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Study of the wear resistance of ion-plasma coatings based on titanium and aluminum and obtained by magnetron sputtering

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Abstract. The paper presents the results of metallographic researches and erosion tests of ion-plasma coatings (based on titanium, aluminum and their nitrides), which were formed on samples of 12Kh13 and EI961 blade steels. Erosion tests and studies of characteristics of obtained by magnetron sputtering coatings were carried out by using a set of research equipment UNU "Erosion-M" NRU "MPEI". It was found that the formed Ti/Al-TiN/AlN coatings increase the duration of blade steels erosion wear incubation period by at least in 1.5 times and have a layered structure with thicknesses of nitride layers 1.3–1.6 μm and intermediate metallic layers 0.3–0.5 μm, with a total thickness of coatings of 10–14 μm for 12Kh13steel samples and 19–21 μm for EI961 steel samples.

1. Introduction

It is possible to enhance the functional properties of traditional ion-plasma nitride coatings by alloying them with other elements and applying technological approaches that reduce the size of individual structural elements of the coating down to several nanometers.

Forming TiN coatings with aluminum and it's nitrides to create TiN/AlN nanostructured compositions allows to increase the operating temperature, reduce the coefficient of friction and, in general, increase the wear resistance of such coatings [1, 2].

It is in practical interest to apply such approaches to improving the functional characteristics of titanium nitride coatings, which are used to protect rotating blades of powerful steam turbines low-pressure part [3].

In the present work, the task was to obtain and to study the characteristics and erosion resistance of Ti/Al-TiN/AlN multilayer nanostructured coatings formed on 12Kh13 and EI961 steels, which are widely used for manufacturing rotating blades of the steam turbines last stages.

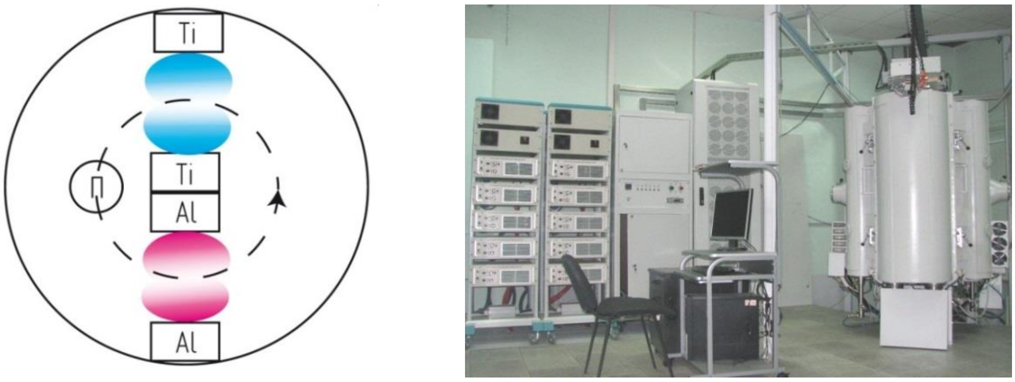
2. Techniques for the formation of ion-plasma coating and conducting metallographic researches and erosion tests

Ti/Al-TiN/AlN coatings were formed in the "Hephaestus+" experimental-industrial installation [4] by using planar unbalanced magnetrons. Titanium grade VT1-0 and aluminum grade A99 were used as the targets-cathodes. The coatings were applied according to the scheme shown in figure 1 on special 12Kh13 and EI961 steel specimens for erosion tests [5]. During the formation of coatings, the samples made a planetary motion. The rates of axial and planetary movement of the samples were 3 and 30 rpm, respectively. Argon and nitrogen of high purity were used as the plasma-forming and reaction gases. Preliminary treatment of the samples surface was carried out by electrolyte-plasma polishing in an aqueous solution of ammonium sulfate (6 %).

The process of ion-plasma formation of Ti/Al-TiN/AlN coatings included the following steps:

– vacuum chamber pumping out with preliminary heating of samples and technological equipment;
– ionic cleaning;
– formation of metal and nitride coating layers.

The regime parameters of the coating formation technological process main stages on the "Hephaestus+" installation are given below (see tables 1–3).



(a) **(b)**

Figure 1. Nanostructured coating formation scheme **(a)** and "Hephaestus+" installation general view **(b)**.

Table 1. Basic operating parameters of the pumping and preheating phase.

