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Application of highly entropic coatings at simultaneous spraying of three cathodes in one cycle

In this work, we consider the application of highly entropic coatings while simultaneously sputtering three cathodes in a single cycle. The cathodes 12Cr15G9ND, Cu, Al were chosen as cathodes. The chemical composition of the cathodes was measured using a TESCAN MIRA 3 electron microscope. Using these cathodes, coatings were applied to polished samples of steel 45 in an NNV-6.6 I1 vacuum ion-plasma apparatus. The measurement was carried out using a MIRA 3 scanning electron microscope, an HVS-1000A microhardness tester, and a tribological research facility. SEM images show that with an increase of ×5000 or more, the droplet phase is clearly detected. The maximum size of the droplet phase reaches 12.0 μm with a variety of morphometric parameters varying, for example, in a geometry coefficient K from 0.8 to 1.0. Analysis of the coating at high magnifications (×7000 and ×20,000) without the droplet phase showed that the grain sizes of the coating are as follows: minimum — 0.23 microns, maximum — 0.65 microns (average of about 0.47 microns); while the coating structure is homogeneous. The analyzed type of structure refers to the zone of the so-called «competing texture», when a dense nanocrystalline structure is present in the lower region of the film, and a columnar structure is above it. The results of this study allow us to conclude that the simultaneous deposition of deposited cathodes of various metals (especially composite) in principle allows to obtain highly entropic coatings. Two points must be taken into account here: firstly, the number of atomized cathodes must be increased; secondly, it is necessary to take into account the values of the erosion coefficient for the cathodes used so that the atomized fluxes are equimolarly proportional. Measurements of coatings showed that the microhardness of Cu+Al+12Cr15G9ND is not inferior to ordinary steels, but of course it lags behind nanostructured coatings (30–50 GPa). The proposed coatings have antifriction properties of 3 or more times and can be used in tribological pairs.

Keywords: multilayer coatings, hardness, ductility, friction, wear resistance, nanostructure, microhardness, cathode, structure.

Introduction

High entropy alloys (wind farms) began to be investigated 15 years ago. Such alloys usually consist of five or more components contained in approximately equal (equimolar) proportions. To date, a little more than 200 wind farms have been studied [1–8]. This is a small number of alloys. In fact, in industry their number is more than 10,000 alloys used. Add to this the fact that the time when the first alloys appeared, is estimated at about 5 thousand years.

In this paper, we consider «The application of highly entropic coatings while simultaneously sputtering three cathodes in a single cycle». In this way, we wanted to show a new method for producing highly entropic coatings. The cathodes 12Cr15G9ND, Cu, Al were chosen as cathodes. The results of this study allow us to conclude that the simultaneous deposition of deposited cathodes of various metals in principle allows highly entropic coatings to be obtained.

This chapter shows for the first time that the wavelength of the studied coatings is of the order of 10^{-4} m, i.e. mass transfer rate is ~10^{-4} m/s. Since the mass transfer rate, for the diffusion coefficient we obtain the estimate $D \approx 10^{-8}$ m^2/s. This corresponds to the mode of low diffusion. The results obtained above fit into the model of macroscopic localization of plastic flow.

Methodology for the preparation of research objects

The 12Cr15G9ND, Cu, Al cathodes cut on a lathe for installing NNV-6.6I1 were polished and cleaned with Kalosha gasoline and wiped with alcohol. The chemical composition of the cathodes was measured using a TESCAN MIRA 3 electron microscope and is presented in Table 1.

The cathodes themselves with a diameter of 80 mm have the form as in Figure 1. Before filling the chamber with working gas (Ar), it was evacuated to a pressure of ~$5 \cdot 10^{-3}$ Pa, after which it was filled with argon to a working pressure of ~0.15 Pa. The pressure was maintained by a constant electromagnetic leakage. As a source of metal plasma, an electric arc evaporator based on an independent cold cathode arc discharge.

Table 1

The chemical composition of the cathodes 12Cr15G9ND, Cu, Al

The chemical composition in% steel 12Cr15G9ND									
C	Si	Mn	Ni	S	P	Cr	Cu	N_2	Fe
0.12	0.75	10.5	1.2	0.03	0.060	15.7	2.0	0.2	69.44
Chemical composition in % copper Cu									
Cu	Bi	O	Pb	other items					
99.90	0.0005	0.040	0.005	0.03					
Chemical composition in % aluminum									
Al	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	–
99.7	0.100	0.250	0.020	0.010	0.020	0.010	0.040	–	–

A bias voltage of 1000 V was applied to the samples under study and ion cleaning and heating to a temperature of 450 ºC were carried out for 20 minutes. Then, three cathodes with an arc current of 80 A were switched on simultaneously and a coating was applied for 20 minutes at a reference voltage of 150 V. After cooling the samples under vacuum for one hour, one sample was removed and the whole process was repeated again.

