

<https://doi.org/10.15407/knit2024.05.019>

UDC 669.018.8

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VACUUM ARC COATINGS FOR COMBUSTION CHAMBERS OF ROCKET ENGINES

An investigation was done for heat-resistant coatings from Ni-Cr and Fe-Ni-Cr alloys, produced by vacuum arc deposition with cathodes from nichrome alloy XH78T and steel 12X18H10T. The microstructural analysis of the studied coatings applied by vacuum arc deposition with different amounts of reactive gas (nitrogen) in a deposition chamber showed the presence of two phases: a matrix and the distributed particles of liquid phase of various sizes and shapes. Vacuum arc deposition at a cathode arc current of 55 A for Ni-Cr and 82 A for Fe-Ni-Cr coatings, a cathode arc voltage of 28 to 32 V, an underlayer temperature of $490 \pm 30^\circ\text{C}$, and no nitrogen in the chamber gives coatings with no apparent orientation of crystallites and a microhardness reaching 330 to 374 HV. The diffusion mobility of atoms decreases at a higher nitrogen pressure, which results in a coarse-crystal, porous-columnar structure of coatings with high porosity, a varying volume, and a rough surface. The influence of nitrogen pressure in a chamber on the strength of coatings was evaluated by their microhardness. A nitrogen pressure gain up to 0.13 Pa results in the increased microhardness of both coating systems, and the further growth in pressure has almost no effect on microhardness. It was discovered that a nitrogen pressure of 0.13 Pa gives the optimum combination of a coating's strength and continuous structure with no pores and apparent columnar crystallites.

Цитування: Nadtoka V. M., Husarova I. O., Kraiev M. V., Borysenko A. Yu., Bondar D. M., Osinovy G. G. Vacuum arc coatings for combustion chambers of rocket engines. *Space Science and Technology*. 2024. **30**, No. 5 (150). P. 19–28. <https://doi.org/10.15407/knit2024.05.019>

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Alloys from Ni-Cr with 80 % Ni are among the most heat-resistant industrial materials with a maximum operating temperature of up to 1200 °C. However, they are expensive, which makes Fe-Ni-Cr alloys more attractive, providing their heat resistance is sufficient for the operating temperature range. Therefore, it is reasonable to conduct further research on both Ni-Cr and Fe-Ni-Cr coatings to develop a deposition technology that can provide the required performance parameters. Controlling nitrogen pressure makes it possible to adjust the structure and properties of the coating during deposition.

Keywords: Ni-Cr, Fe-Ni-Cr heat-resistant coatings, microstructure, microhardness.

INTRODUCTION

A combustion chamber, where fuel and oxidizer mix and burn, is one of the core elements in liquid-propellant rocket engines. Pressure in a combustion chamber may reach 1 to 30 MPa at gas flow temperatures of 3000 to 3800 °C [8, 20]. Most chambers of liquid-propellant rocket engines have external cooling to reduce thermal loads, where a cooling medium flows in a channel formed by the internal and external shells of a chamber. The temperature of a chamber wall that heats up when the engine is operating must not decline the required strength of the structure. Therefore, it is necessary to use heat-resistant alloys with high thermal conductivity coefficients. Along with this, structural materials for combustion chambers are selected considering the design specificity of a chamber and the cost limitations.

The early combustion chambers in the large engines made in the United States, Europe, and Japan had a tubular design and were made from strengthened nickel (Nickel 200), stainless steels AISI 316, 347, and alloys 285, Inconel, and X-750 with a high nickel content. However, for the Shuttle spacecraft with higher combustion temperature and pressure in the chamber, it appeared necessary for a combustion

chamber to have a much stronger structure of the hot wall with copper alloy channels of high thermal conductivity. A new copper alloy, NARloy-Z (3 % Ag, 0.5 % Zr), was developed to meet this necessity. The strength and the thermal fatigue of NARloy-Z at the operating temperatures appeared to surpass the existing copper alloys, and the thermal conductivity of NARloy-Z was 80 % higher than in pure copper [16].

In the Soviet Union, the configurations of combustion chambers and nozzles had channeled or multilayered walls [20]. Normally, the internal shell was made from copper-chromium alloy (~3 % Cr), where slots and channels were cut out, and this shell was soldered to the external wall from stainless steel or nickel alloy (Figure 1).