Stage duration, min	Voltage on the substrate, V	Total power of magnetrons, kW	Substrate temperature, °C	Pressure in the vacuum chamber, Pa
45–60	–	–	100–150	no more $8 \cdot 10^{-3}$

Table 2. Basic regime parameters of the ionic cleaning phase.

Stage duration, min	Voltage on the substrate, V	Total power of magnetrons, kW	Substrate temperature, °C	Pressure in the vacuum chamber, Pa
15–30	700–1200	–	no more 350	no more 0.2

Table 3. Basic regime parameters of the coatings formation stage.

Stage duration, min	Voltage on the substrate, V	Nitrogen consumption, l/h	Total power of magnetrons, kW	Substrate temperature, °C	Pressure in the vacuum chamber, Pa
300–400	60–100	0–5.4	16–24	no more 350	0.25–0.30

Determination of the formed coatings characteristics was carried out by using the complex of research equipment of the UNU "Erosion-M" of the NRU "MPEI", which allows carrying out the researches of the protective coatings composition, structure, thickness, microhardness and erosion resistance.

By using the TESCAN MIRA 3 LMU scanning electron microscope equipped with X Max 50 spectrometer (Oxford Instruments), the composition, structure and thickness of the coatings were studied. In addition to the general energy-dispersive microanalysis of the coating composition, its layer-by-layer analysis was performed on the GD Profiler 2 glow discharge optical spectrometer (Horiba Jobin Yvon) to obtain the distribution profiles of the elements in the coatings.

The metallographic sections were made by using a complex of preparation equipment, including: abrasive cutting machine with linear movable cutting system POWERMET 3000 (Buehler GmbH), electro-hydraulic press SIMPLIMET 1000 (Buehler GmbH), grinding-polishing machine BETA/1 (Buehler GmbH).

The microhardness of the coatings, as well as of the uncoated samples, was determined by using a DuraScan 20 (Emco-Test) hardness tester. The measurements were carried out according to the Vickers method at a load of 0.05 kgf (0.491 N).

Tests of the samples to the high-speed impact of liquid droplets resistance were carried out on the erosion test rig "Erosion-M". "Erosion-M" is a rotary-type testing rig that works as follows: two test samples are attached to the ends of a rod rotating in a vacuum chamber and cross each vertical rotation of liquid droplets emerging from a special droplet generator. At the "Erosion-M" rig it is possible to simulate various conditions for the interaction of liquid droplets with the surface of experimental samples: impact velocity, the size (diameter) of liquid droplets, the number of droplet flows, the attack angle. Within the framework of present research, erosion tests were carried out at a sample velocity of 300 m/s and a droplet diameter (distilled water) of 800 μm. To perform a comparative analysis of the erosion tests results, were plotted the dependences of the erosion wear average depth *E*, (m) on the liquid mass deposited on the eroded surface *m* (kg/m^2).

3. Results and discussion

As a result of the carried out experiments series were obtained Ti/Al-TiN/AlN coatings with a thickness of 7 to 21 μm. For all these coatings erosion tests were carried out and in this work presented the coatings with better erosion resistance.

Figures 2 and 3 show images of transverse sections and a characteristic view of the EI961 and 12Kh13 blade steels specimens surface with coatings.

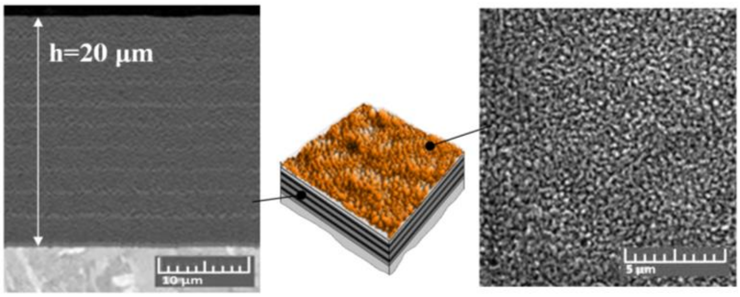


Figure 2. Image of the surface morphology and microstructure of the Ti/Al-TiN/AlN coating based on the EI961 specimen.