Figure 1. Three cathodes: aluminum (left), copper (in the middle) and 12Cr15G9ND (right)

Thus, 8 samples 12Cr15G9ND+Cu+Al were obtained in a gas medium of argon and nitrogen, the deposition time was 20, 40, 60, 80, 100, 120, 140, 160 minutes. For the deposition of coatings, polished disks of steel 45 with a diameter of 20 mm were used as substrates (Fig. 2*a*), which were simultaneously placed on the substrate holder in order to deposit the coating on different substrates under identical conditions.

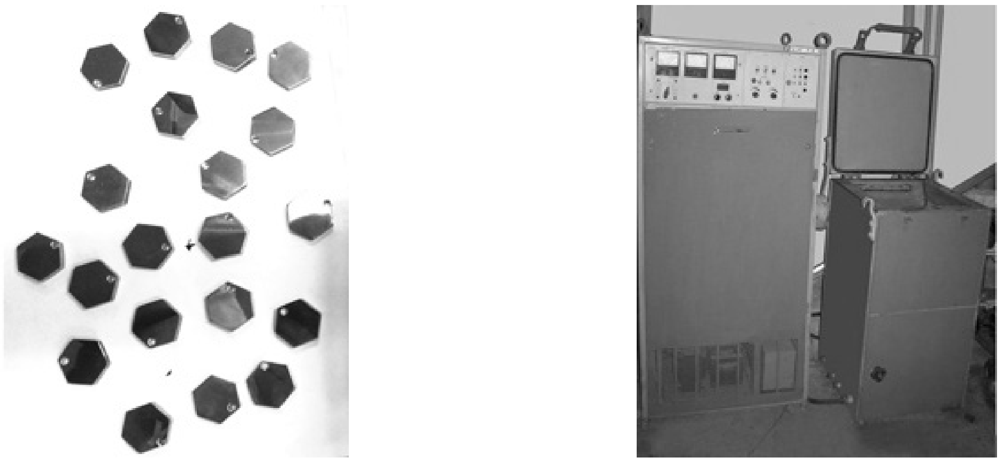
Polished discs were prepared as follows:

1. Grinding and polishing a sample on a MetaServ 250 grinding and polishing machine.
2. Sanding SiC with CarbiMet P180 abrasive paper with cold water feed.
3. Polishing with a circle (a fabric with a hard weave, without pile) and diamond paste with particles: 1) with a diameter of 9 microns, 2) with particles of 3 microns.

4. Circle polishing (soft porous chemically resistant fabric, without lint) using a polished suspension based on Al_2O_3 (particles with a diameter of 0.05 microns).

As a result, the specimen surface was obtained, the roughness parameters were studied using an NT-206 atomic force microscope (AFM) and, as an example, are shown in Table 2. The average roughness was ~13 nm.

Before deposition, the substrates were cleaned in an ultrasonic bath in a solution of acetone with ethanol (Fig. 2*b*).



a *b*

Figure 2. Polished disks made of steel 45 (*a*) and an ultrasonic cleaning bath (*b*)

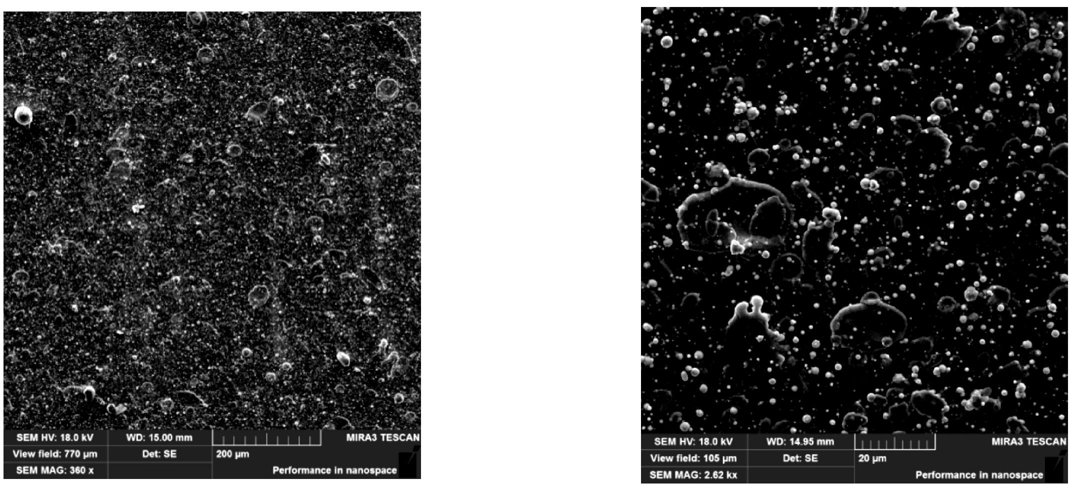
T a b l e 2

Roughness parameters and statistical characteristics of substrates

Substrate	Roughness R_a, nm	Dispersion R_q	Asymmetry R_{sk}	Excess R_{ku}
Steel 45–1	13.34	18.23	0.17	7.35
Steel 45–2	13.00	16.6	0.14	6.46
Steel 45–3	13.89	21.42	0.15	8.97

Electron microscopic examination of coatings

Electron microscopy was performed using a TESCAN MIRA 3 scanning electron microscope (SEM) (Fig. 3). The studies were carried out at an accelerating voltage of 20 kV and a working distance of about 15 mm.



