SURFACE MORPHOLOGY AND SPUTTERING OF FeCrAl COATING ON STEEL EXPOSED TO LOW-ENERGY DEUTERIUM PLASMAS

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Processes of sputtering and surface modification of FeCrAl coatings deposited on steel by vacuum arc was studied under the influence of low-energy (500 eV) deuterium plasma with fluence ($4\cdot10^{24}$) D^+/m^2 at room temperature. It was determined the composition of coatings by an energy dispersive X-ray spectrometer allowed to establish that the elements in the coatings are distributed more evenly when it coated in a nitrogen atmosphere. Results of erosion studies indicated that the sputtering yields for deuterium on coatings are 1.3…0.45 at./ion and at least two-three times less in comparison with initial alloys and published data for pure iron and chromium. For coatings deposited in a nitrogen atmosphere found that the obtained sputtering coefficients are almost an order of magnitude smaller in comparison with published data for pure iron and chromium and only 1.8 times higher compared to tungsten.

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INTRODUCTION

Many candidate structural materials have been studied for developing a fusion reactor, such as ferritic/ martensitic, austenitic steels and various alloys [1]. Recently, however, FeCrAl alloys have been also considered as a promising candidate for a fusion blanket application [2] owing to their excellent formability, high strength, and oxidation resistance at high temperature.

To assess the possibility to use the FeCrAl alloys as plasma-facing materials, there is a need to examine a behavior of these materials under plasma exposure. Sputtering of plasma-facing materials due to interaction with energetic ions (particularly hydrogen isotopes) is an essential issue in magnetically confined fusion devices because it is directly related to impurity generation as well as to the lifetime of plasma – facing components [3]. For a better understanding of the sputtering processes on elements of FeCrAl alloys it is necessary firstly to know the sputtering of FeCrAl as whole.

The goal of this work is to experimentally determine the sputtering yields and surface morphology change of FeCrAl coatings exposed to low-energy, high-flux deuterium plasma and compares the obtained data of sputtering yields with existing published data for Fe, Cr, and W.

1. MATERIAL AND METHODS

Experiments for coatings deposition were carried out in a vacuum arc setup equipped with a plasma source with magnetic confinement of the cathode spot [4].

The commercial Kanthal-type FeCrAl alloys were used as cathodes with diameter 60 mm. The chemical compositions of this alloy are specified in Table 1. The arc discharge current in all experiments was 100 A. The plasma source was mounted on a flange of a vacuum chamber with a diameter and a length of 500 mm. The coatings were deposited on polished stainless steel (18Cr10NiTi) samples Ø-20 mm, thickness of 3 mm, which were placed on a substrate holder in the center of the chamber at a distance of 250 mm from the cathode.

Table 1

Typical nominal and measured compositions of Kanthal-type alloy FeCrAl

Initial bar (commercial)	Concentration, wt. %						
	Fe	**Cr**	**Al**	Other elements			
nominal	bal.	22…24	5…5.8	**Si** - to 0.5	**Ti**- 0.2-0.5	**Mn**- to 0.3	**Ni**- to 0.6
measured	bal.	22.65±0.05	4.04±0.02	0.53±0.03	0.21±0.08	0.28±0.06	0.13±0.06

Before deposition of coatings, the chamber was pumped out by a diffusion pump to a residual gas pressure of ~ 10^{-3} Pa and the samples surface was sputtered with metal plasma of the cathode material for 3 min at a negative voltage on the substrate holder of 1300 V. Coatings were deposited in vacuum of ~ $2\cdot10^{-3}$ Pa (series F1) and at a pressure of nitrogen (series F2) in the chamber ~ 0.05 Pa for 60 min. The rotation speed of the samples during deposition is ~ 9 rpm. The negative bias potential on the samples was -50 V, and the temperature ~ 450°C.

The thickness of the deposited coatings was measured using an MII-4 optical interference microscope by the "shadow knives" method and were ~ 20 μm in all cases.