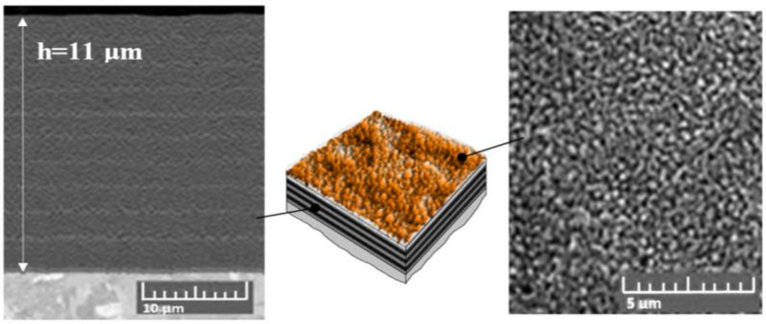


Figure 3. Image of the surface morphology and microstructure of the Ti/Al-TiN/AlN coating based on the 12Kh13 specimen.

It was revealed that the coatings have a layered structure, the thickness of the nitride layers is 1.3–1.6 μm, and the intermediate metallic layers 0.3–0.5 μm. The total thickness of the coating is 10–13 μm for 12Kh13 steel specimens and 19–21 μm for EI961steel specimens. The average thickness values of the obtained coatings are shown in table 4. The results of the elemental composition energy-dispersive microanalysis with the determination of the average content of elements in the coating are presented in table 5.

Table 4. Thickness of the formed coatings.

Sample	Sample description	*h*, µm
1	Ti/Al-TiN/AlN coating on EI961 steel	20.1 ± 1.3
2	Ti/Al-TiN/AlN coating on 12Kh13 steel	11.8 ± 1.7

Table 5. Results of elemental composition of coatings analysis.

Sample	Average content of elements in the coating, weight %						
	N	O	Al	Si	Ti	Cr	Fe
Ti/Al-TiN/AlN coating on EI961 steel	4.2 ± 1.3	–	31.2 ± 0.5	–	62.2 ± 0.8	0.3 ± 0.1	1.3 ± 0.2
Ti/Al-TiN/AlN coating on 12Kh13 steel	4.0 ± 1.1	–	29.6 ± 0.8	–	63.9 ± 0.9	0.4 ± 0.1	1.5 ± 0.2

It is not possible to determine the thickness of individual Ti (TiN) or Al (AlN) nanolayers, and the structure of coatings obtained under the conditions of current experiments is more similar to coatings obtained from targets (cathodes) based on Ti-Al alloys. The surface of the coatings has a granular structure with a characteristic grain size of 200–300 nm.

Figure 4 shows the profiles of the elements distribution along the thickness of coatings obtained on an optic-emission spectrometer of a glow discharge. In coatings, the aluminum content is about 30 %, titanium is about 63 %, nitrogen is about 4 %. In the upper layers of coatings, the nitrogen concentration reaches 7–9 % by weight. It also noted the presence of iron (1.3–1.5 %) and chromium impurities, which, possibly, fall into the coatings due to the spraying of the tooling certain elements.

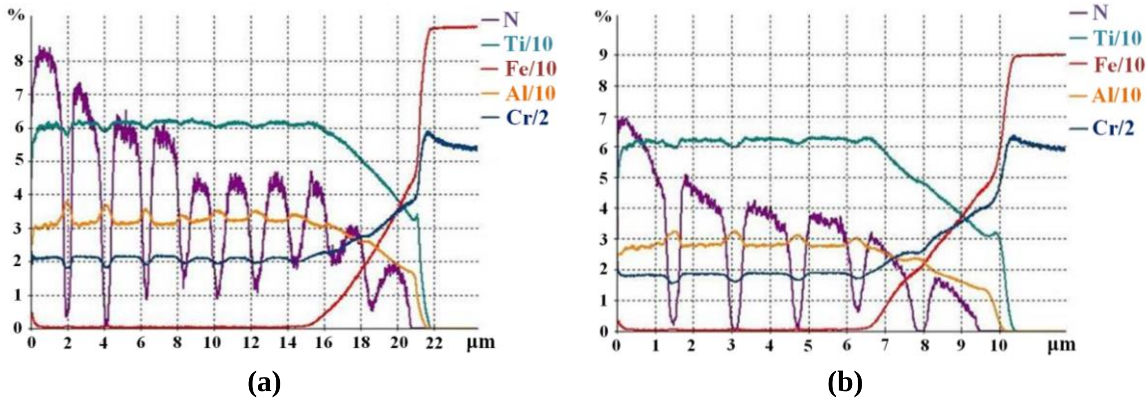


Figure 4. Profiles of the elements distribution in coatings on of EI961 **(a)** and 12Kh13 **(b)** steels specimens.

Table 6 presents the mean values of the microhardness of the surface of the EI961 and 12Kh13 blade steel specimens with and without coatings. The analysis of the data in table 6 shows that the formed coatings increase the microhardness of the surface up to 4–5 times compared with the reference level.