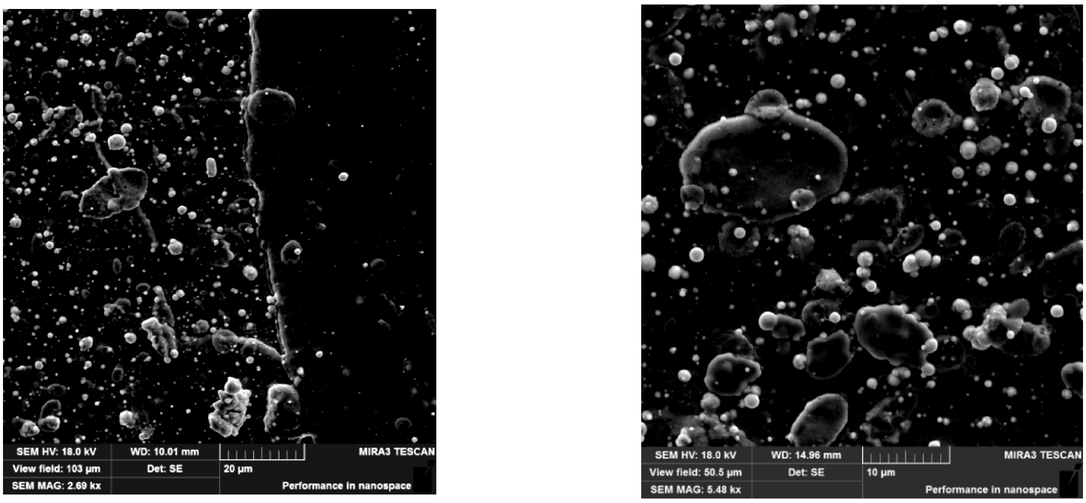


Figure 3. SEM-image of a coating in argon of different resolutions

With an increase of ×5000 or more, the droplet phase is clearly detected. The maximum size of the droplet phase reaches 12.0 μm with a variety of morphometric parameters varying, for example, in a geometry coefficient K from 0.8 to 1.0. Analysis of the coating at high magnifications (×7000 and ×20,000) without the droplet phase showed that the grain sizes of the coating are as follows: minimum — 0.23 microns, maximum — 0.65 microns (average about 0.47 microns); while the coating structure is homogeneous.

SEM-image of the coating in argon is shown in Figure 4.

