–Cu–Fe–Al + Ti in the gas environment of nitrogen after a laser irradiation

In table 7 factors of a friction of samples Cr–Mn–Si–Cu–Fe–Al + Ti in the environment of argon without laser processing and after laser processing are resulted.

Table 7

**Factors of a friction of covering Cr–Mn–Si–Cu–Fe–Al + Ti, received in the environment of nitrogen**

The sample	Factors of a friction		
	a covering — a covering		a covering — a covering
Cr–Mn–Si–Cu–Fe–Al + Ti to a laser irradiation	0,243	0,304	0,431
Cr–Mn–Si–Cu–Fe–Al + Ti after a laser irradiation	0,229	0,370	0,320

In table 8 values of microhardness on Vickers (HV) samples Cr–Mn–Si–Cu–Fe–Al + Ti in the environment of argon without laser processing and after laser processing

Table 8

**Microhardness of covering Cr–Mn–Si–Cu–Fe–Al + Ti, received in the environment of nitrogen**

The sample	Test loading, kg	Microhardness, HV
Cr–Mn–Si–Cu–Fe–Al + Ti to a laser irradiation	0,025	804,4
Cr–Mn–Si–Cu–Fe–Al + Ti after a laser irradiation	0,025	365,5

As follows from tables 5 and 6 for covering Cr–Mn–Si–Cu–Fe–Al + Ti in the environment of argon the factor of a friction after laser processing decreases, and microhardness increases. We connect the first effect with reduction of a roughness of a covering at its fusion by a laser beam. The second effect is caused by formation of structure of a covering with dispositions at sharp heating-cooling that is clearly visible from comparison figures 10 and 11.

At drawing of coverings Cr–Mn–Si–Cu–Fe–Al + Ti in the environment of nitrogen in the last the areas containing nitrides of the titan and chrome, and the maintenance both component approximately equally are formed. The size of particles of nitride of the titan and chrome according to electronic microscopy makes 100–150 nanometers. Microcrystals of nitrides of the titan and chrome have primary orientation (presumably in a direction (200)), that is distinct from spherical symmetry of microcrystals of the pure titan. All it, along with cellular structure of a covering, leads to its high microhardness (table 8). After laser processing of covering Cr–Mn–Si–Cu–Fe–Al + Ti, received in the environment of nitrogen, the friction factor changes slightly, and microhardness decreases more than in 2 times (tab. 8). It means, that in this case we deal not about covering hardening, and with its holiday as it is observed in the tempered steels at heats. However the mechanism of loss of hardening in our case is distinct from the mechanism of holiday of a steel where last is caused by phase transition. One of the probable reasons of loss of hardening of covering Cr–Mn–Si–Cu–Fe–Al + Ti, received in the environment of nitrogen, is formation of a congestion of microcrystals of the titan and the chrome, clearly visible on fig. 11 both 13 and occurring at cooling of the fused covering after a laser irradiation.

### The conclusion

The results of research of process of formation of ionic-plasma multiphase coverings resulted in the present work show, that types of structures arising thus are not so diverse, as, for example, at crystallisation of alloys. Basically the structure and, less often, globular, not closed and cellular structures is observed. However, physical properties can essentially differ depending on element structure of coverings. As the size of a superficial tension (superficial energy) in most cases is size additive each element which is a part of a covering, brings the contribution to the general superficial energy. In case of simultaneous dispersion of multiphase cathodes coverings with the high entropy, possessing the ordered structure and the raised mechanical properties are formed. Thus, some overall picture appears: For reception of the superfirm structured coverings it is necessary to use multiphase (multielement) cathodes, it is desirable at their simultaneous dispersion. Certainly, it is impossible to forget and about optimisation technological parametres of process of sedimentation of coverings.

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В.Ч.Лауринас, С.А.Гученко, О.Н.Завацкая

## Көпфазалық ионды-плазмалы жабындының құрылымы және қасиеттері

Мақалада ионды-плазмалық жабулардың микроқатандығын зерттеу нәтижелері талданды. Жабуларды жасау үшін бір және көп камералы плазмалық ағындар қолданылды. Жабуларды жасағанда аргон және азот атмосферасы пайдаланылып, әр түрлі өзгеріске ұшырайтыны байқалған. Эксперименттік мәліметтер бойынша, жабулардың микроқатандығы үлгі бетінде квазипериодты өзгеретіні көрінеді. Жабулардың периодтық құрылымын қалыптастыруға арналған модель ұсынылды. Модель теориясының негізі, жабуларды қалыптастырған кезде буландырылатын ағындар компоненттерінің концентрациялық градиенті есебінен пайда болатын автотербелістік қозғалыстардың тууына негізделген.

В.Ч.Лауринас, С.А.Гученко, О.Н.Завацкая

## Структура и свойства многофазных ионно-плазменных покрытий

В работе обсуждаются результаты исследования процесса формирования ионно-плазменных многофазных покрытий. Показано, что типы возникающих при этом структур не столь многообразны, как, например, при кристаллизации сплавов. В основном наблюдается глобулярная структура и, реже, незамкнутые диссипативные и ячеистые структуры. Показано, что физические свойства могут существенно отличаться в зависимости от элементного состава покрытий. Авторами отмечено, что свойства покрытий Cr–Mn–Si–Cu–Fe–Al + Ti, полученные в среде аргона и азота, после лазерного облучения изменяются различным образом. Это связано с особенностями поведения нитридной фазы при воздействии лазерного излучения. Определено, что свойства покрытий Fe–Al после лазерного облучения практически не изменяются. Это связано с особенностями структуры этих покрытий, которая представляет собой незамкнутую структуру.

*Ключевые слова:* плазма, покрытие, микротвердость, трение, микроструктура, ионное облучение, лазерное облучение.

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