The results of erosion tests in the form of kinetic erosion curves ($E = f(m)$) are presented in figures 5 and 6. Analysis of the obtained dependences shows that Ti/Al-TiN/AlN coatings increase the incubation period of the erosive wear process of 12Kh13 and EI961 steels not less than in 1.5 times.

4. Conclusions

The Ti/Al-TiN/AlN coatings considered in present work are promising for solving the problem of improving titanium nitride coatings used to protect the powerful steam turbines wet steam stages rotating blades from water droplet erosion, but they will not solve the wear problem during all periods of this process. The solution of this problem, according to the authors' opinion, is possible in a combination of surface modification and the formation of ion-plasma coatings.

Table 6. Results of the mechanical properties study of 12Kh13 and EI961 steel specimens with Ti/Al-TiN/AlN coating and without coating.

Sample	Sample description	Vickers microhardness H0,05, HV
1	12Kh13 without coating	271 ± 5
2	EI961 without coating	280 ± 7
3	12Kh13 with coating based on TiAlN	1150 ± 30
4	EI961 with coating based on TiAlN	1140 ± 30

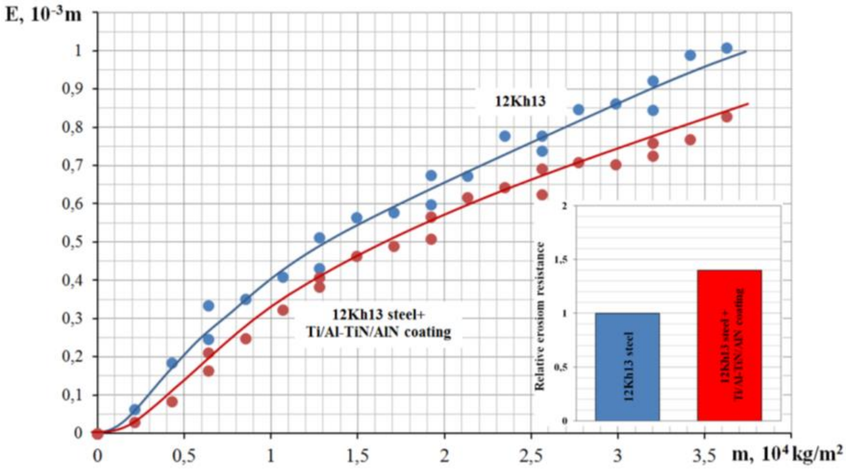


Figure 5. Curves of erosion wear of 12Kh13 steel uncoated and coated with Ti/Al-TiN/AlN at a collision velocity C_{imp} = 300 m/s and a droplet diameter d_d = 800 μm.

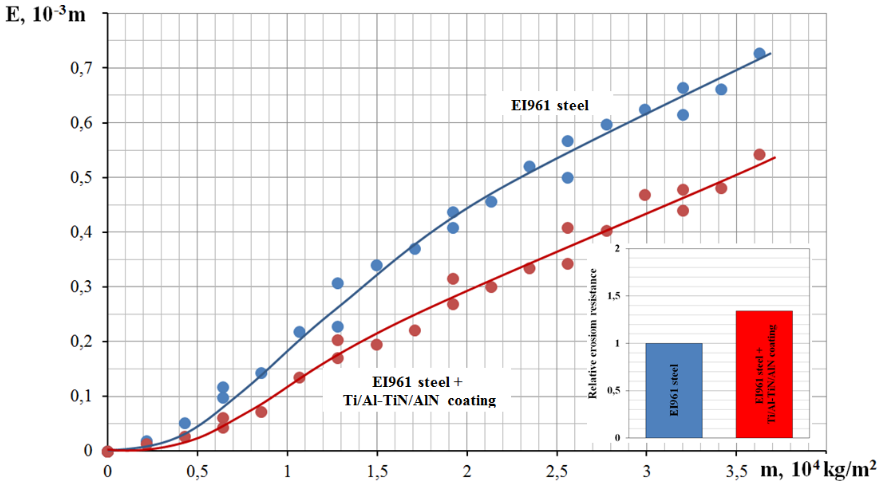


Figure 6. Curves of erosion wear of EI961 steel uncoated and coated with Ti/Al-TiN/AlN at a collision velocity C_{imp} = 300 m/s and a droplet diameter d_d = 800 μm.

Acknowledgements
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