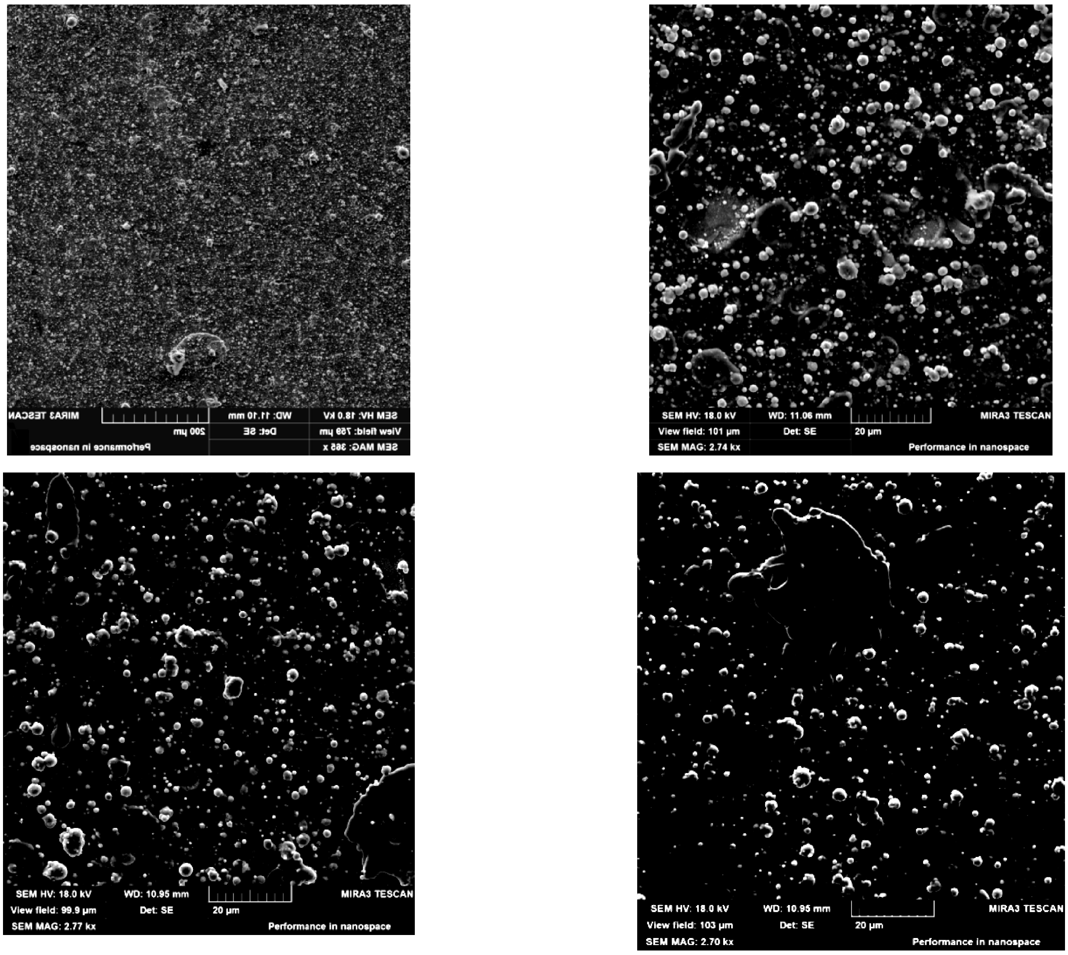


Figure 4. SEM-image of a coating in nitrogen of different resolutions

In both cases, a globular structure is observed when the concentration of additives varies, depending on the homological temperature. That is, most likely, the analyzed type of structure refers to the zone of the so-called «competing texture», when a dense nanocrystalline structure is present in the lower region of the film, and a columnar structure is above it.

X-ray fluorescence electron spectroscopy of coatings

X-ray fluorescence electron spectroscopy (XPS) of coatings for argon and nitrogen is shown in Figure 5 and 6. In argon and nitrogen, the XPS spectra of the coating are significantly different: iron in argon is ~40 weight. %, and in nitrogen ~22 weight. %, i.e. twice smaller; on the contrary, copper in argon is ~14 weight. %, and in nitrogen ~28 weight. %, i.e. twice as much. This is observed in all studied points. The XPS spectra of coatings in nitrogen contain on average: Cu ~28 wt. %, Fe ~18 wt. %, Al ~16 wt. %, Cr ~9 wt. %. High entropy alloys (wind farms) should, as a rule, consist of five or more basic elements with a concentration between 5 and 35 %. We got 4 main elements in the range of 5–35 weight. %, typical for wind farms, but in non-equimolar proportion.