Therefore, in most up-to-date engines, the internal wall of a combustion chamber is made from copper alloys (bronze) with high thermal conductivity necessary to increase engine efficiency [15, 16]. However, the low heat resistance and heat strength of copper alloys limit the service lives of these combustion chambers. This problem needs to be solved in the present-day rocket engineering industry, where space and rocket technologies are reusable.

The service life of a reusable combustion chamber is at least 50 cycles of 170 to 200 seconds [20], and the wall temperature of this combustion chamber may exceed 800 °C regardless of cooling [21]. The load from high internal pressure in the cooling channels, thermomechanical fatigue, and creep may cause damage to these channels [18, 19].

The influence of heat fluxes is reduced, and the service lives of combustion chambers from copper alloys are enhanced by applying thermal barrier coatings (TBC). The most common coatings for gas turbines are composed of three layers: a heat-resistant underlayer from NiCrAlY to ensure the strong adhesion of the thermal barrier coating with the surface, protect this surface at operating temperatures of 900 to 1100 °C, and reduce the difference in the coeffi-

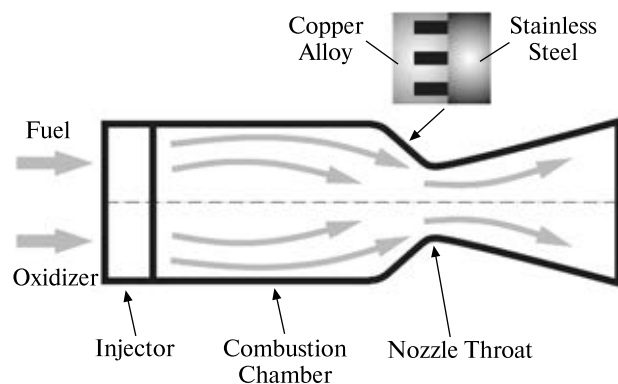


Figure 1. The combustion chamber of a liquid-propellant rocket engine with an internal wall from copper alloy

cients of thermal linear expansion of the metal base and the external ceramic layer; an intermediate layer from Al_2O_3 that adheres the ceramic layer and the heat-resistant underlayer and serves as a barrier for oxygen diffusion to the underlayer; and the external layer from $\text{ZrO}_2 \text{ Y}_2\text{O}_3$. These coatings are applied by electron beam evaporation and plasma spraying under an atmospheric or reduced pressure [7].

Another coating was developed for the Ogbuji rocket engines to protect their combustion chamber walls. It was a CuCr coating from Cu-8%Cr-5%Nb alloy. With a 21 % chromium content, this alloy demonstrated 10 hours of service during tests. In addition, a two-layer coating comprising the external layer of NiCrAlY with a CuCr binding layer on the underlayer from Cu-8%Cr-4%Nb [7].

The analysis of the existing thermal protection coatings for the copper walls of combustion chambers showed that further development efforts are necessary for enhancing the service lives and the performance parameters of chambers. These thermal protection coatings shall have CLTEs similar to that of copper or include binding layers with corresponding CLTEs to reduce thermal stresses in the material. Furthermore, these coatings must perform under the influence of the oxidizer and propellant combustion products, keep their performance parameters within the entire temperature range, and have an affordable price.

Electrophysical techniques of coating application show promise for the protection of metals and alloys, particularly the methods of depositing condensed metals and chemical compounds in a vacuum: thermal evaporation, cathode spraying, magnetron sputtering, reactive electron beam evaporation in plasma, activated reactive evaporation, plasma spraying under ion bombardment (vacuum arc deposition) [17].

All these technologies, except for vacuum arc deposition, are low-energy and, therefore, cannot give the high adhesion of coatings at underlayer temperatures lower than recrystallization temperatures. Electron-ionic installations make it possible to control and adjust the plasma flow energy and, consequently, the underlayer temperature and adhesion within a wide range. However, these installations did not become common in the industry. The primary reason is that these systems are too sophisticated because they use complex electron guns and high voltage (up to 5 kV) [12].

Most of these disadvantages are not the case for the method of vacuum plasma spraying under vacuum bombardment. The principal distinguishing feature of this method is the deposition of a coating under the influence of an electrostatic field and a high-energy plasma flow generated by a vacuum arc. Compared to other methods of coating spraying in a vacuum, the technique of vacuum gas deposition has significant advantages, such as a simple and highly effective process, low power consumption, and the possibility to control the primary process parameters, which makes it possible to adjust the structure and the properties of obtained coatings [12].