The specimens have been irradiated with deuterium ions using glow gas-discharge plasma at 1000 V. The design and principle of operation of the gaseous plasma source used for irradiation of the samples is described in detail in [5]. The specimen temperature was measured by the thermocouple and was (30±2.5)°C during irradiation.

The dominant ion component generated in the ion source is D_2^+. This ion was chosen as the bombarding species to achieve higher particle fluxes. These molecular D_2^+ ions are considered to be identical to 2 individual D ions impinging with the same velocity as the molecular ion. Breakup of the molecular D_2^+ ions on the target surface results in emergence of two D atoms with the kinetic energy of 1/2 experimentally applied ion energy and the flux is two times the measured ion flux. In our experiments the FeCrAl targets were sputtered with atomic D^+ ions at the ion energy of 500 eV with flux of $3.2\cdot10^{20}$ $m^{-2}\cdot s^{-1}$. The maximum irradiation flu-

ence was $4 \cdot 10^{24}$ m^{-2}. The experimental ion flux and fluence were calculated from the measured ion currents and beam spot areas.

The erosion yield was primarily evaluated by a weight-loss technique. Before and after plasma exposure, the weight of each target was measured by a microbalance system, and the erosion rate was then calculated from the weight loss and the total deuterium fluence.

Investigations of surface microstructure before and after irradiation were performed using scanning electron microscope JEOL JSM-7001F. Chemical composition of the FeCrAl coatings was determined by energy dispersive X-ray spectroscopy – EDS.

2. RESULTS AND DISCUSSION

Fig. 1 shows an SEM image the surface microstructure of the coating series F1 in the initial state and element maps. The corresponding elemental concentrations of EDS scans are given in Table 2. It should be noted that elements concentration given in the table as spectrum # 1 was determined by scanning over a sufficiently large area (about 1 mm^2) and as spectrum #2-6 at individual "points" (size of 5…10 μm^2).

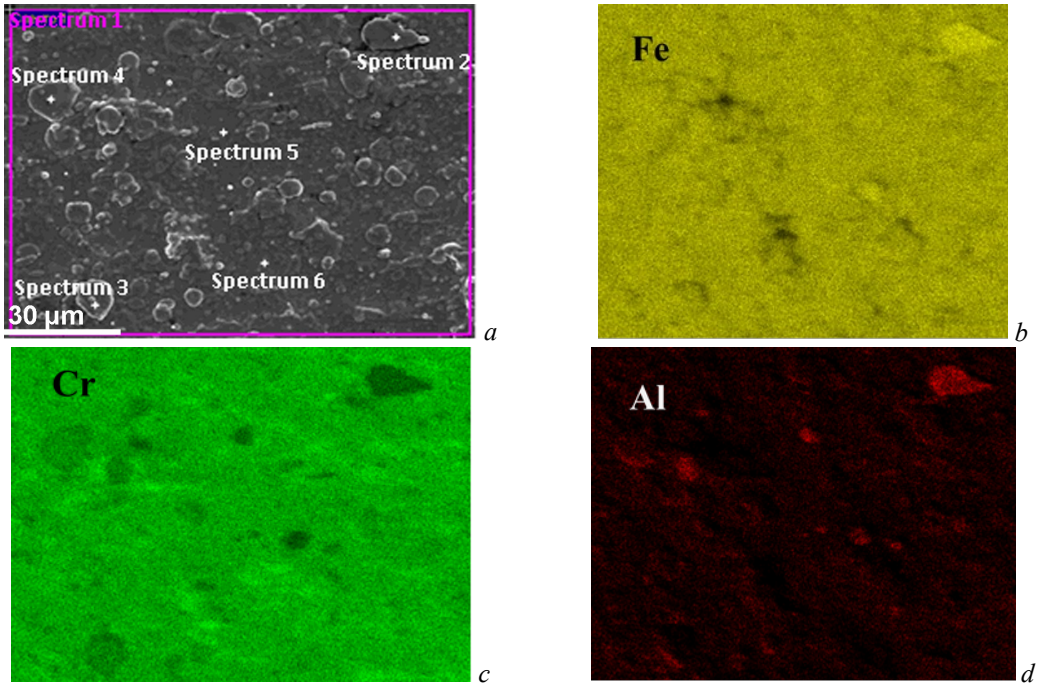


Fig. 1. SEM image (a) and EDS mapping of FeCrAl coatings series F1 (b, c, d) in initial state