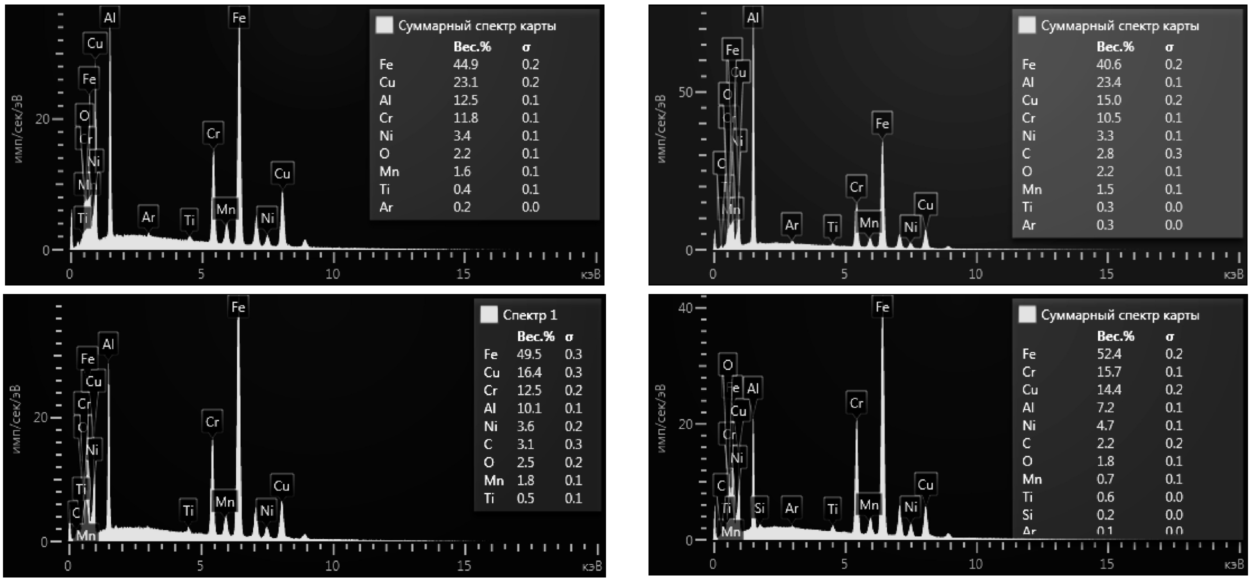


Figure 5. XPS of the coating 12X15G9ND + Cu + Al in argon at 4 points

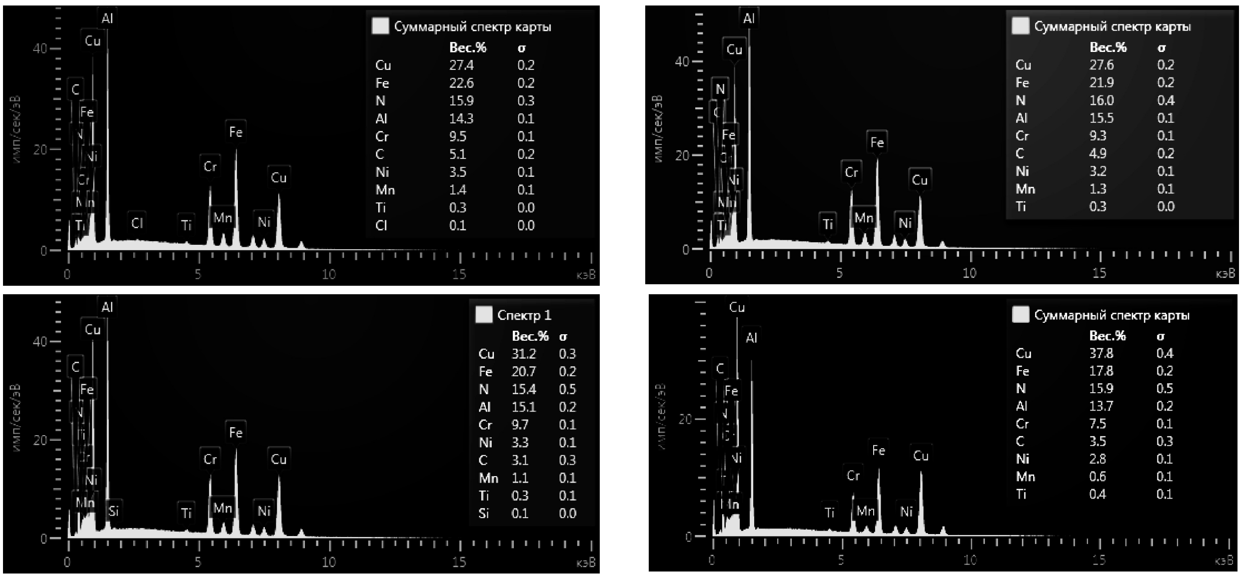


Figure 6. XPS of the coating 12X15G9ND + Cu + Al in nitrogen at 4 points

The results of this study allow us to conclude that the simultaneous deposition of deposited cathodes of various metals (especially composite) in principle allows to obtain highly entropic coatings. Two points must be taken into account here: firstly, the number of atomized cathodes must be increased; secondly, it is necessary to take into account the values of the erosion coefficient for the cathodes used (Table 3) [9], so that the sprayed flows are equimolarly proportional.

Table 3

The values of the coefficient of erosion for some materials [9]

Cathode material	T_m, K	Erosion coefficient, g/Kl
Cd	1038	$6.55 \cdot 10^{-4}$
Zn	1180	$6.2 \cdot 10^{-4}$
Mg	1380	$0.36 \cdot 10^{-4}$
Al	2740	$1.2 \cdot 10^{-4}$
Cu	2668	$1.15 \cdot 10^{-4}$
Cr	2956	$0.4 \cdot 10^{-4}$
Ni	3003	$1.0 \cdot 10^{-4}$
Fe	3343	$0.73 \cdot 10^{-4}$
Ti	3558	$0.52 \cdot 10^{-4}$
C	4173	$0.17 \cdot 10^{-4}$
Mo	4923	$0.47 \cdot 10^{-4}$
W	5973	$0.62 \cdot 10^{-4}$

It is interesting to find out what hardness and tribological characteristics the obtained alloy has.

Microhardness of coatings 12Cr15G9ND + Cu + Al

In the Table 4 and 5 show the microhardness of the coating in argon and nitrogen.

Microhardness is crowned twice in nitrogen. Compare the microhardness with some well-known materials. The microhardness of Cu + Al + 12Cr15G9ND is not inferior to ordinary steels, but of course it lags behind nanostructured coatings (30–50 GPa).

Table 4

Microhardness of samples from AiSi steel coated with Cu + Al + 12Cr15G9ND, 1 hour in argon

Number of measurements	The microhardness of the coating Cu + Al + 12Cr15G9ND, MPa — in argon						
	HV1 = = 9.807N	HV0.5 = = 4.903N	HV0.3 = = 2.942N	HV0.2 = = 1.961N	HV0.1 = = 0.981N	HV0.05 = = 0.49N	HV0.025 = = 0.245N
1	394	357.2	440.8	385.7	551.4	454.6	357.2
2	317.7	336.6	371.1	430.2	517.6	530.5	351.5
3	379.5	322.6	363.1	467.6	483.3	504.4	445.6
4	366	435.3	354.7	383.2	524	475.4	367.8
5	346.9	343.9	398.1	386.5	441.2	507.6	487.5
6	337.4	397.5	360.6	423.1	465.1	555.2	416.7
7	310.6	339.3	363.5	360.2	521.5	547	502.8
8	312.6	328.4	345.1	417.1	450.6	469.3	454.5
Average	**339.7**	**362**	**382.6**	**402.8**	**489.4**	**498.7**	**436**

Table 5

Microhardness of samples from AiSi steel coated with Cu + Al + 12Cr15G9ND, 1 hour in nitrogen