The purpose of this work is the development of protective coatings from Ni-Cr and Fe-Ni-Cr alloys for combustion chambers, applied by vacuum arc deposition with modification by nitrogen to increase hardness and wear resistance.

SELECTING COATING COMPOSITION AND APPLICATION METHOD

The intensity of stress in a coating and the adhesion between a coating and its underlayer depend on the thermal expansion coefficients of the coating and the combustion chamber wall. These coefficients must be similar to avoid coating delamination because of the rapid heating and cooling of the structure. Steels from Fe-Ni-Cr meet this requirement (see Table 1).

Cyclic variation in temperature has also a significant influence on the heat resistance of materials and alloys [9]. In multi-phase alloys, regularly repeated cycles of heating and cooling may cause phase recrystallization and separation of secondary phases, which results in the occurrence of internal stress. Therefore, the alloys with no phase and structural transformations within the operating temperature range have the best properties.

Table 1. Thermal expansion coefficients of materials

Material	Thermal expansion coefficient ($10^{-6} \text{ }^\circ\text{C}^{-1}$) at different temperatures ($^\circ\text{C}$)		
	20	600	800
Copper [23]	17	18	20
Bronze [23]	19	—	—
Steel 12X18H10T [23]	17.0	18.2	18.9
Nichrome		~14	

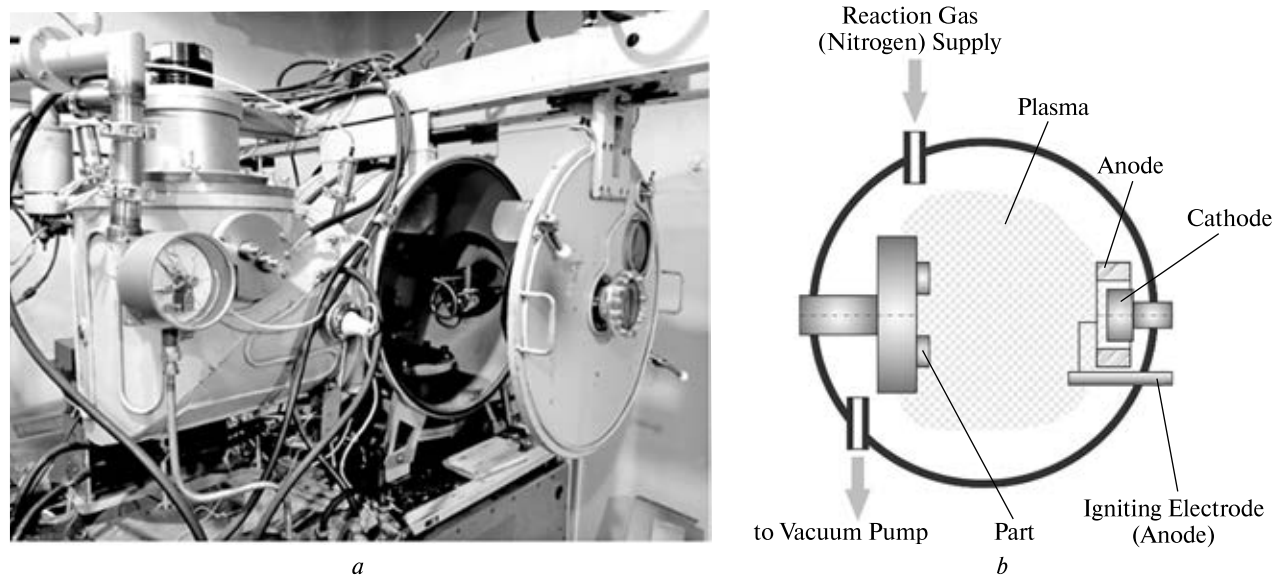


Figure 2. Vacuum arc deposition installation (a) and profile (b)

Table 2. Conditions of ionic cleaning for the surfaces of samples

Cathode material	Pressure in the chamber [Pa]	Duration [min]	Cathode arc current [A]	Cathode arc voltage [V]	Sample bias voltage [V]
12X18H10T	4×10^{-3}	2.0–2.5	70–80	28	600–900
XH78T	1×10^{-2}	1.1–2.7	50	27	600–800

Austenitic steels such as 12X18H9 are single-phase at temperatures of up to 600 °C and can operate in medium-aggressive environments at these temperatures [23]. The heat resistance of these steels is reached mainly by dissolving alloying elements in iron [9]. However, the phase composition and the internal structure of coatings applied from these steels by vacuum arc deposition will differ from the phase composition and the internal structure of stainless steel produced by a traditional metallurgical method. Therefore, a thorough investigation is necessary concerning the temperature and concentration limits for the separation and dissolution of the α -phase and carbides in vacuum-arc coatings from stainless steel.