Table 2

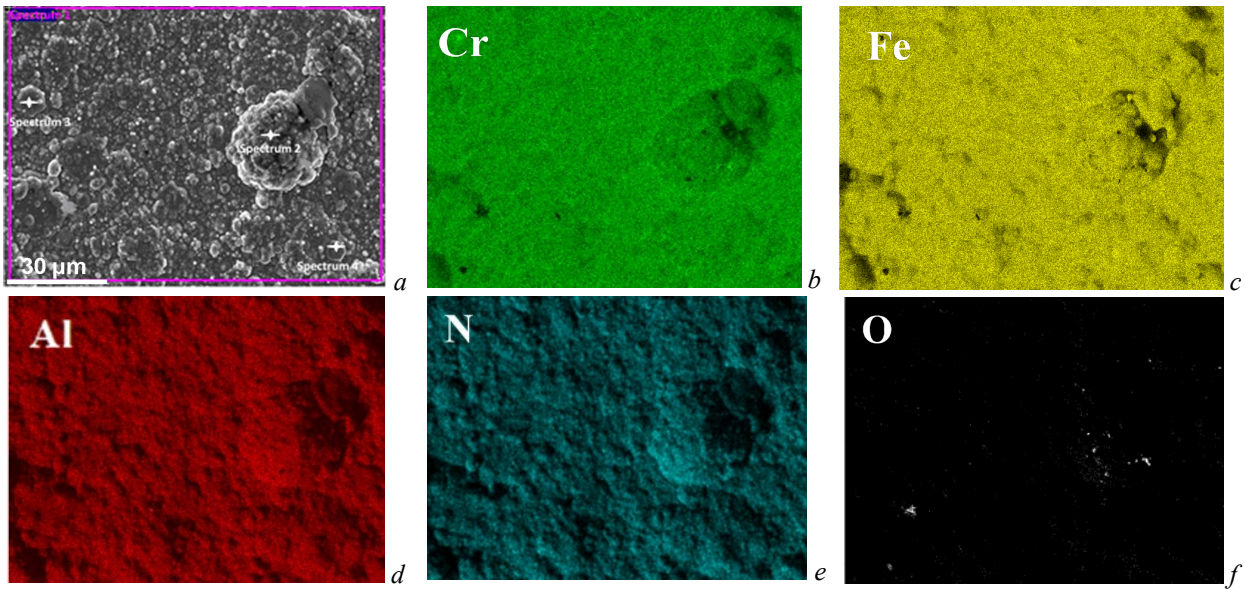
Measured compositions of FeCrAl coatings series F1 in initial state

Spectrum	Concentration, wt. %					
Element	**Fe**	**Cr**	**Al**	**Si**	**Ti**	**Ni**
# 1	65.78	34.01	0.21	-	-	-
# 2	74.77	20.82	3.11	0.85	0.22	0.23
# 3	69.05	30.45	0.5	-	-	-
# 4	68.72	30.77	0.51	-	-	-
# 5	65.98	34.02	-	-	-	-
# 6	65.75	34.25	-	-	-	-

The surface of the coating's series F1 in the initial state was smooth with exhibit specific features as unevenly distributed plates that are irregular in shape and a wide range of sizes. EDS scan a sufficiently large area (spectrum #1) and surface without plates (spectra # 5, 6) indicates that coating is composed of Fe and Crwith a minor amount of aluminum. Aluminum is mainly found in some plates (spectrum # 2).