Number of measurements	The microhardness of the coating Cu + Al + 12Cr15G9ND, MPa — in nitrogen				
	HV1 = = 9.807N	HV0.5 = = 4.903N	HV0.3 = = 2.942N	HV0.2 = = 1.961N	HV0.1 = = 0.981N
1	2	3	4	5	6
1	383.6	439	520.6	531.7	821.3
2	391.5	437.2	538.2	518.3	824.6
3	380.5	414.7	495.4	533	789.2

Continuation of Table 5

1	2	3	4	5	6
4	416.2	423.4	517	555.8	723.7
5	354.7	488.1	580.8	540.6	733.4
6	418.2	425.9	546.9	511.9	751.4
Average	**392.7**	**435.4**	**522.4**	**531.1**	**783.3**

Table 6

Microhardness of samples of some materials

Material	Temporary resistance, $\delta_{в}$, MPa	Relative extension, δ_5, %
Steel 12Cr18N10T	530	40
Steel 3sp	360–460	22
Copper M1	200–260	36
Aluminum A5M	60	30

Tribological characteristics of the coating 12Cr15G9ND + Cu + Al

The friction coefficient in argon and nitrogen are presented in Table 7 and 8.

Table 7

Coefficients of friction coated with Cu + Al + 12Cr15G9ND in nitrogen

Number of measurements	Coefficient of friction			
	40 min	1 hour	2 hour	2 hour 20 min
1	0.265	0.296	0.309	0.385
2	0.293	0.340	0.297	0.332
3	0.264	0.316	0.291	0.397
4	0.254	0.284	0.276	0.382
5	0.242	0.296	0.263	0.319
Average	**0.274**	**0.298**	**0.282**	**0.353**

Table 8

Coefficients of friction coated with Cu + Al + 12Cr15G9ND in argon

Number of measurements	Coefficient of friction			
	40 min	1 hour	2 hour	2 hour 20 min
1	0.256	0.265	0.296	0.276
2	0.215	0.293	0.340	0.256
3	0.244	0.264	0.316	0.238
4	0.225	0.254	0.284	0.224
5	0.252	0.242	0.296	0.245
Average	**0.228**	**0.274**	**0.298**	**0.258**

On average, the friction coefficients of the coatings in argon and nitrogen are almost the same and amount to about 0.270. Let us make a comparison with the friction of various metals (Table 9).

Table 9

Friction coefficients for the same pairs of materials [10]

Material combinations		Coefficient of friction
1	2	3
Al	Al	(1.05–1.35)
Cu	Cu	1.0
Steel	Steel	0.8
Fe	Fe	1.0
Cd	Cd	0.5

Continuation of Table 9

1	2	3
Cr	Cr	0.41
Mg	Mg	0.6
Ni	Ni	(0.7–1.1)
Pt	Pt	1.2
Ag	Ag	1.4
Zn	Zn	0.6

The proposed coatings have antifriction properties of 3 or more times and can be used in tribological pairs.

High-entropy autowave processes

The microhardness of the coating was measured on an HVS-1000A instrument with data output to a computer through 0.1 millimeter. The total area of the sample was 20 x 20 mm. Measurements were taken along and across. For the deposition of coatings, polished disks of steel 45 with a diameter of 20 mm were used as substrates (Fig. 2*a*), which were simultaneously placed on the substrate holder in order to deposit the coating on different substrates under identical conditions. Before deposition, the substrates were cleaned in an ultrasonic bath in a solution of acetone with ethyl alcohol (Fig. 2*b*)

A small part (of 50 pieces) of the results is shown in Figure 7. From Figure 7 that the wavelength is of the order of 10^{-4} m, i.e. mass transfer rate is ~10^{-4} m/s. Since the mass transfer rate, for the diffusion coefficient we obtain the estimate $D \approx 10^{-8}$ m^2/s. This corresponds to the mode of low diffusion.

The experimental results obtained by us above fit into the model of macroscopic localization of plastic flow developed in [11].

In this work, it is shown that the localization of plastic flow in metals and alloys has a pronounced wave character. Moreover, at the stages of easy slip, linear and parabolic strain hardening, and also at the stage of preliminary fracture, the observed localization patterns are different types of wave processes.

