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CORROSION-RESISTANT COMPLEX-STRUCTURED COATINGS FOR ALUMINIUM ALLOYS

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Abstract. *This paper presents the results of a study of the microhardness and corrosion resistance of a number of protective coatings applied to the surface of D16 alloy products. Various protective coatings (chromium, chromium nitrite, and a chromium nitrite layer with a chromium layer deposited on top) are proposed, each with varying degrees of Vickers microhardness (from HV = 80 to HV = 1114) and corrosion resistance. A chromium layer deposited on the surface of D16 alloy is shown to have very low hardness (HV = 80). A chromium nitrite layer deposited on the surface of D16 alloy is determined to have high hardness (HV = 655), but very low corrosion resistance. A double protective coating consisting of a chromium nitrite layer deposited on top of a chromium layer is shown to have satisfactory hardness (HV = 236) and corrosion resistance. It was found that the introduction of a highly hard layer between the surface of a D16 alloy part and an external chromium layer exceeds the surface hardness of the D16 alloy parts several times. It was shown that treating protective chromium-containing coatings with active oxygen species significantly increases the corrosion resistance and hardness of these coatings.*

Keywords: coating, microhardness, corrosion, corrosion resistance, chromium, chromium nitrite.

1. Introduction

Metal corrosion is one of the main processes leading to the destruction of metal structures and machine parts [1]. Under certain external conditions, the corrosion process can either accelerate or slow down. Land areas bordering seas and oceans are zones of increased corrosion due to the aggressive chemical composition of sea air [2, 3]. Halogen ions are the main factors in the destruction of metal products in seawater and in the atmosphere of coastal areas [4, 5]. One of the most widely used classes of metal alloys is duralumin alloys. Products made from these alloys are lightweight compared to steel parts and have sufficient strength [6].

Aluminium alloy D16 (duralumin, duralumin) belongs to the group of duralumin alloys with a high copper content. Close analogues of alloy D16 are 2024, $AlCu_4Mg_1$, $AlCuMg_1$, AA2024, AA2124. Alloy D16 is similar in composition to the group of aluminium-magnesium alloys - magnalium's of the AMg1.5 type (AMg-aluminium-magnesium alloys). The percentage of magnesium in the D16 sample is 1.2-1.8%, which allows this alloy to be classified as belonging to the AMg1.5 group of alloys (Table 1) [7]. The D16 alloy, which has a low density and high strength for aluminium alloys (Table 2) [8-10], is widely used in various fields of technology, such as aircraft construction [11]. In [10], it is noted that magnesium in AMg alloys is present in the form of Mg_2Al_4 groups, which are a rhombic subsystem of aluminium cells. It has been shown that the more aluminium cells contain Mg_2Al_4 groups, the higher the mechanical strength of the alloy. In AMg1.5 group alloys, the number of cells containing this group averages 18% of the total number of

aluminium cells [10]. It has been shown that a 1% increase in the number of cells containing the Mg_2Al_4 group in an aluminium alloy increases the strength of the aluminium alloy by 5.83 MPa. It has been shown that the structural formula of the Mg_2Al_4 compound is a rhombic system (Figure 1, a). Replacing two aluminium atoms in the rhombic subsystem with magnesium atoms significantly increases the strength of the alloys [10].

Table 1. Chemical composition of magnalium alloys (wt.%)

	Al	Mn	Cr	Ti	Cu	Zn	Mg	Fe	Si
D16	90,9-94,7	0,3-0,9	0.1%	0.155	3,8-4,9	0.25%	1,2-1,8	0.5%	0.5%
D16*	91.8%	91.8%	0.1%	0.155	4.3%	0.25%	1.6%	0.5%	0.5%
AMg1,5	96,45-98,9%	0,1%;	0,1%	-	0,2%;	0,25%;	1,1-1,8%	0,5%	0,5%
D19	91,095-94%	0,5-1%	0.1%	0.1	3.8-4.3	0.1	1.7-2.3%	0,5%	0,5%

* Calculated mass percentages of elements in alloy D16 based on the assumption that the number of copper atoms in alloy D16 is equal to the number of magnesium atoms.

Alloy D16 differs from the simple AMg1.5 alloy in that it has a significantly higher copper content, which leads to a significant increase in strength and hardness (Table 2). In the D16 alloy, the number of magnesium atoms is approximately equal to the number of copper atoms. In the D19 alloy, which is an improved version of the D16 alloy, the number of copper and magnesium atoms is actually equal, which has increased the impact strength of this alloy [12]. Considering that aluminium atoms are trivalent, while magnesium and copper atoms are divalent, it was concluded that in the D16 alloy, aluminium cells contain the $MgAl_4Cu$ group instead of the Mg_2Al_4 group. One magnesium atom in the rhombic subsystem of the aluminium lattice is replaced by a copper atom (Figure 1, b).

