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STRENGTH PROPERTIES OF 25CrMoV STEEL MODIFIED BY COMPLEX ION PLASMA TREATMENT WITH DEPOSITION OF INTERLAYER METAL COATINGS

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To improve erosion resistance, strength, and other protective properties, a comprehensive modification of the surface layers of 25CrMoV steel, which is widely used in turbine construction, was performed. For comparative studies, modifications with different interlayer materials (Mo and Ti) and modifications without interlayer were used. The Mo and Ti layers were deposited on a nitrided ion plasma surface. The outer protective layer for all modifications was unchanged and consisted of a Mo2N coating. To determine the role of the deposition of interlayer metal coatings on the strength properties of the complex modified coatings, the distributions of hardness (H, GPa), Young's modulus (E, GPa), and other strength parameters (H/E and H3/E2) measured by cross sections (h, μm) were investigated. The hardness of the Mo_2N coating was ~30 GPa, and the hardness of the nitrided layer was ~12 GPa. The modulus of elasticity for the Mo_2N coating was ~415 GPa, and for the nitrided steel - ~270 GPa. It was found that the main factor influencing the strength properties of a multilayer structure is related to the different materials of the metal layers. For the Mo and Ti layers, the values of E differ significantly (~ 340 GPa and ~ 180 GPa, respectively), with almost identical values of H (~ 6.5 GPa). The distributions of elastic modulus E = f(h) measured in the modified layers correlate well with the distributions of nitrogen concentration C_N = f(h). The distributions of H/E = f(h) and H^3/E^2 = f(h) for the modifications with Mo and Ti layers show a decrease in mechanical properties in the areas of the intermediate layers (Mo and Ti). For the modification without interlayer, the distributions of these indicators do not show such a drawback. The cavitation resistance of the comprehensively modified 25CrMoV steel is up to 2 times higher than that of the steel in the original condition. NSC KIPT performed extensive ion plasma modification on a pilot batch of turbine parts. These products, which are part of the steam distribution mechanisms, were manufactured by Ukrainian Power Machines JSC (Kharkiv) for the thermal power industry.
Keywords*: Complex surface modification; Steel; Ion-plasma nitriding; Vacuum-arc coatings; Hardness; Elastic modulus; Wear resistance; Strength*
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INTRODUCTION

An effective way to protect against various types of erosive wear is ion-plasma modification of the surface of structural materials, which can be realized both by ion-plasma nitriding [1, 2] and by deposition of vacuum-arc PVD coatings [3]. One of the disadvantages of nitriding, which limits the prospects of its application, is considered to be the low hardness of the nitrided surface compared to nitride coatings, which causes its increased erosive wear. Methods of ion-plasma treatment of working surfaces, including the combined effect associated with nitriding and deposition of hardening coatings, provide protection against a complex of various destructive factors [4, 5]. Thus, in order to increase the service life of parts of power engineering, where it is necessary to ensure both the strength of working surfaces against the effects of high mechanical loads and their various anti-erosion resistance, a complex ion-plasma modification of 25CrMoV steel was used [6, 7]. It consisted of surface hardening by ion nitriding followed by deposition of protective anti-erosion coatings. The advantages of such a complex treatment are the creation of a strong structure of modified surface layers with high interlayer adhesion properties. The structure of this layered structure with a hardness gradient in depth from the surface can be represented by modified layers: nitride coating with high hardness H ≥ 25 GPa and nitrided steel H ≥ 8 GPa. The initial value of hardness of the non-nitrided surface of 25CrMoV steel is ~ 4 GPa.

At the same time, metal layers are used to improve the adhesion of nitride layers to various substrate materials during vacuum arc deposition. Such layers are usually conveniently deposited from the same cathode material as the nitride layers. When metal films are used in a multilayer structure, the strength and performance of the structure may depend on the material and its mechanical properties.

The distributions of hardness (H, GPa), elastic modulus (E, GPa), and other strength indices (H/E and H^3/E^2) measured from cross-slits have been investigated to determine the role of interlayer metal coating deposition on the strength characteristics of complex-modified layers. The dependence of the distributions of these mechanical properties and nitrogen distributions on the distance across the interlayer boundaries (h, μm) was considered. For comparative studies intermediate layers of Mo and Ti metals were used. The metal layers were deposited on the surface of 25CrMoV steel nitrided by ion-plasma method. The outer protective layer for all variants of modifications was unchanged and consisted of Mo_2N coating. The erosion resistance of complexly modified steel samples with different composition of intermediate layers was investigated during cavitation tests.