It may be sensible to make heat-resistant coatings from Ni-Cr alloys with a 22 percentage of chromium maximum and the highest operating temperature of 1200 °C. This can be seen from the phase equilibrium diagram of a Ni-Cr system [22], showing that the Ni-Cr alloy with the specified chromium content remains a solid solution at temperatures of up to 1250 °C.

Although the thermal expansion coefficient of ni-chrome is 20–23 % lower than that of bronze, the high energy of ions and ionic cleaning directly before coating application give a sufficient adhesion of the coating with the underlayer in a vacuum arc deposition process [3]. When a high negative potential is applied to the underlayer, the surface is dispersed by the bombarding ions from cathode material plasma. Such cleaning and activation of the underlayer's surface enables the highest adhesion between the underlayer and the applied coating compared to other methods [2].

The surfaces of the samples were prepared by grinding with sandpaper with a grain size of P240, etching with a 5 % solution of nitric acid for one minute, and ionic cleaning in a vacuum chamber in the conditions specified in Table 2.

Coatings were applied in a vacuum arc deposition installation built on the basis of a vacuum station YBH-75 (Figure 2) with the oil backing vacuum pump replaced with an oil-free spiral pump, and the high-vacuum oil-vapor pump substituted with a tur-

bomolecular one. Furthermore, the installation was outfitted with electric-arc metal evaporators.

The following primary parameters have the greatest influence on the structure and properties of coatings [4, 6, 10, 14]: arc current, underlayer temperature, and reactive gas pressure in the chamber.

Experimental coatings from steel 12X18H10T and nichrome XH78T were applied by vacuum arc deposition on the surfaces of Cu-Cr-Zr bronze samples that simulated the material of a wall of a combustion chamber in a liquid-propellant rocket engine in the conditions specified in Table 3.

The temperatures of samples during coating application were limited by the recrystallization initiation temperature of Cu-Cr-Zr bronze (550 °C) and reached 490 ± 30 °C.

Heat-resistant coatings from Ni-Cr and Fe-Ni-Cr for combustion chambers were applied by vacuum arc deposition from nichrome XH78T and steel 12X18H10T with the chemical compositions specified in Table 4 [11]. The distances from the cathode to a sample were 130–140 mm when coatings from nichrome XH78T were applied and 120 mm for coatings from steel 12X18H10T.

The coating thickness was measured by the gravimetric and metallographic methods.

The metallographic tests of the obtained samples were done using a MIM-10 microscope. The micro-

hardness of the coatings was measured for the transverse microsections using a microhardness gauge PMT-3 and a load of 100 g.

The microstructure of coating samples was determined by the electrolytic etching in an Electro-P installation using a 10 % solution of oxalic acid.

EXPERIMENT RESULTS

The relation between the structure and properties of Ni-Cr and Fe-Ni-Cr coatings and the pressure of reactive gas (nitrogen) in a chamber was analyzed in this work.

The microstructure investigations of heat-resistant protective coatings from Ni-Cr and Fe-Ni-Cr in different conditions of vacuum arc deposition showed that all the coatings were composed of a matrix of condensed substance and inclusions of the droplet phase (Figure 3), which corresponds to their known composition [1, 5]. Coatings from Ni-Cr and Fe-Ni-Cr have general relations between the generated structure and the pressure of reactive gas (nitrogen) in a chamber. The coating surface relief corresponds to the underlayer relief and defines the appearance of the formed crystals. When no nitrogen is used, the correspondence between the coating and underlayer surfaces decreases as the coating thickness increases. Coatings with the minimum roughness are formed in this way. The influence of the underlayer's relief on the orientation of the coating surface's relief is de-

Table 3. Conditions of the vacuum arc deposition of coatings

Sample No.	Coating	Chamber pressure [Pa]	Nitrogen supply	Duration [min]	Cathode arc current [A]	Cathode arc voltage [V]	Sample temperature [°C]	Coating thickness [micron]
1	XH78T	0.004	—	145	54	28	500–460	217
4	XH78T	0.13	min	180	55	30	500–360	228
5	XH78T	0.67	mid	180	55	28	480–330	184
3	XH78T	1.3	max	180	55	27	400–320	212
9	12X18H10T	0.01	—	90	82	31	520–480	245
12	12X18H10T	0.067	min	90	82	32	520–460	201
10	12X18H10T	0.13	mid	90	82	32	520–480	243
11	12X18H10T	0.67	max	90	82	29	520–460	201