Table 2. Mechanical characteristics of aluminium alloys

	Tensile strength, MPa	Yield strength, MPa	Brinell hardness (HB), Kgs/mm^2	Vickers hardness (HV)
Pure aluminium	70	25	25	26.31579*
AMg1	140	50	30	31.57895*
AMg2	170	80	45	47.36842*
AMg3	200	100	58	61.05263*
AMg4	250	120	75	78.94737*
AMg5	280	150	65	68.42105*
AMg6	320	160	100	105
D16	440	300	114*, 125 [7]	120 [13], 132*
D19	460	340	125 [7]	132*

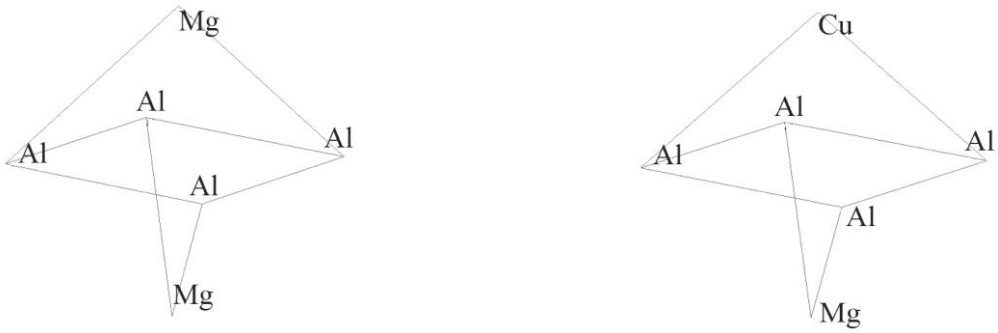
* Data is given in accordance with the formula HB=0.95* HV.

Increasing the copper content in the D16 alloy created a problem in that it reduced corrosion resistance. At the same time, the D16 alloy does not have a sufficiently high surface hardness (according to Vickers, HV = 120-132, Table 2). It is known that products made from the D16 alloy have insufficient corrosion resistance to seawater [14]. To protect against corrosion damage, these products are usually coated with protective coatings, such as varnishes [15], which creates problems during their use. For example, over time, protective varnishes deteriorate and flake off; protective coatings, such as bitumen, can also ignite.

However, the corrosion resistance of these alloys is low, and therefore various research groups are working to increase the corrosion resistance of products made from these alloys. One way to improve corrosion resistance is to apply inorganic protective coatings to aluminum alloy products [1]. In this case, it is important to increase the hardness of the protective coatings. Technologies for strengthening the surface layers of parts and creating protective coatings with high physical, mechanical, and chemical properties are a widely researched topic. These methods are discussed in the review article [1].

For example, reviews [16] the most promising innovative technology for surface hardening of aluminium alloys – plasma electrolytic oxidation (PEO). It considers the possible conditions and mechanisms for the formation of protective coatings on the surface of aluminium alloys. The influence of the main parameters of PEO treatment (electrical parameters, electrolyte composition and concentration, influence of alloying elements) on the structure and properties of oxide-ceramic coatings is studied. The qualitative characteristics of the surface layer of samples and finished products made of aluminium alloys demonstrated the effectiveness

of the PEO technology, which allows obtaining ceramic coatings with high hardness, strength, and increased wear and corrosion resistance.



a) b)

Fig.1. Diagram of the possible arrangement of atoms in an elementary cell: a) Rhombic subsystem of the AMg alloy containing a solid solution in the form of Al_4Mg_2; b) Rhombic subsystem of the AMg alloy containing a solid solution in the form of $MgAl_4CuMgAl_4Cu$.

Possible areas of application for highly effective technologies for applying protective PEO coatings to the surfaces of aluminium alloy products are proposed. Another example of the development of protective coatings is the work [17]. It proposes a new method of high-dose ion implantation of chromium and oxygen ions for the anti-corrosion treatment of VNS-5 aviation steel. The resulting protective layer of chromium oxide with a thickness of up to 100 nm is rich in Cr and poor in Fe, which slows down oxidation. The results of the work showed an improvement in corrosion resistance without compromising mechanical properties, which is especially useful for marine vessel parts.

The aim of this work was to develop protective coatings for aluminium alloys based on chromium, which are both corrosion-resistant and highly durable. The cathodic sputtering method was used for production, followed by additional oxidation of the protective coating. This made it possible to obtain coatings with increased hardness and corrosion resistance.