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EXPERIMENTAL DETAILS

The ion-plasma treatment of samples and working surfaces of parts was carried out on the "Bulat" equipment, specially modernized for complex modification (Fig. 1).

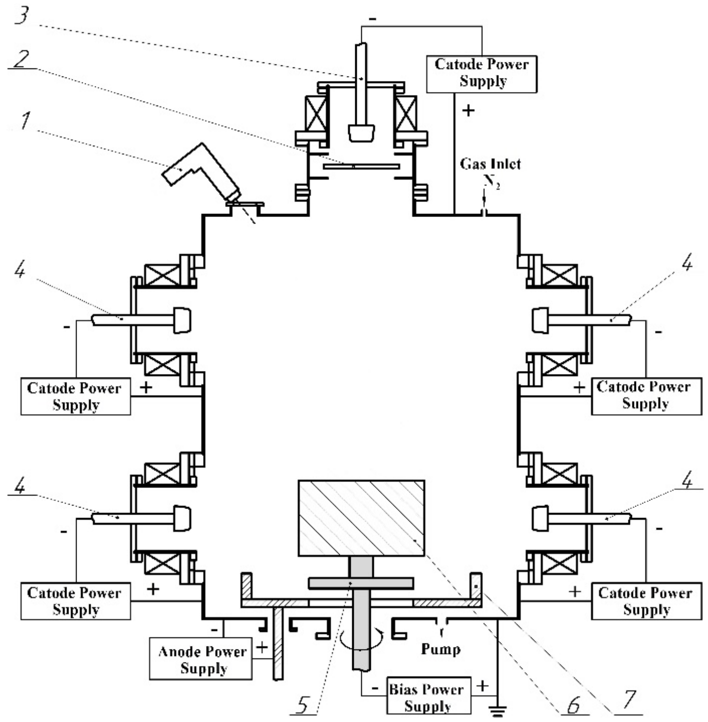

Figure 1. Scheme of the experiment: 1 - pyrometer, 2 - shield, 3 - plasma source of the first stage of two-stage discharge, 4 - plasma sources with Mo or Ti cathodes, 5 - rotation device, 6 - model of the part, 7 - electrode of the second stage of discharge

The dimensions of the vacuum chamber (600 mm × 650 mm × 850 mm) and the equipment of the unit (Fig. 1) allow both small and large parts weighing up to 15 kg to be subjected to complex processing. A detailed scheme of the experiment with placement of the part model and research samples inside the vacuum chamber is presented in [8]. 25CrMoV steel was subjected to surface modification. At the initial stage of the complex modification in the working chamber of the equipment ion nitriding of products in a two-stage vacuum arc discharge took place [9]. In this case, the pressure of the working gas, nitrogen, automatically supplied to the vacuum chamber was 2.7×10^{-1} Pa. The current density of gas ions accelerated by the potential - 500 V applied to the substrate was ~ 10 mA/cm^2. The temperature of the substrate was kept between 550 °C and 600 °C and monitored by a pyrometer. The nitriding time was 40 minutes. The next step in the complex process was the vacuum arc deposition of an interlayer coating of Mo or Ti metals. The deposition of this layer was performed under high vacuum conditions ~ 2.7×10^{-3} Pa. The thicknesses of the layers were 3 µm and 6 µm. Part of the experiments was carried out without deposition of the intermediate layer. In the final stage of the complex modification, a Mo2N protective coating with a thickness of ~ 10 µm was deposited at a nitrogen pressure of 3×10^{-1} Pa.

The following mechanical properties were used to study the strength properties of the modified layers:

H - hardness, GPa; E - Young's modulus, GPa;

H/E - coefficient characterizing the resistance of the material to mechanical action (elastic fracture deformation) [10] or its resistance to wear;

$H^3\backslash E^2$ - coefficient characterizing the material's resistance to plastic deformation [11] (it is used to assess the strength of coatings).

The study was conducted using the Nanoindenter G200 with a Berkovich diamond indenter. The G200 instrument meets the requirements of the international standard ISO 14577 [12]. Hardness and Young's modulus measurements of the modified coatings were performed on cross sections of the samples with the indenter passes at an acute angle (~ 10º) to the interlayer boundaries. A step-by-step indentation mode with 2 µm spacing and 200 nm nanoindenter penetration depth was used.