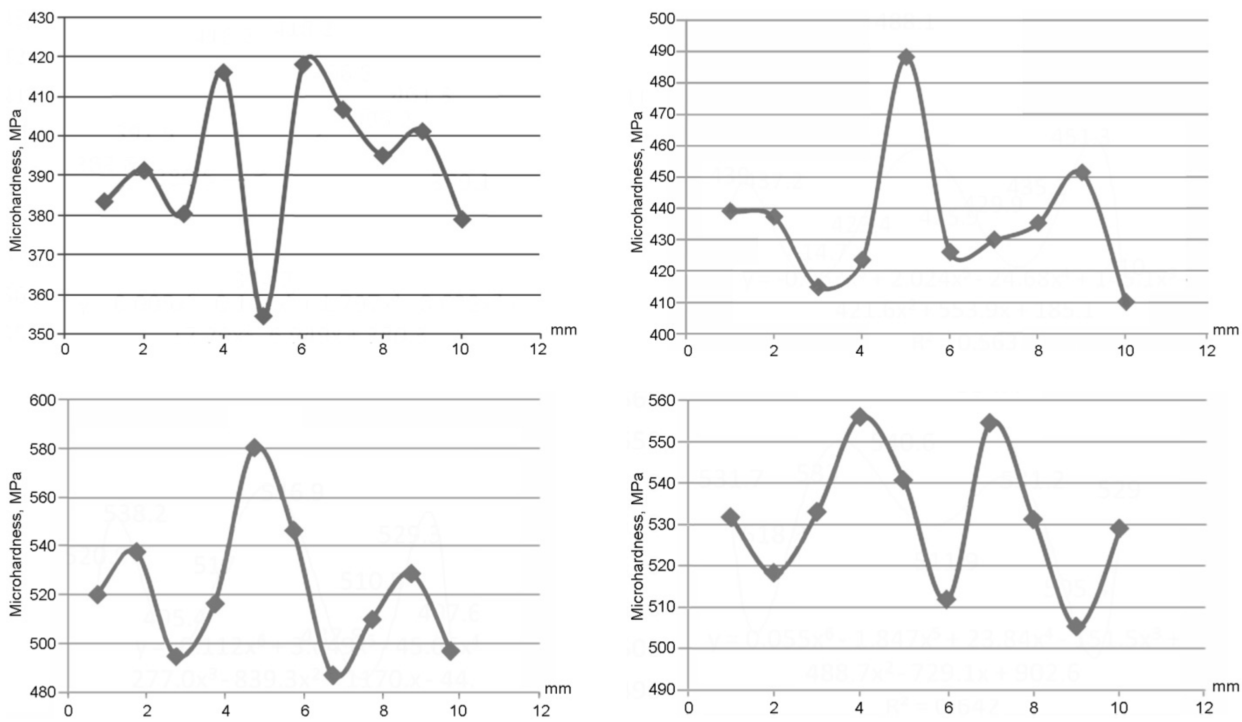


Figure 7. Autowaves in an ion-plasma coating

Conclusion

1. This section discusses the application of highly entropic coatings while simultaneously sputtering three cathodes in a single cycle. The cathodes 12Cr15G9ND, Cu, Al were chosen as cathodes.

2. The chemical composition of the cathodes was measured using a TESCAN MIRA 3 electron microscope. Using these cathodes, coatings were applied to polished samples of steel 45 in an NNV-6.6 I1 vacuum-ion-plasma unit. The measurement was carried out using a MIRA 3 scanning electron microscope, an HVS-1000A microhardness tester, and a tribological research facility.

3. SEM images show that with an increase of ×5000 or more, the droplet phase is clearly detected. The maximum size of the droplet phase reaches 12.0 μm with a variety of morphometric parameters varying, for example, in a geometry coefficient K from 0.8 to 1.0. Analysis of the coating at high magnifications (×7000 and ×20,000) without the droplet phase showed that the grain sizes of the coating are as follows: minimum — 0.23 microns, maximum — 0.65 microns (average of about 0.47 microns); wherein the coating structure is homogeneous.

4. The analyzed type of structure refers to the zone of the so-called «competing texture», when a dense nanocrystalline structure is present in the lower region of the film, and a columnar structure is above it.

5. In argon and nitrogen, the XPS spectra of the coating differ significantly: iron in argon is ~40 weight. %, and in nitrogen ~22 weight. %, i.e. twice smaller; on the contrary, copper in argon is ~14 weight. %, and in nitrogen ~28 weight. %, i.e. twice as much. The XPS spectra of coatings in nitrogen contain on average: Cu ~28 wt.%, Fe ~18 wt.%, Al ~16 wt.%, Cr ~9 wt.%. High entropy alloys (wind farms) should, as a rule, consist of five or more basic elements with a concentration between 5 and 35 %. We got 4 main elements in the range of 5–35 weight. %, typical for wind farms, but in non-equimolar proportion.

6. The results of this study allow us to conclude that the simultaneous deposition of deposited cathodes of various metals (especially composite) in principle allows to obtain highly entropic coatings. Two points must be taken into account here: firstly, the number of atomized cathodes must be increased; secondly, it is necessary to take into account the values of the erosion coefficient for the cathodes used so that the atomized fluxes are equimolarly proportional.

7. Measurements of coatings showed that the microhardness of Cu + Al + 12Cr15G9ND is not inferior to ordinary steels, but of course lags behind nanostructured coatings (30–50 GPa). The proposed coatings have antifriction properties of 3 or more times and can be used in tribological pairs.

8. The experiments showed that the wavelength of the studied coatings is of the order of 10^{-4} m, i.e. mass transfer rate is ~10^{-4} m/s. Since the mass transfer rate, for the diffusion coefficient we obtain the estimate $D \approx 10^{-8}$ m^2/s. This corresponds to the mode of low diffusion. The experimental results obtained above fit into the model of macroscopic localization of plastic flow.