Fig. 2 shows an SEM image the surface microstructure of the coating series F2 in the initial state and corresponding element maps. The chemical compositions for this alloy are specified in Table 3.

A distinctive feature of series F2 coatings is their more complex composition and almost uniform distribution of elements over the sample. It should also be noted the presence of a significant amount of nitrogen and, in some cases, oxygen. The surface morphology of the F2 series coatings is presented in the form of scales, plates, and macroparticles with an irregular surface (see Fig. 2,a). Concentrations of nitrogen, oxygen, titaniumand zirconium for these structural elements of the surface are differed (see Fig. 2 and Table 3).



a *b* *c* *d* *e* *f*

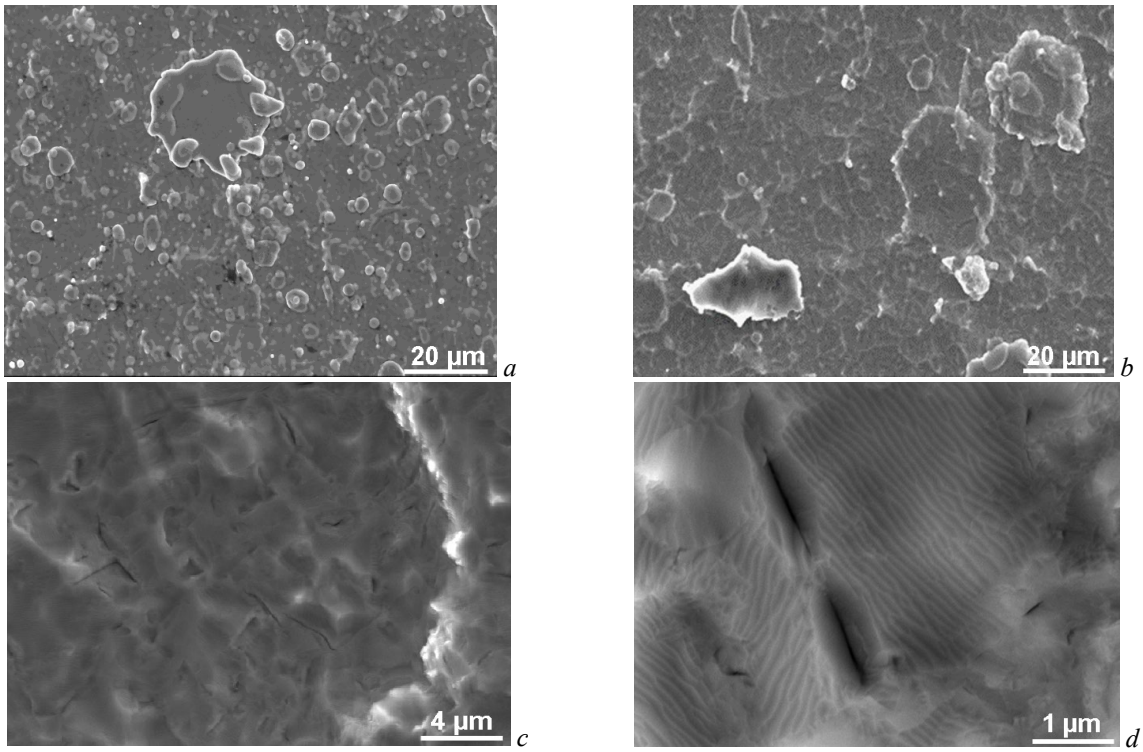
Fig. 2. SEM image (a) and EDS mapping of FeCrAl coatings series F2 (b, c, d, e, f)

Table 3

Measured compositions of FeCrAl coatings series F2 in initial state

Spectrum	Concentration, wt.%								
	Fe	**Cr**	**Al**	**N**	**O**	**Si**	**Ti**	**Mn**	**Zr**
# 1	62.73	23.35	3.43	7.61	0.06	0.17	0.16	0.38	1.84
# 2	56.36	21.26	3.48	9.08	4.96	1.11	0.57	0.38	3.00
# 3	56.26.	21.86	2.58	6.60	6.36	1.13	1.26	-	4.56
# 4	65.79	22.14	2.89	7.39	1.79	-	-	-	-