The distributions of nitrogen concentrations along the cross sections of the modified layers were studied using a scanning electron microscope (SEM) JSM 700-1F (Jeol, Japan) equipped with an X-ray energy dispersive microanalysis system.

Cavitation tests of samples with complex modified surface were performed on the apparatus [13]. The setup was an ultrasonic generator providing oscillations of the emitter at a frequency of 20 kHz. Erosion of the surface of the modified sample occurs when the device is operated in water, where a cavitation region is created under the end of the emitter. The total time of cavitation action on each sample was 6 hours. The degree of coating destruction was determined by weight loss (Δ p, mg) as a function of cavitation exposure time (t, hours).

RESULTS AND DISCUSSION

Fig. 2 shows hardness distributions (H, GPa) obtained by indenting the surface of cross sections of complexly modified 25CrMoV steel specimens with different thicknesses of Mo and Ti interlayer coatings. The vertical lines in the figure indicate the boundaries between the modified layers.

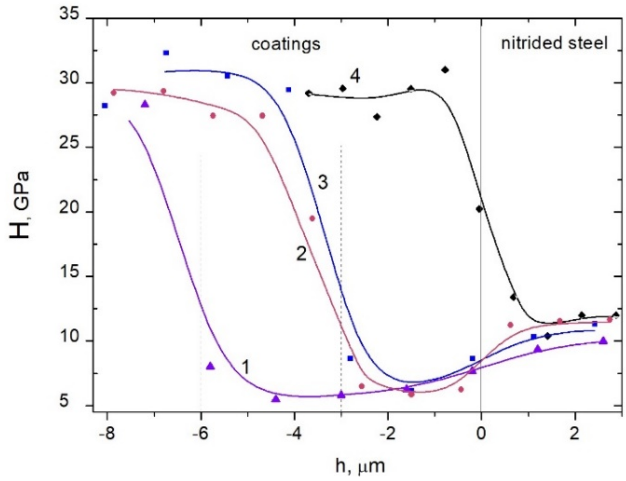

Figure 2. Hardness distributions along cross sections of modified coatings with Mo (1, 3), Ti (2) interlayers and no metal layer (4). The layer thicknesses are: 3 μm (2, 3) and 6 μm (1).

Figure 2. illustrates that all hardness distributions (1, 2, 3, and 4) exhibit similar characteristics. Curve 4, which represents the distribution for the sample without a metal layer, shows a sharp transition from the hardness values of the Mo2N coating (approximately 30 GPa) to the hardness of nitrided steel (approximately 12 GPa). Curves 2 and 3, which represent modifications with metal layers of the same thickness (3 μm) but different materials (Ti and Mo), are nearly identical. During these curves, we observe the same transition in Mo_2N hardness values. However, the transition occurs up to approximately 6.5 GPa in the metal layers section. To the right of the coating-nitrided steel interface, there is a further increase in hardness to the level of nitrided steel, which is approximately 12 GPa. The distributions for the same layer material (Mo) but with different thicknesses (3 μm or 6 μm) differ in the width of the section with a hardness of 6.5 GPa by a factor of almost 2. The differences in the distributions are due to the difference in the thicknesses of the metal layers.

Figure 3 displays the distributions of Young's modulus (E, GPa) in the modified layers and nitrogen concentrations (C_N, at. %) for steel specimens with different coatings as a function of distance h across the specimen slit.