2. Materials and experimental details

The study used samples of D16 aluminum alloy measuring 15 mm × 10 mm × 4 mm. Before being loaded into the working chamber, the samples were degreased in isopropyl alcohol. In the working chamber, before coating, the surface of the samples was ionically cleaned using a plasma source with a hot cathode (PINK) [18]. In order to increase the corrosion resistance of the surface of the D16 samples, chromium or chromium nitride with a thickness of 2 μm was applied to their surface. Chrome target ERKh 99.95 (TU 14-22-138-2000) were used. When chromium films were applied to the surface of the chromium nitride film, the thickness was 0.5 μm. The films were applied by cathodic sputtering with an accelerating voltage of 1000 volts on the measuring setup described in [18, 19]. The chromium films were obtained in an argon atmosphere with a chamber pressure of 0.5 Pa. During the process of obtaining chromium nitride coatings, the working gas in the working chamber is changed from argon to nitrogen. The working gas pressure in the chamber was 0.5 Pa.

Samples with protective coatings were placed in a working chamber and heated to 185 °C in an atmosphere for 12 hours, while the atmosphere was exposed to ultraviolet (UV) radiation. More details on the treatment method are provided in articles [10, 20].

For the purposes of this article, the following abbreviations were chosen for the names of samples subjected to various treatments:

D16_X – a layer of chromium sprayed onto the surface of a product made of D16 alloy;

D16_X_U12 – a layer of chromium sprayed onto the surface of a product made of D16 alloy and then exposed to air for 12 hours, subjected to UV radiation (further processed samples);

D16_(N+X) – surface of the D16 alloy covered with a layer of chromium nitride;

D16_(N+X)_U12 – surface of the D16 alloy covered with a layer of chromium nitride and then subjected to 12 hours of treatment;

D16_(N+X)_X – a layer of chromium nitride is sprayed onto the surface of the D16 alloy, then a layer of chromium is applied to the chromium nitride layer;

D16_(N+X)_X_U12 – a layer of chromium nitride is sprayed onto the surface of the D16 alloy, then a layer of chromium is applied to the chromium nitride layer and subjected to 12 hours of treatment.

In these designations: X – application of a chromium film to the surface of the sample, U12 – treatment of the sample surface with ultraviolet radiation for 12 hours, N+X – chromium nitride films applied with the addition of nitrogen to the working chamber. A schematic representation of the process of applying protective coatings and the process of their treatment is shown in Figure 2.