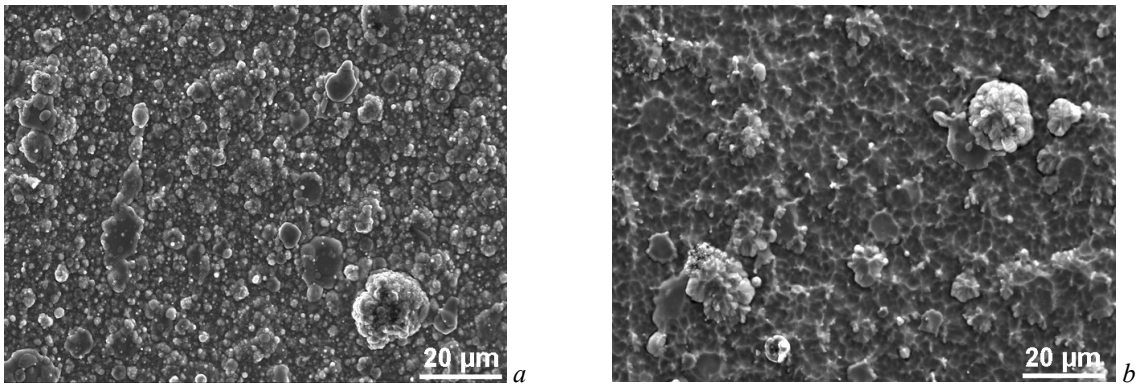
The evolution of the surface of coating F1 (Fig. 3,a) due to the sputtering process after exposure to a high-density deuterium plasma is shown in Fig. 3,b-d. Small macroparticles were completely sputtered, while large particles were reduced in size and, in addition, areas towering above their surface were sputtered. EDS point scan indicates that composition of surface and macro-particlesis contained of elements which consistent with the sputtering target (see Table 2). The details of the surface at higher magnification (see Fig. 3,c,d) are shown that in addition to sputtering the surface plates, sputtering and expansion of cracks occur.



a *b* *c* *d*

Fig. 3. Plan-view of the coating series F1 before (a) and after sputtering (b).
The details of the surface (b) at higher magnification (c, d)

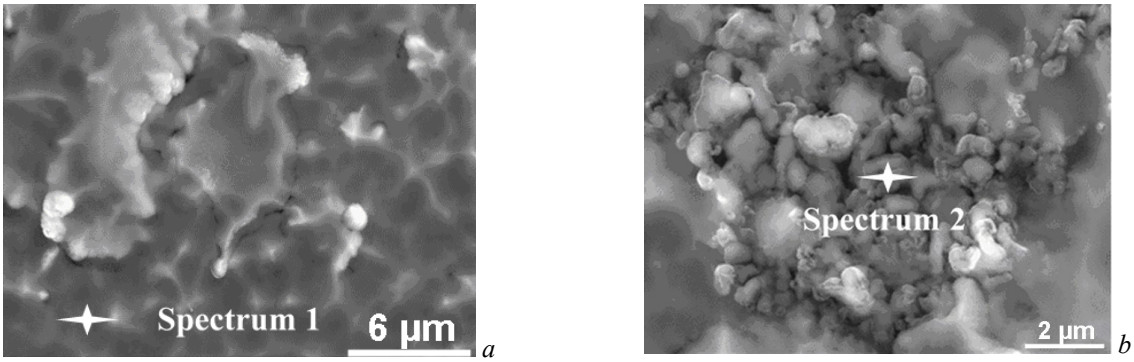
In the case coating of series F2 (Fig. 4,a) after exposed to plasma the surface plates were sputtered almost completely (Fig. 4,b). Large "cauliflower" – like defects also slightly sputtered but still retained their shape. No cracks on the surface were noted.