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Үш катодтың бір циклінде бір уақытта тозаңдату кезіндегі жоғары энтропиялық жабындарды жағу

Берілген жұмыста үш катодты тозаңдатудың бір циклінде жоғары энтропиялы жабындыларды бір уақытта жағу қарастырылған. Катодтар ретінде 12Х15Г9НД, Cu, Al катодтары таңдап алынды. Катодтардың химиялық құрамы TESCAN фирмасының MIRA 3 электронды микроскопында өлшенген. Бұл катодтардың көмегімен ННВ-6.6 И1 вакуумдық ионды-плазмалық қондырғыда 45 болаттан жасалған, өңделген үлгілерге жабындылар жағылды. Өлшеулер MIRA 3 растрлық электронды микроскопында, HVS-1000A микроқаттылықты өлшегіште және трибологиялық зерттеулерге арналған қондырғыда жүргізілген. РЭМ-суреттер ×5000 және одан да көп ұлғайту кезінде тамшылы фазаның айқын байқалатындығын көрсетті. Тамшылы фазаның ең максималды өлшемі морфометриялық параметрлердің әртүрлілігі кезінде 12,0 мкм-ге дейін жетеді, мысалы, K геометрия коэффициенті бойынша 0,8-ден 1,0-ге дейін. Тамшылы фазасыз (×7000 және ×20 000) ұлғаю кезінде жабындылар қаптамаларының талдауы жабынды дәндерінің өлшемдері мынадай болатынын көрсетті: ең кішісі — 0,23 мкм, ең үлкені — 0,65 мкм (орташасы шамамен 0,47 мкм); бұл жағдайда жабындылар құрылымы біртекті. Құрылымның талданатын типі қабыршақтың төменгі аймағында тығыз нанокристалдық, ал оның жоғары жағында бағаналы құрылым болып келетін «бәсекелес құрылым» деп аталатын аймаққа жатады. Бұл зерттеудің нәтижелері келесі қорытындыны жасауға мүмкіндік береді: әртүрлі металдардың (әсіресе композициялық) тұндырылған катодтарын бір уақытта жағу жоғары энтропиялық жабындыларды алуға мүмкіндік береді. Бұл жағдайда екі сәтті ескеру қажет: біріншіден, тозаңдатушы катодтар санын арттыру қажет; екіншіден, тозаңдатушы ағындар эквимолярлы пропорционал болуы үшін қолданылатын катодтарға арналған эрозия коэффициентінің мәндерін ескеру қажет. Жабындылардың өлшеулері Cu + Al + 12Х15Г9НД микроқаттылығы кәдімгі болаттардан кем емес екенін көрсетті, бірақ әрине, олар наноқұрылымды жабындылардан (30–50 ГПа) артық емес. Ұсынылған жабындардың антифрикциялық қасиеттері 3 немесе одан да көп және оларды триботүйіндесу жұптарында қолдануға болады.

Кілт сөздер: көп қабатты жабындылар, қаттылық, иілгіштік, үйкеліс, тозуға төзімділік, наноқұрылым, микроқаттылық, катод, құрылым.

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Нанесение высокоэнтропийных покрытий при одновременном распылении в одном цикле трех катодов

В статье рассмотрено нанесение высокоэнтропийных покрытий при одновременном распылении в одном цикле трех катодов. В качестве материала катодов были выбраны 12Х15Г9НД, Cu, Al. Химический состав определялся на электронном микроскопе MIRA 3 фирмы TESCAN. С помощью этих катодов были нанесены покрытия на полированные образцы из стали 45 на вакуумной ионно-плазменной установке ННВ-6.6 И1. Измерение осуществлялось на растровом электронном микроскопе MIRA 3, микротвердомере HVS-1000A и установке для трибологических исследований. РЭМ-изображения показывают, что при увеличении ×5000 и более отчетливо выявляется капельная фаза. Максимальный размер капельной фазы достигает 12,0 мкм при разнообразии морфометрических параметров, варьирующихся, например, по коэффициенту геометрии K от 0,8 до 1,0. Анализ покрытия при больших увеличениях (×7000 и ×20 000) без капельной фазы показал, что размеры зерен покрытия следующие: минимальный — 0,23 мкм, максимальный — 0,65 мкм (средний порядка 0,47 мкм); при этом структура покрытия однородная. Анализируемый тип структуры относится к зоне так называемой «конкурирующей текстуры», когда в нижней области пленки присутствует плотная нанокристаллическая, а выше ее — столбчатая структура. Результаты настоящего исследования позволяют сделать вывод, что одновременное нанесение осаждаемых катодов различных металлов (особенно композиционных), в принципе, позволяет получать высокоэнтропийные покрытия. Для этого нужно, во-первых, увеличить число распыляемых катодов; во-вторых — учесть значения коэффициента эрозии для используемых катодов, чтобы распыляемые потоки были эквимолярно пропорциональными. Измерения покрытий показали, что микротвердость Cu + Al + 12Х15Г9НД не проигрывает обычным сталям, однако проигрывает наноструктурным покрытиям (30–50 ГПа). Предложенные покрытия в 3 и более раз обладают антифрикционными свойствами и могут быть использованы в парах трибосопряжения.

Ключевые слова: многослойные покрытия, твердость, пластичность, трение, износостойкость, наноструктура, микротвердость, катод, структура.

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