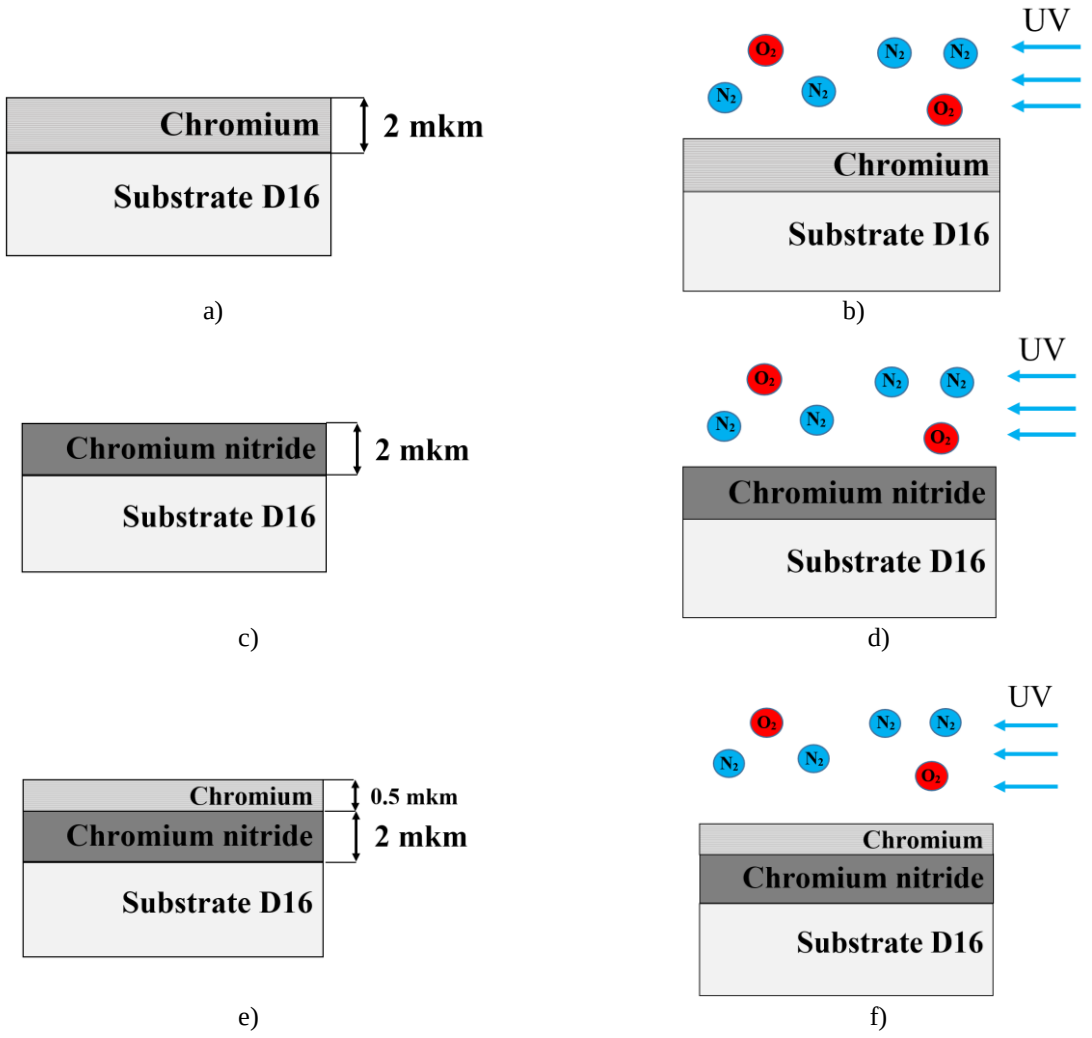


Fig.2. Schematic representation of the process of applying protective coatings and the process of their treatment: a) D16_X; b) D16_X_U12; c) D16_(N+X); d) D16_(N+X)_U12; e) D16_(N+X)_X; f) D16_(N+X)_X_U12.

The surface morphology of the synthesized films was investigated using a scanning electron microscope (SEM) (MIRA 3LMU, TESCAN) fitted with an energy-dispersive X-ray analyser (EDX, INCAPentaFET x3, Oxford Instruments, UK).

The microhardness of the obtained samples was measured using an HVS-1000A microhardness tester. A load of 100 g was used. The enlarged surface areas were also analyzed using an optical microscope of the HVS-1000A microhardness tester. Next, to test corrosion resistance using the method described in article [10], samples D16 and D16, coated with various protective films, were placed in a test solution simulating the corrosive effect of seawater for a control period (72 hours). A 5% solution of iodine in ethanol was used as the test solution. The mass of the sample was measured before and after placement in the solution. The results are given as mass percentages of mass loss, where the mass of the sample before placement in the solution is taken as 100%. Information is also provided in the form of the number of atoms lost from the surface of the samples as a result of corrosion. For this purpose, the mass loss was divided by the mass of one atom of the protective coating. The mass of samples was measured on electronic scales RADWAG AS 60/220.R2 with an accuracy

of 10^{-4} grams. The area of corrosion damage was estimated from measurements of areas affected by corrosion and without corrosion, obtained using an HVS-1000A optical microscope microhardness tester.

Image scaling and the subsequent measurement of structural elements were carried out in ImageJ following preliminary calibration using a reference object.

3. Results and discussion

3.1 Surface morphology of protective coatings based on chromium and chromium nitride

The surface morphology of the D16_(N+X), D16_(N+X)_U12, D16_(N+X)_X and D16_(N+X)_X_U12 samples is shown in Figures 3, a-d. Figures 3, a-d show the formation of a uniform structure on the surface of the D16 samples. The films are homogeneous, free from cracks and significant defects. Particles ranging in size from 50 nm to 2.5 μm are also present on the film surface. However, their proportion is not significant compared with the film surface without particles. The formation of particles is associated with the film deposition parameters and the use of an industrial cathodic metal sputtering system [18]. Treatment of the samples in an oxygen atmosphere under UV irradiation does not lead to significant changes in the film structure.

The map of elemental distribution across the sample surface on the Figure 4 shows the formation of a homogeneous film with no changes in stoichiometric composition across the sample surface. The remaining samples exhibit similar film quality on the surfaces of the D16 samples. EDX analysis determined the stoichiometric composition of the films to be $Cr_{2.7}N$. UV treatment of the film surfaces does not result in any change to their stoichiometric composition.