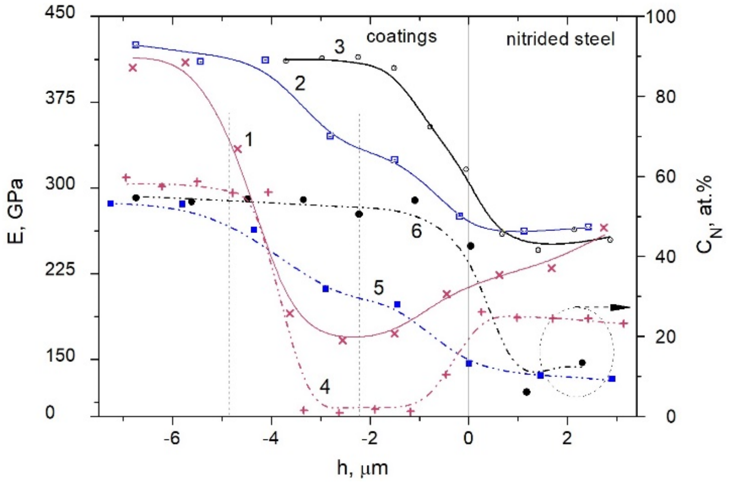

Figure 3. Distributions of Young's modulus (1, 2, 3) and nitrogen concentrations (4, 5, 6) along the cross sections of modified layers with Ti (1, 4), Mo (2, 5) and without metal layer (3, 6) coatings. The thicknesses of the layers (1, 2, 4 and 5) are 3 μm.

Fig. 3 shows that the Young's modulus distributions (1, 2, and 3) are nearly identical for both the Mo2N coating (approximately 415 GPa) and nitrided steel (approximately 270 GPa). The difference in the curves is due to the intermediate layer materials. Specifically, the E values for the Mo and Ti layers are significantly different, at approximately 340 GPa and 180 GPa, respectively. It can be observed from Figure 2 that the hardness of these layer materials is practically the same. Additionally, Figure 3 shows a strong correlation between the distributions of elastic modulus E = f (h) and nitrogen concentration C_N = f (h), indicating a direct relationship between the elasticity characteristics of the modified layers and their nitrogen content.

The nitrogen content in the Ti layer is low, at approximately 2 at.%. In contrast, the Mo layer has a significantly higher nitrogen content, at approximately 30 at.%. This difference can be attributed to the formation of Ti_xN compounds at the boundaries of the Ti layer, which reduces the mobility of N_2 and prevents its penetration into the layer. The Mo layer has a lower affinity for nitrogen than titanium. As a result, the hardness values of the Mo layer (Fig. 2 (3)) are lower compared to the high values typically found in molybdenum nitride compounds.

Figure 4 displays the distribution curves of wear resistance H/E and strength H3/E^2 at the boundaries of the modified layers for steel specimens with various interlayer coatings.

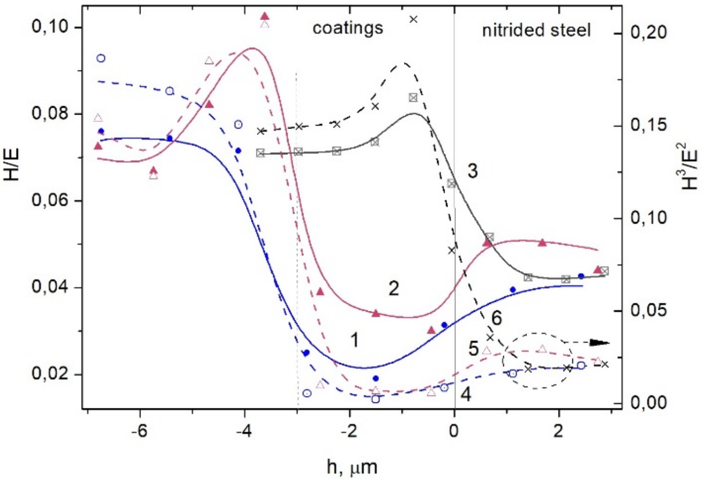

Figure 4. Distributions of H/E and H^3/E^2 ratios across the interlayer boundaries of modified layers using Mo (1, 4) and Ti (2, 5) coatings as well as without metal layer (3, 6). The thicknesses of the layers are: 3 μm.

Figure 4 shows the correlation between the wear resistance characteristics H/E = f(h) (1, 2, 3) and strength characteristics H^3/E^2 = f(h) (4, 5, 6) for the modified specimens with the same intermediate layers. The curves behave differently for the Mo, Ti, or no layer. For instance, the H/E ratio average for Mo layer material (1) is approximately 0.02, while for Ti layer (2) it is higher at around 0.035. The figure shows that the H/E values of the surface layer of nitrided steel for samples with Mo layer and without an intermediate layer (1, 3) are about 0.04, whereas for samples with Ti layer (2) it is significantly higher at around 0.05. The wear resistance characteristics of Mo_2N coating are high, with an index value of approximately 0.072 for all modifications (1, 2, 3). Curves 2 and 3 show maxima in the transition to Mo_2N sites, unlike curve 1. These maxima can be attributed to the formation of structural zones during the formation of transition layers. The thickness of these zones, as shown in the figure, is approximately 2 μm or more.