a *b*

Fig. 4. Plan-view of the of the coating series F2 before (a) and after sputtering (b)

The details SEM image of the surface coatings series F2 (Fig. 4,b) at higher magnification shown in Fig. 5. Can be seen the surface with a cellular structure (see Fig. 5,a). In several places, the formation of structures resembling an accumulation of microscale "barrels" was recorded (see Fig. 5,b). The corresponding elemental concentrations of EDS scans for site with almost completely sputtered plates and "barrels" are given in Table 4.



a *b*

Fig. 5. The details SEM image of the surface coatings series F2 (Fig. 4, b) at higher magnification: site with almost completely sputtered plates (a) and structures resembling "barrels" (b)

Table 4

Measured compositions of FeCrAl coatings F2 after sputtering

Spectrum	Concentration, wt. %				
	Fe	**Cr**	**Al**	**N**	**O**
# 1	61.94	22.29	4.13	8.73	2.19
# 2	9.26	47.44	0.33	3.63	39.35

As seen from Table 4 the corresponding elemental composition for places with almost completely sputtered plates (spectrum #1) almost coincides with measured compositions of FeCrAl coatings series F2 in initial state (see Table 3). An elemental concentration of EDS scans for site with "barrels" (spectrum # 2) shows iron, aluminium and nitrogen depletion and chromium and oxygen enrichment. The high chromium (47.44 wt. %) and oxygen (39.35 wt. %) content in "barrels" indicates the formation of Cr_2O_3.

As noted above in our experiment's deuterium plasmas sputtering erosion of FeCrAl coating was obtained using the weight loss technique. The sputtering yield was then calculated from the weight loss and the total deuterium fluence.

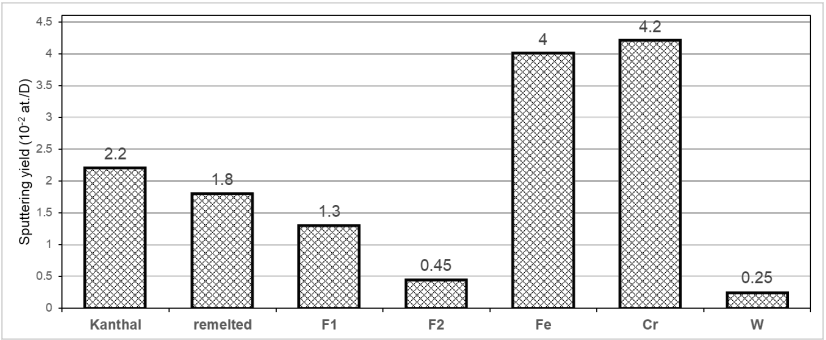


Fig. 6. Experimentally determined sputtering yields of FeCrAl alloys and coatings series F1 and F2. For comparison sputtering yields of Fe, Cr and W are shown [8]

Fig. 6 shows the results for the sputtering yield of FeCrAl coatingin comparison with FeCrAl alloy that were used as cathodes [6] and published data of pure iron, chromium and W sputtering yields determined experimentally and by numerical simulation [7, 8].

As seen in Fig. 6, values of the experimentally measured sputtering yield of the FeCrAl coatings series F1 exposed to the D plasma are one and a half times lower compared to Kanthal-type sample and its remelted version and three times compared to Fe and Cr. The FeCrAl coatings series F2 have the sputtering coefficient $0.45 \cdot 10^{-2}$ at./D which is almost an order of magnitude smaller comparison with published data for pure iron, chromium. It should be noted that the sputtering coefficients of the coatings F2 deposited in nitrogen are only 1.8 times higher compared to tungsten. At present, the sputtering coefficient of tungsten with hydrogen isotopes is considered to be the lowest. FeCrAl coatings with middle-Z admixed elements can been considered as a candidate for armor materials of plasma-facing components of tokamak devices.