Table 4. Chemical compositions of cathodes used in the experiments

Material	Ni	Cr	Ti	Mn	Fe	C	Si	P	S
XH78T	base	19–22	0.15–0.35	≤ 0.7	≤ 1.0	≤ 0.12	≤ 0.8	≤ 0.015	≤ 0.010
12X18H10T	9–11	17–19	5C–0.8	≤ 2.0	base	≤ 0.12	≤ 0.8	≤ 0.035	≤ 0.020

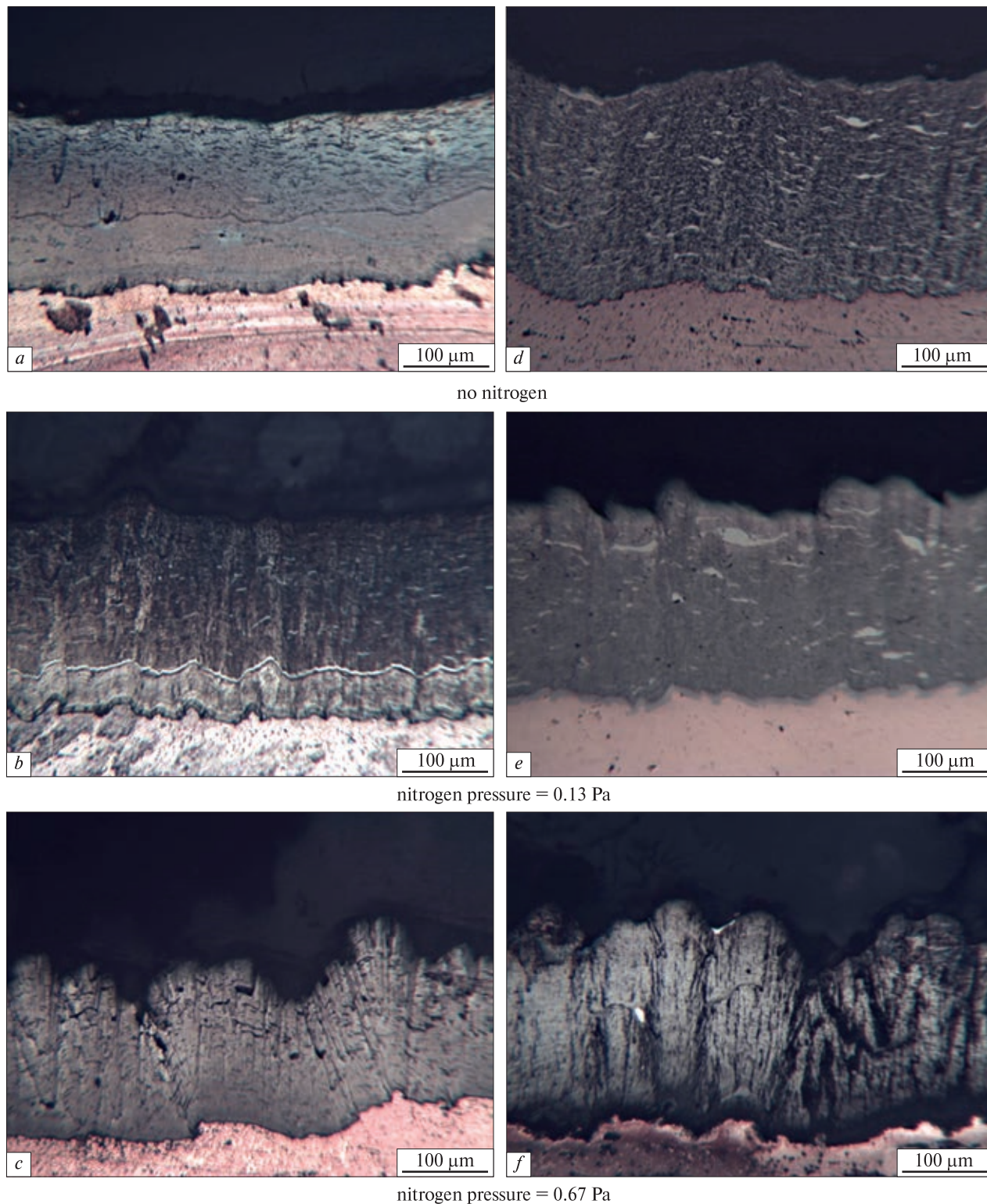


Figure 3. Typical structures of coatings depending on nitrogen pressure in a chamber: *a, b, c* — a coating from Ni-Cr; *d, e, f* — a coating from Fe-Ni-Cr