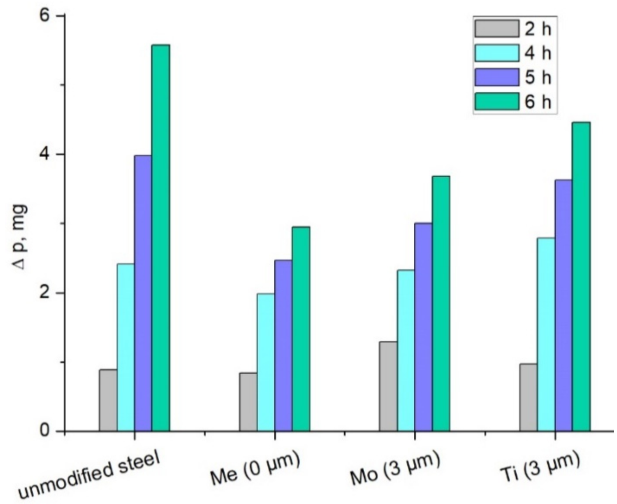

Figure 5. Weight loss during cavitation tests of unmodified steel samples and complex-modified samples with interlayers of different compositions

The wear resistance and strength of the modified steel layers depend on the material of the deposited metal layer. The analysis shows that modifications with Mo and Ti layers exhibit a decrease in mechanical properties in the areas of metal layers (Mo and Ti), which can affect the strength properties of the entire multilayer structure. It is important to note that H/E = f (h) and H^3/E^2 = f (h) distributions are also affected. Modifications without an intermediate layer at the transition section of the nitrided steel coating do not exhibit a decrease in characteristics.

The results indicate that when using a metal layer material in a multilayer structure, reducing the thickness of interlayer coatings by at least half can prevent a decrease in mechanical properties.

Figure 5 displays the weight loss diagram (Δp = f(t)) for the complex modified samples resulting from cavitation tests. Tests were conducted on modified samples of 25CrMoV steel with different coating materials (Mo, Ti or no coating) for 6 hours (t, hours). The Mo and Ti layers had a thickness of 3 μm.

As can be seen from Figure 5, the lowest wear up to the cavitation impact at each time stage of the test is characterized by the specimen that has undergone complex treatment without the deposition of a metal layer. Furthermore, the wear increases almost linearly from the sample with Mo layer to the sample with Ti layer. Therefore, the cavitation wear of the

Mo_2N layer for the complex modification with Ti interlayer is higher than that for the modification with Mo layer. The maximum wear is that of the untreated steel sample. As can be seen from the diagram, complex modification improves the surface resistance of 25CrMoV steel to cavitation-erosion wear up to 2 times. The highest erosion resistance is shown by the modification without interlayer coating.

Figure 6 shows images of the surface microstructure of the original steel and modified samples after 6 hours of cavitation testing.

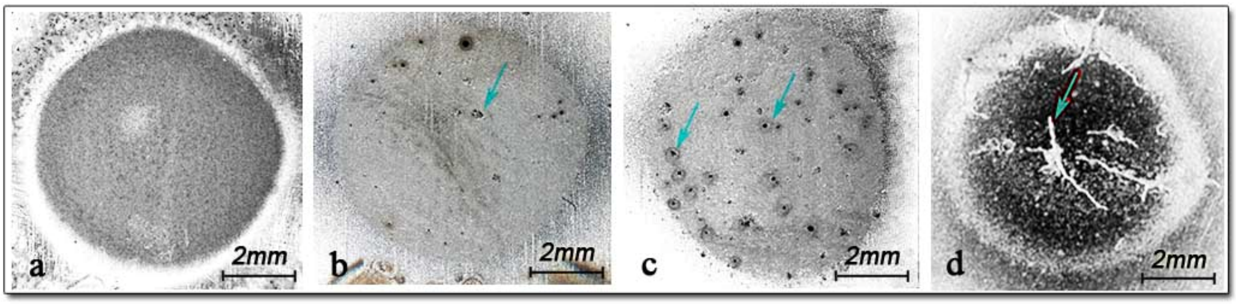

Figure 6. Surface microstructure of specimens after 6 h of cavitation tests:
untreated steel (a), Mo_2N coating without interlayer (b), Mo_2N with 3 µm Mo layer (c) and Mo_2N with 3 µm Ti layer (d).