CONCLUSIONS

Processes of sputtering and surface modification of FeCrAl coatings deposited on steel by vacuum arc was studied under the influence of low-energy (500 eV) deuterium plasma with fluence $4 \cdot 10^{24}$ D^+/m^2 at room temperature.

The structures that were firstly sputtered are the flakes, plates and macroparticles with an irregular surface that were deposited on the surface at construction of coatings.

The sputtering yields for deuterium on coatings are 1.3…0.45 at./ion and at least two-three times less in comparison with initial alloys and published data for pure iron and chromium. For coatings deposited in a nitrogen atmosphere found that the obtained sputtering coefficients are almost an order of magnitude smaller in comparison with published data for pure iron and chromium and only 1.8 times higher compared to tungsten

Results reported in this work to be of interest for an understanding of mechanisms of possible formation of erosion structures on the FeCrAl coatings surfaces under exposure to the deuterium plasmas and predicting possibilities of using these coatings as of plasma – facing materials of fusion reactors.

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МОДИФИКАЦИЯ ПОВЕРХНОСТИ И РАСПЫЛЕНИЕ FeCrAl-ПОКРЫТИЙ НА СТАЛИ ПРИ ВОЗДЕЙСТВИИ НИЗКОЭНЕРГЕТИЧЕСКОЙ ДЕЙТЕРИЕВОЙ ПЛАЗМЫ

В.Н. Воеводин, Г.Д. Толстолуцкая, А.В. Никитин, Р.Л. Василенко, А.С. Куприн, В.А. Белоус, В.Д. Овчаренко

Процессы напыления и модификации поверхности покрытий FeCrAl, нанесенных на сталь вакуумной дугой, изучали под воздействием низкоэнергетической (500 эВ) плазмы дейтерия с флюенсом $4 \cdot 10^{24}$ $D^+/м^2$ при комнатной температуре. Был определен состав покрытий с помощью энергодисперсионного рентгеновского спектрометра: это позволило установить, что элементы в покрытии распределяются более равномерно при нанесении покрытия в атмосфере азота. Результаты эрозионных исследований показали, что коэффициенты распыления дейтерием покрытий составляют 1,3…0,45 ат./ион и, как минимум, в два-три раза меньше по сравнению с исходными сплавами и опубликованными данными для чистого железа и хрома. Для покрытий, нанесенных в атмосфере азота, установлено, что полученные коэффициенты распыления почти на порядок меньше по сравнению с опубликованными данными для чистого железа и хрома и лишь в 1,8 раза выше по сравнению с вольфрамом.

МОДИФІКАЦІЯ ПОВЕРХНІ І РОЗПИЛЕННЯ FeCrAl-ПОКРИТТІВ НА СТАЛІ ПРИ ДІЇ НИЗЬКОЕНЕРГЕТИЧНОЇ ДЕЙТЕРІЄВОЇ ПЛАЗМИ

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Процеси напилення і модифікації поверхні покриттів FeCrAl, нанесених на сталь вакуумною дугою, вивчали під впливом низькоенергетичної (500 еВ) плазми дейтерію з флюенсом $4 \cdot 10^{24}$ $D^+/м^2$ при кімнатній температурі. Було визначено склад покриттів за допомогою енергодисперсійного рентгенівського спектрометра: це дозволило встановити, що елементи в покритті розподіляються більш рівномірно при нанесенні покриття в атмосфері азоту. Результати ерозійних досліджень показали, що коефіцієнти розпилення дейтерію на покриттях становлять 1,3…0,45 ат./іон і, як мінімум, в два-три рази менші в порівнянні з вихідними сплавами і опублікованими даними для чистого заліза і хрому. Для покриттів, нанесених в атмосфері азоту, встановлено, що отримані коефіцієнти розпилення майже на порядок менші в порівнянні з опублікованими даними для чистого заліза і хрому та лише в 1,8 рази вищі в порівнянні з вольфрамом.