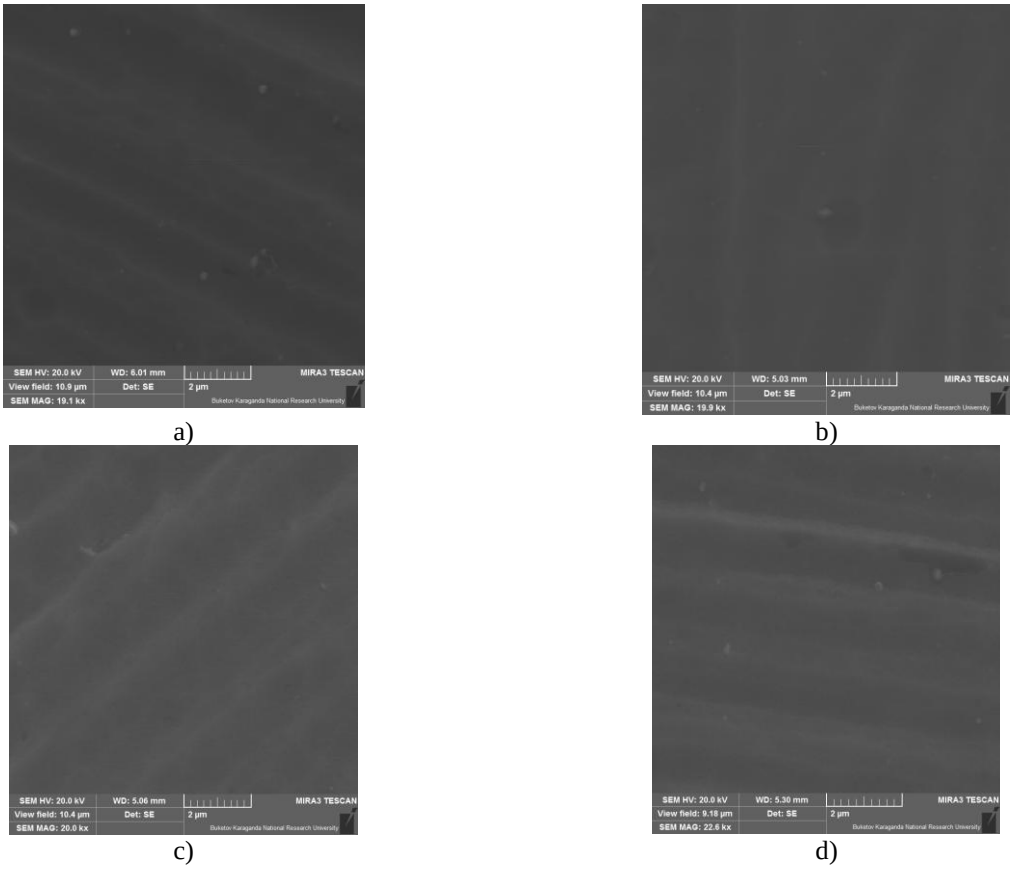


Fig.3. Surface morphology of coatings obtained by SEM: a) D16_(N+X); b) D16_(N+X)_U12; c) D16_(N+X)_X; d) D16_(N+X)_X_U12.

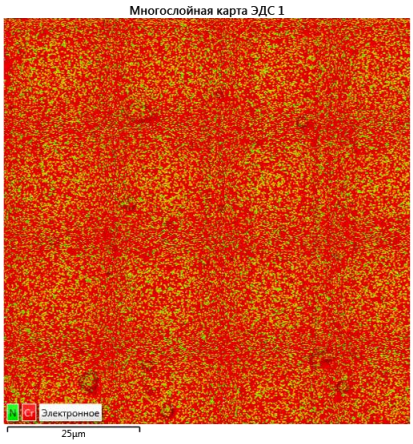


Fig.4. Map of distribution of elements on the sample surface of D16_(N+X).

3.2 Chromium-based protective coatings treated with ultraviolet light

Initially, in order to increase corrosion resistance, the surface of sample D16 was coated with a 2 μm layer of chromium. Next, to test corrosion resistance using the method specified in [10], samples D16 without additional protective coatings and D16 coated with chromium were placed in a test solution for a control period. The experiment showed a significant reduction in the corrosion rate in the test solution. The number of atoms lost by the D16 sample as a result of applying a layer of chromium to the surface decreased by 1.65 times (Table 3). However, a significant decrease in the surface hardness of the sample coated with pure chromium (1.5 times) was also recorded.

Table 3. Summary information on the loss of atoms in the samples studied, the area of corrosion damage, and the microhardness of D16 samples without coatings and with various protective coatings.

Number of ator lost by the surface due to corrosion	D16	D16_X	D16_X_U12	D16_(N+X)	D16_(N+X)_ U12	D16_(N+X) _X	D16_(N+X)_ X_U12
Number of ator lost by the surface due to corrosion	$9{,}868*10^{20}$	$5{,}992*10^{20}$	$4{,}072*10^{20}$	--	---	$5{,}611*10^{20}$	$4{,}19*10^{20}$
Surface hardne according to Vickers (HV)	150	80	120	655	1114	236	350
Area of corrosion damage*	-	30%	5%	53% .	6%;	23%	12%
Sample mass loss (%)	-	2,66%	1,81%	4,12 %	2,56%	2,49 %	1,86%

* Determined as a percentage of the total area of the micrograph at 160x magnification.

In [20], a significant increase in the corrosion resistance of martensitic stainless steel X17 was revealed as a result of treating the surface of stainless-steel samples with ozone (ozone was generated by exposing oxygen molecules in the air to ultraviolet radiation). It was shown that the corrosion resistance of martensitic stainless steel X17 increased by 71% due to a significant increase in the number of chromium-oxygen-chromium bonds (the oxygen content on the surface increased 5.71 times from 0.7 to 4 mass percent). Based on the results of [20], it was assumed that the corrosion resistance of D16 samples coated with chromium could increase after prolonged treatment of the surface of these samples with air exposed to UV radiation.