Figure 6 illustrates that the wear characteristics of the surfaces differ qualitatively. The untreated steel surface (a) exhibits uniform erosive wear across the area, while the Mo_2N coating surface deposited without an interlayer (b) and with a Mo layer (c) is characterized by pitting wear, which occurs in the places where macroparticles of the cathode material are deposited [15]. The figure shows that the number of pitting failures is higher for the sample with Mo interlayer. The defects on the surface of the Mo_2N coating with Ti layer (d) after cavitation impact are different from those on the samples with Mo layer. In this modification, larger defects in the form of 'crack stars' are observed on the surface of Mo_2N.

Fractures are caused by the microshock action of cavitation bubbles, which apply pressure on the surface areas of the specimen, leading to their vibration and deformation [16, 17]. It is important to note that in multilayer structures, there is a softer metal layer beneath the hard coating of molybdenum nitride. As a result, the outer Mo_2N layer of such specimens' experiences more intense vibration and deformation. Furthermore, molybdenum nitride is known for its increased internal stress, which makes it quite brittle [7, 18]. These factors contribute to the formation of microcracks, which can lead to increased erosion into the material over time. Samples that are strengthened in a complex manner without metal coating do not have a soft layer and the process of microcrack formation is slower.

The nature of Mo_2N coating surface failures varies depending on the material of the intermediate layers (Mo or Ti). This can be attributed to the different physical and mechanical properties generated during the deposition process. The dependencies presented in Fig. 3 show that the intermediate layers have different values of Young's modulus: for Mo ~ 340 GPa and for Ti ~ 180 GPa. When the multilayer structure contains a more ductile titanium coating, the surface Mo_2N layer experiences greater deformations in the vibrating areas, resulting in larger-scale failures in the form of 'star-cracks' in the pitting areas.

The NSC KIPT utilized the complex ion-plasma modification method to strengthen a research batch of turbine parts. JSC Ukrainian Energy Machines, Kharkiv, manufactures these products for nuclear and thermal power engineering. The hardened parts are utilized in hinge joints and other friction pairs of steam distribution mechanisms.

Figure 7 displays images of steel parts that have been modified with Mo_2N coating deposition on the nitrided surface. The 'Pull rod' part (a) was hardened with Mo layer deposition, while the 'Axis' parts (b) were hardened with both modifications: with and without layer deposition.

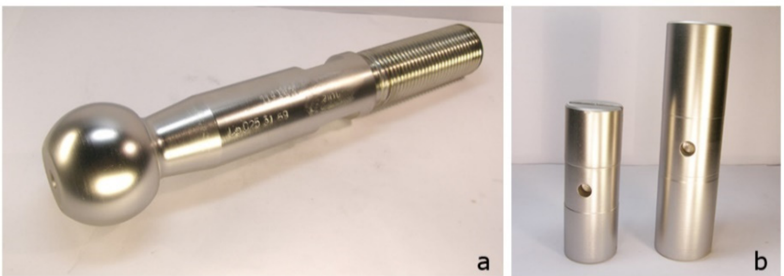

Figure 7. Image of parts "Pull rod" (a) and "Axle" (c), strengthened by complex method with Mo_2N deposition and different thicknesses of interlayer coatings.

Figure 8 displays the friction pair used in the steam distribution mechanisms of the K-325 turbine, consisting of the 'Axle' (located in the center) and the 'Insert' parts. The 'Axle' part is coated with Mo_2N, with an intermediate layer of Mo (0.7 µm) after ion nitriding. The 'Insert' part is coated with titanium nitride TiN, without an intermediate layer.

Figure 8. Images of parts ("Axis" - in the center and "Insert" - on the edges) of the friction pair included in the steam distribution unit of the K-325 turbine.

Table 1 shows the dimensional characteristics and types of protective coatings of parts strengthened by complex ion-plasma treatment.

Table 1. Specifications of parts strengthened by complex ion-plasma treatment.