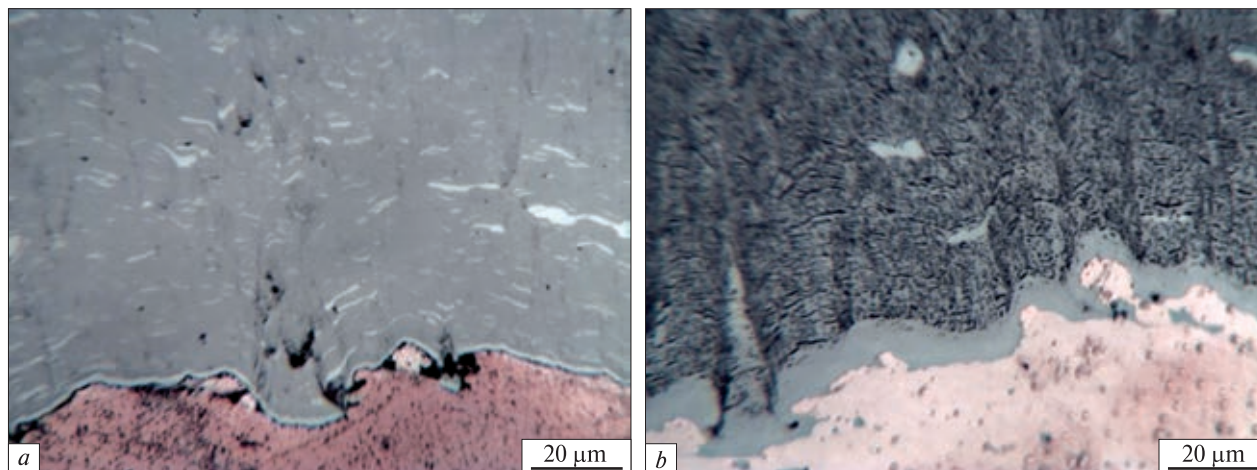


Figure 4. A coating's structure in the boundary with a bronze underlayer: *a* — Ni-Cr (5×10^{-3} torr), *b* — Fe-Ni-Cr (1×10^{-3} torr)

finied by the number and the shapes of the crystal origins and the specificity of crystal growth and interaction, which depends on the chemical composition of the coating. The primary consequence of this is the different roughness and continuity of coatings from Ni-Cr and Fe-Ni-Cr (Figure 3).

When nitrogen is not used, it gives the maximum diffusion mobility of atoms and coating formation in the conditions of the best crystal fusion and the minimum quantity of pores. Pores occur because of the effect of pore wedging-out, i.e., gradual thinning and stop of crystal formation because of the hindrance to growth formed by the surfaces of the crystals growing nearby (Figure 3, *a, d*). Increasing the pressure by supplying nitrogen results in weaker diffusion mobility of atoms, the separated growth of crystals, worse conditions of growth for the crystal surfaces, and greater volume alteration. This effect of nitrogen is more common for coatings from Ni-Cr than Fe-Ni-Cr (Figure 3). For relatively high pressure (0.67 Pa), the structures of Ni-Cr and Fe-Ni-Cr coatings are composed of columnar crystallites separated by large pores (Figure 3, *c, f*).

An intermediate layer is observed along the boundary between the primary structure and the underlayer in almost all coatings (Figure 4). In the initial phase of coating application by ion plasma spraying, the underlayer's surface is cleaned by ion bombardment that eliminates gases and heats the surface. During surface bombardment, the diffusion processes inten-

sify between the ions in the surface and the coating. Interaction between the underlayer material being sprayed and the depositing atoms produces intermediate layers, the properties of which differ from those of a coating applied without ion bombardment [13].

The microstructure investigation showed that, besides the amount of nitrogen being in direct proportion to its pressure in a vacuum chamber, the structures of the studied coatings depend also on the chemical composition of the cathode material (nichrome or stainless steel).

In Ni-Cr coatings, the droplet phase inclusions are more dispersed than in Fe-Ni-Cr coatings. This regularity is held for both zero and any nitrogen amounts. Increasing the content of nitrogen for both coating systems results in a smaller amount of such inclusions.