To verify this assumption, D16 alloy samples coated with a 2 μm thick layer of chromium were treated with air containing active oxygen species (AOS) at a temperature of 185°C for 12 hours. Each of the sample surfaces of D16_X and D16_X_U12 was treated for 12 hours. As a result of corrosion resistance testing, it was found that the corrosion rate of D16 samples coated with chromium and further processed by AOS decreased

by 32% in mass compared to the corrosion rate of D16 alloy samples coated with chromium (1.47 times) (Table 3). At the same time, the surface hardness of the samples coated with chromium and further processed by AOS increased by 33%.

Figure 5 shows photographs of D16 alloy samples coated with a layer of chromium after being kept in the test solution for a control period of time. There is a large difference in the area of damage to the D16 alloy samples. On samples coated with chrome and treated with AOS D16_X_U12, a dozen pinpoint corrosion pits are observed. On the surface of the sample coated with chrome D16_X without further treatment, moderate corrosion damage is observed: the number of pinpoint pits is an order of magnitude greater than on the sample subjected to prolonged treatment AOS.

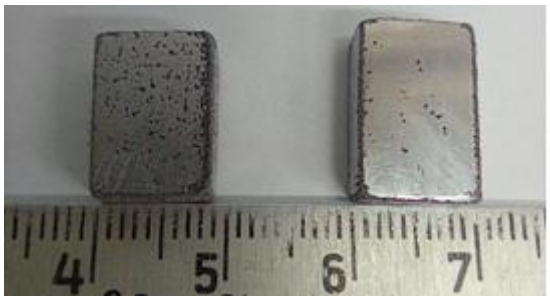
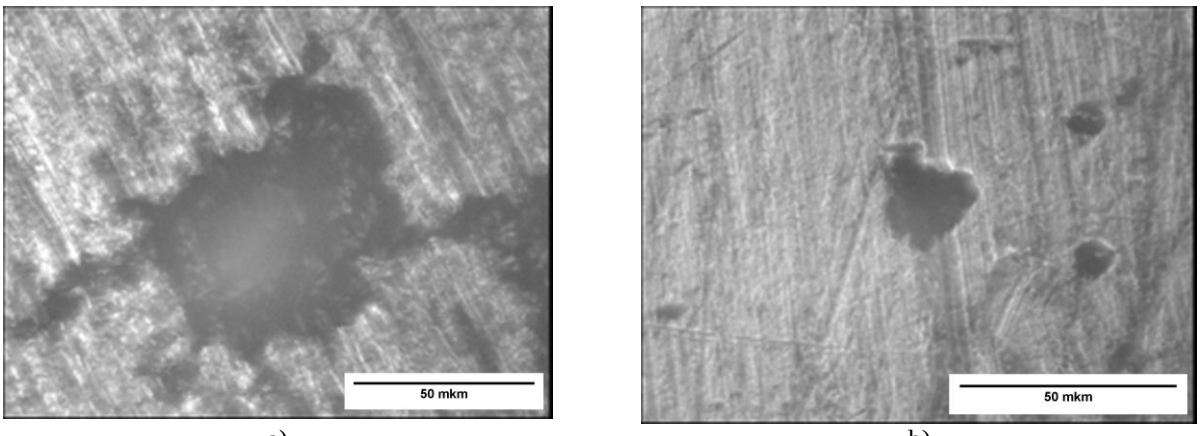


Fig.5. Photographs of D16 alloy samples with a protective chrome film after exposure to the test solution for the control time. On the left is a D16 sample coated with a layer of chrome. On the right is a D16 alloy sample coated with a layer of chrome and then subjected to 12 hours of treatment with air exposed to UV radiation.

Figure 6 shows micrographs of the surface of D16 alloy samples after exposure to the test solution for the control time. It is clearly visible that the total area of corrosion damage has significantly decreased on the D16_X_U12 sample, which was subjected to prolonged treatment. For these samples, the size of individual corrosion lesions has significantly decreased. The area of a single corrosion lesion is 5% of the studied area of the treated sample D16_X_U12 and 30% of the sample area D16_X if no treatment was performed. On average, the radius of individual lesions on ozonated samples D16_X_U12 decreased 6 times compared to samples D16_X coated with chromium without further treatment. Also, on the treated samples D16_X_U12, there are no destruction channels outside the main corrosion ulcer. It can be seen that outside the corrosion ulcers, the surface microstructure has practically not been affected. The absence of damage to the surface microstructure indicates that the destruction of the chrome-coated surface begins with isolated foci, which increase in area and depth over time. Iodine ions interact with chromium atoms and D16 alloy atoms at the edges of the primary corrosion hole, gradually increasing its area.



a) b)

Fig.6. Samples D16_X and D16_X_U12 after exposure to the test solution for the control time: a) D16_X; b) D16_X_U12. Samples magnified 160 times.