Name of the parts	Overall dimensions (mm)	Material of parts	Weight (kg)	Protective coating
Pull rod	∅ 60×285	35CrMo	2.8	Mo_2N
Axle	∅ 40×150	25CrMoV	1.5	Mo_2N
Axle	∅ 35×98	25CrMoV	0.7	Mo_2N
Insert	∅ 35×25	25CrMoV	0.2	TiN

The strengthened parts were handed over to the manufacturer for installation in mechanisms working in operational conditions of steam turbines.

CONCLUSIONS

1. To enhance the protective characteristics of 25CrMoV steel, a complex ion-plasma modification was performed. This involved depositing external Mo_2N and interlayer coatings of either Mo or Ti on the surface.
2. The Mo_2N coating in the multilayer structure has a hardness of approximately 30 GPa, while the nitrided steel layer has a hardness of approximately 12 GPa. The Young's modulus E of these coatings is ~ 415 GPa and ~ 270 GPa, respectively.
3. The elastic properties of the interlayer coatings are the main factor influencing the wear resistance and strength properties of the modified layers. For Mo and Ti layers, the average E values are significantly different (around 340 GPa and 180 GPa, respectively), while the H values are practically the same (around 6.5 GPa).
4. The resistance of 25CrMoV steel surface to cavitation-erosion wear can be improved up to two times by making complex modifications. The modification without an interlayer has the highest resistance. The inclusion of a more ductile Ti layer in the multilayer structure leads to large-scale surface failures.
5. NSC KIPT performed ion-plasma strengthening of a research batch of parts for steam distribution mechanisms of turbines produced by JSC "Ukrainian Energy Machines". The strengthened parts will be installed in the mechanisms working under the operating conditions of steam turbines.

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ХАРАКТЕРИСТИКИ МІЦНОСТІ МОДИФІКОВАНОЇ КОМПЛЕКСНОЮ ІОННО-ПЛАЗМОВОЮ ОБРОБКОЮ СТАЛІ 25ХМ1Ф З ОСАДЖЕННЯМ МІЖШАРОВИХ МЕТАЛЕВИХ ПОКРИТТІВ

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З метою підвищення ерозійностійкості, міцності та інших захисних характеристик проведено комплексне модифікування поверхневих шарів сталі 25CrMoV, яка широко застосовується в турбобудуванні. Для проведення порівняльних досліджень використано модифікації з різними матеріалами проміжних шарів (Mo і Ti) і модифікації без проміжного шару. Шари з Mo і Ti осаджували на азотовану іонно-плазмовим способом поверхню. Зовнішній захисний шар для всіх модифікацій був незмінним і складався з покриття Mo_2N. Для визначення ролі осадження міжшарових металевих покриттів на характеристики міцності модифікованих комплексно шарів досліджено розподіли твердості (H, GPa), Модуля пружності (E, GPa) та інших показників міцності (H/E та H^3/E^2), виміряні за поперечними шліфами (h, μm). Твердість покриття Mo_2N склала ~ 30 GPa, а твердість азотованого шару ~ 12 GPa. Модуль пружності для покриття Mo_2N склав ~ 415 GPa, для азотованої сталі - ~ 270 GPa. Встановлено, що основний фактор, який впливає на характеристики міцності багатошарової конструкції, пов'язаний із різним матеріалом металевих шарів. Для Mo і Ti шару значення E істотно відрізняються (~ 340 GPa і ~ 180 GPa, відповідно), при практично однакових значеннях H (~ 6,5 GPa). Розподіли модуля пружності E = f(h), виміряні в модифікованих шарах, добре корелюють із розподілами концентрації азоту C_N = f(h). Розподіли H/E = f (h) і H^3/E^2 = f (h) для модифікацій з Mo і Ti шарами демонструють зниження механічних характеристик на ділянках проміжних шарів (Mo і Ti). Для модифікації без проміжного шару розподіли цих показників такого недоліку не мають. Кавітаційна стійкість комплексно модифікованої сталі 25CrMoV до 2 разів вища, ніж сталі у вихідному стані. В ННЦ ХФТІ було проведено комплексну іонно-плазмову модифікацію дослідної партії деталей турбін. Ці вироби, що входять до механізмів паророзподілу, виготовлені АТ "Українські енергетичні машини" (м. Харків) для теплової енергетики.

Ключові слова: *комплексна модифікація поверхні; сталь; іонно-плазмове азотування; вакуумно-дугові покриття; твердість; модуль пружності; зносостійкість; міцність*