Therefore, the microstructure analysis of the investigated vacuum-arc coatings with different amounts of reactive gas (nitrogen) in a deposition chamber showed that two phases are present: a matrix and the droplets of various dimensions and shapes dispersed in that matrix. When the nitrogen pressure rises, the diffusion mobility of atoms decreases, which results in the formation of coarse-crystal, porous-columnar structures of coatings with high porosity, a varying volume, and a rough surface.

The influence of nitrogen in a chamber on coating strength was estimated by coating microhardness. Figure 5 shows a graph of the relation between the

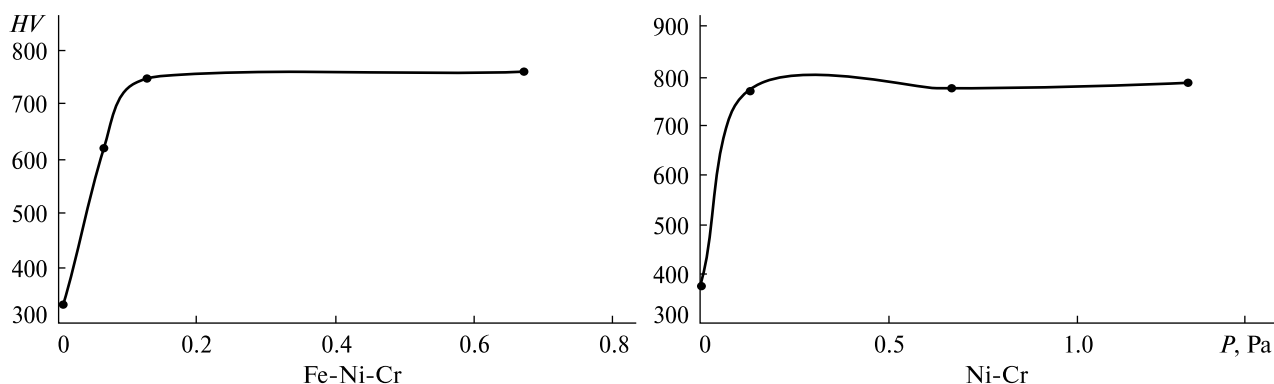


Figure 5. Microhardness of experimental coatings versus nitrogen pressure

microhardness of Ni-Cr and Fe-Ni-Cr coatings and the nitrogen pressure. The microhardness of Cu-Cr-Zr bronze before coating application is 160 HV and does not change within the investigated range of pressure in a chamber. Applying coatings from Fe-Ni-Cr without nitrogen makes it possible to increase the surface microhardness more than twice, up to 329 HV and 374 HV for coatings from Ni-Cr alloy. Supplying nitrogen at a pressure of 0.13 Pa gives a higher coating microhardness for both alloy systems, and further pressure gain has almost zero influence on microhardness. The reason may be in the occurrence of two competing processes: strength increase by nitrogenizing, which gives new strengthening phases, and the worsening of coating properties when the structure of condensates changes, i.e., the transition from a dense columnar structure with no space between the fused columns to a porous columnar structure composed of columnar grains with sharp peaks, separated by pores or cavities.

Nitrogenizing increases microhardness to 759 HV for Fe-Ni-Cr coatings and 785 HV for Ni-Cr coatings, i. e., roughly twice.

The relation between microhardness and nitrogen pressure in a chamber is similar for both coating systems. It is defined by structural alterations in the material. Both coating systems have similar microhardness within the entire range of the investigated parameters.

For the combustion chamber of a rocket engine, coatings from Ni-Cr demonstrate a better combination of properties compared to coatings from Fe-Ni-Cr. The structure of a Ni-Cr coating is more uniform, with no intermediate layer inside the coating

(Figure 3, *a, b*), and less prone to the formation of a columnar structure compared to a Fe-Ni-Cr coating at the same pressure in a vacuum chamber. Furthermore, the chemical composition of a Ni-Cr coating gives a higher limit of thermal loads.

A good coating from Ni-Cr with nitrogen strengthening is deposited as long as a uniform structure is produced until the occurrence of columnar macrocrystals. From the results of the experiments, a nitrogen pressure of up to 0.13 Pa can give this result.