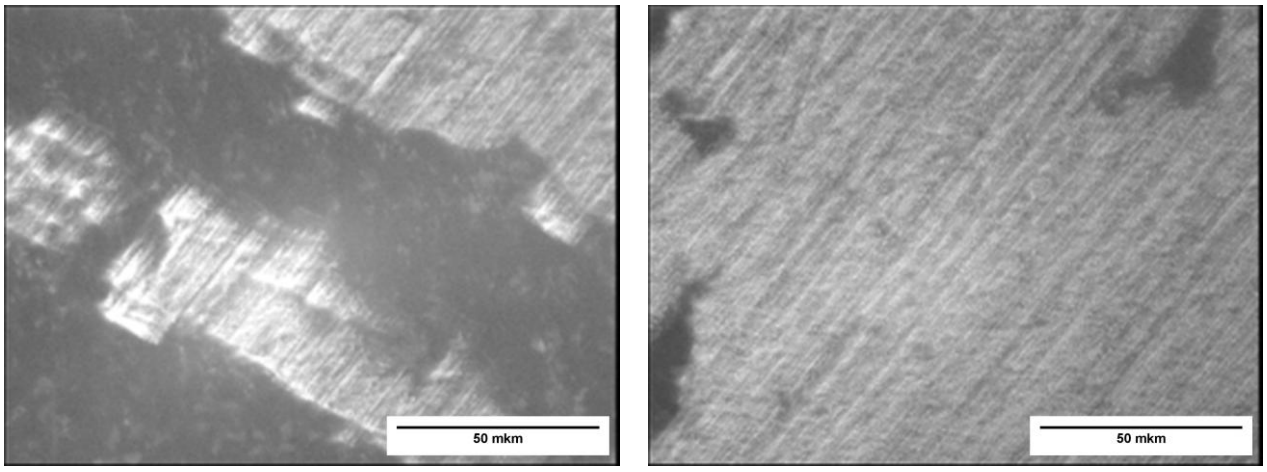
Considering the above, it can be concluded that samples coated with a layer of chromium D16_X without further processing can be used in an atmosphere containing halogen ions in the absence of abrasive particles in the atmosphere.

3.2 Protective coatings based on chromium nitride

There is often a need to use products made of D16 alloy in an atmosphere containing abrasive particles and chemically active vapors. It is known that after applying coatings (chromium nitride) to metal products, the surface hardness of the products increases significantly [21]. It was assumed that coating the samples with a layer of chromium nitride would increase the surface hardness of products made of D16 alloy, which would expand the technological application of this alloy. For example, increasing the surface hardness of products will allow them to be used in sandy desert conditions, where the atmosphere contains abrasive impurities. To test this hypothesis, a 2 μm thick chromium nitride coating was applied to the D16 sample using cathodic sputtering.

Studies of the obtained D16_(N+X) samples showed a significant increase in the surface hardness of the D16_(N+X) samples to HV100g = 655. Next, a study of the corrosion resistance of the D16_(N+X) samples was conducted. The D16_(N+X) samples were placed in a test solution for a control period. The experiment showed that the mass loss of the D16_(N+X) sample was 4.12% of the sample mass (Table 3). The surface of the D16_(N+X) sample became reddish. This fact indicates the destruction of the chromium nitride coating and the destruction of the upper layers of the surface of the D16 alloy sample (the red color of the surface indicates the release of copper from the D16 alloy structure). In addition to significant mass loss, significant corrosion damage was found on the surface of the test sample (Figure 7). Micrographs at 160x magnification showed that the corrosion process created wide areas of continuous damage between slightly damaged areas (Figure 8, a). The total area of corrosion damage was 53%.

Fig.7. Samples D16_(N+X) and D16_(N+X)_U12 after exposure to the test solution for the control time: on the left is sample D16_(N+X); on the right is sample D16_(N+X)_U12.



a) b)

Fig.8. Micrographs of the surface of samples D16_(N+X) and D16_(N+X)_U12 after exposure to the test solution for the control time: a) D16_(N+X); b) D16_(N+X)_U12. Magnification 160x.

Thus, severe damage to the micro- and macrostructure of the surface of samples D16_(N+X) was observed when these samples were placed in the test solution. For samples D16_(N+X), a significant increase in hardness leads to a significant decrease in corrosion resistance (Table 4). Products coated with chromium

nitride D16_(N+X) can be used in atmospheres containing abrasive particles but no halogen ions. For example, chromium nitride coating cannot be used in products used in seaports.