The critical parameters of coatings for the combustion chamber walls in rocket engines are the resistance to oxidizing environments and the cost of initial materials. Naturally, nickel-chromium alloys with 80 % Ni are one of the most heat-resistant industrial materials with maximum operating temperatures of up to 1200 °C, surpassing heat-resistant steels by this parameter. However, the cost of nichrome is high, making coatings from Fe-Ni-Cr more attractive, providing that their heat resistance is adequate for the application temperatures. Therefore, it is sensible to investigate both Ni-Cr and Fe-Ni-Cr coatings further to develop a deposition technique that can give the necessary level of performance parameters.

The results of this work point out that the investigated systems are effective for making coatings with the possibility of adjusting their structures and properties by controlling process parameters.

CONCLUSIONS

1. Protective coatings from Ni-Cr and Fe-Ni-Cr show promise and may be used in the combustion chambers of reusable rocket engines. The method

of vacuum arc deposition for applying these coatings ensures their outstanding quality.

2. It was found from the results of the conducted investigations that vacuum arc deposition at a cathode arc current of 55 A for Ni-Cr and 82 A for Fe-Ni-Cr coatings, a cathode arc voltage of 28 to 32 V, an underlayer temperature of 490 ± 30 °C, and no nitrogen in the chamber gives coatings with no apparent orientation of crystallites and a microhardness reaching 330 to 374 HV.

3. When a reactive gas (nitrogen) is supplied to the chamber during coating deposition, the structure of a

coating changes: crystallite orientation occurs, a columnar structure is formed, and nitrogen atoms are included in the crystal lattice of the coating material, which results in a coating microhardness increase to 759–785 HV.

4. Nitrogen pressure control makes it possible to adjust the structure and properties of a coating during deposition. It was discovered that a nitrogen pressure of 0.13 Pa gives the optimum combination of a coating's strength and continuous structure with no pores and apparent columnar crystallites.

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Стаття надійшла до редакції 02.07.2024

Після доопрацювання 06.09.2024

Прийнято до друку 06.09.2024

Received 02.07.2024

Revised 06.09.2024

Accepted 06.09.2024

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ВАКУУМНО-ДУГОВІ ПОКРИТТЯ ДЛЯ КАМЕР ЗГОРЯННЯ РАКЕТНИХ ДВИГУНІВ

Досліджено захисні жаростійкі покриття на основі сплавів Ni-Cr, Fe-Ni-Cr, що отримано вакуумно-дуговим осадженням з використанням катодів зі сплавів ніхром ХН78Т і сталь 12Х18Н10Т. Мікроструктурний аналіз досліджуваних вакуумно-дугових покриттів за різного вмісту реакційного газу азоту в камері осадження показав наявність двох фаз — матриці з розподіленими в ній частинками краплинної фази різного розміру і форми. При вакуумно-дуговому осадженні покриттів на основі Ni-Cr та Fe-Ni-Cr при струмі дугового розряду на катоді — 55 А для Ni-Cr, 82 А для Fe-Ni-Cr, напрузі горіння дуги на катоді 28–32 В та температурі підкладки 490 ± 30 °С за відсутності азоту отримують покриття, структура яких не має вираженої орієнтації кристалітів, мікротвердість дорівнює 330–374 НВ. При підвищенні тиску азоту дифузійна рухливість атомів зменшується, що призводить до формування грубокристалічної «пористо-колончастої» структури покриттів з високою пористістю, несучільністю в об'ємі та шорсткістю поверхні. Вплив тиску реакційного газу азоту в камері на характеристики міцності покриттів оцінювали по їх мікротвердості. Додавання азоту до тиску до 0.13 Па призводить до підвищення мікротвердості обох покриттів, подальше збільшення тиску майже не впливає на мікротвердість. Встановлено, що тиск азоту до 0.13 Па забезпечує оптимальне поєднання міцності та суцільної структури покриттів без утворення пор та виражених стовпчастих кристалітів. Жаростійкі сплави Ni-Cr з 80 % Ni є одними із найбільш жаростійких промислових матеріалів з максимальною робочою температурою 1200 °С, однак їх вартість досить висока, що підвищує привабливість використання Fe-Ni-Cr за умови достатньої жаростійкості в робочому діапазоні температур. Тому доцільно проводити подальше дослідження як Ni-Cr, так Fe-Ni-Cr покриттів для розробки технології осадження, що забезпечить необхідний рівень функціональних параметрів. Регулювання тиску азоту дозволяє керувати структурою та властивостями покриття під час їх осадження.

Ключові слова: жаростійкі покриття на основі сплавів Ni-Cr, Fe-Ni-Cr, мікроструктура, мікротвердість.