In [20], it was shown that an increase in the number of chromium-oxygen-chromium chemical bonds leads to a significant increase in the corrosion resistance of X17 stainless steel. Considering that the chromium-nitrogen coating applied to the surface of D16 alloy samples contains chromium in a basic valence state of 3, it was assumed that converting chromium to a higher valence state (from 4 to 6) would create additional chemical bonds in the coating. As a result of the increase in the density of chromium chemical bonds in the coating (chromium nitride), the corrosion resistance of the D16_(N+X) coating will increase. To activate the additional valence of chromium, active oxygen molecules were used, generated by the effect of ultraviolet radiation on oxygen molecules in the air (ultraviolet irradiation time 12 hours). The D16_(N+X) samples were subjected to 12 hours of UV radiation treatment at a temperature of 185°C. As a result, a significant increase in the hardness of the D16_(N+X) samples to HV = 1114 was recorded (a very significant increase in hardness). As a result of additional oxidation, the surface hardness of the D16_(N+X)_U12 sample increased 1.7 times at low loads.

However, a significant increase in surface brittleness was also observed. Under loads exceeding 50 grams, cracks formed around the test area. This fact prevents the use of this coating in cases of high loads, but does not prevent its use in atmospheres containing abrasive particles. Corrosion resistance testing showed a significant reduction in the corrosion process of samples coated with chromium nitride and then subjected to prolonged exposure to active forms of oxygen (D16_(N+X)_U12). A 2.56% loss in sample mass was recorded as a result of exposure to the test solution for the control time (Table 3). In other words, additional oxidation of the chromium nitride surface increased corrosion resistance by a factor of 1.61. Visually, there are no corrosion pits on the surface of the treated sample D16_(N+X)_U12 (Figure 7). The colour of the surface has not changed, and no reddish tint has appeared. With identical sample sizes, mass loss decreased by 37.8% (1.61 times) for D16_(N+X)_U12 samples. Figure 6, b shows a micrograph of the D16_(N+X)_U12 samples after the control time in the test solution. The micrograph shows that outside the corrosion pits, the surface microstructure has not changed. The total area of corrosion damage is no more than 6%.

3.3 Protective layered coatings based on chromium nitride and chromium layer

Applying a chromium nitride coating to the surface of alloy D16 revealed a significant increase in hardness to HV=655, but at the same time, corrosion resistance decreased significantly (Table 3). It was assumed that additional spraying of a chromium layer over the chromium nitride layer would increase the corrosion resistance of the product surface and, at the same time, the high hardness of the chromium nitride layer would increase the hardness of the applied chromium layer.

To test this hypothesis, a chromium nitride coating plus an additional 0.5 μm thick chromium layer was applied to sample D16 by cathodic spraying. Studies of the obtained samples D16_(N+X)_X showed a surface hardness of $HV_{100\ g}$=236. This hardness is significantly lower than the surface hardness of the D16_(N+X) samples, which is HV = 655, but 1.6 times higher than the surface hardness of the D16 alloy, which is HV = 150, and 3 times higher than the surface hardness of the D16_X samples, which is HV = 80 (Table 3).

The presence of a high-hardness surface under the chromium layer in sample D16_(N+X)_X HV = 655 resulted in the hardness of the chromium coating becoming significantly greater than HV = 240, which is the hardness of the chromium coating layer applied directly to the surface of alloy D16 (D16_X) HV=80.

At the same time, the chromium film applied to the surface with high hardness - chromium nitride HV=655 - protects the hard layer of chromium nitride HV=655 from the effects of halogen ions contained in seawater. Next, the corrosion resistance of the D16_(N+X)_X samples was studied. The experiment showed that during the control period, the mass loss of the D16_(N+X)_X sample was 2.49% of the sample mass (Table 3). The loss of atoms from the surface of sample D16_(N+X)_X decreased by 1.76 times compared to the surface of alloy D16 (Table 3). Figure 9 shows the appearance of sample D16_(N+X)_X after exposure to the test solution for the control period. Severe pitting is observed for this sample. However, there is no reddish tint on the surface, which indicates that the top layer of chromium and the layer of chromium nitride were not completely destroyed and corrosion on the surface of the D16 alloy did not begin.

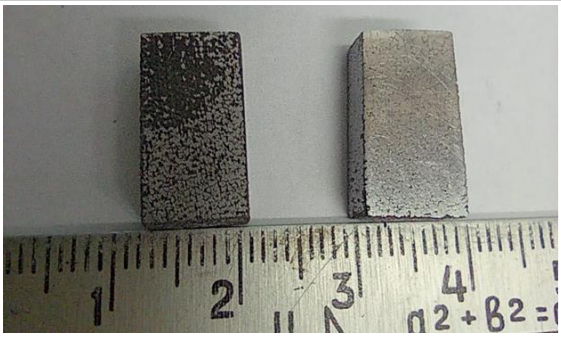
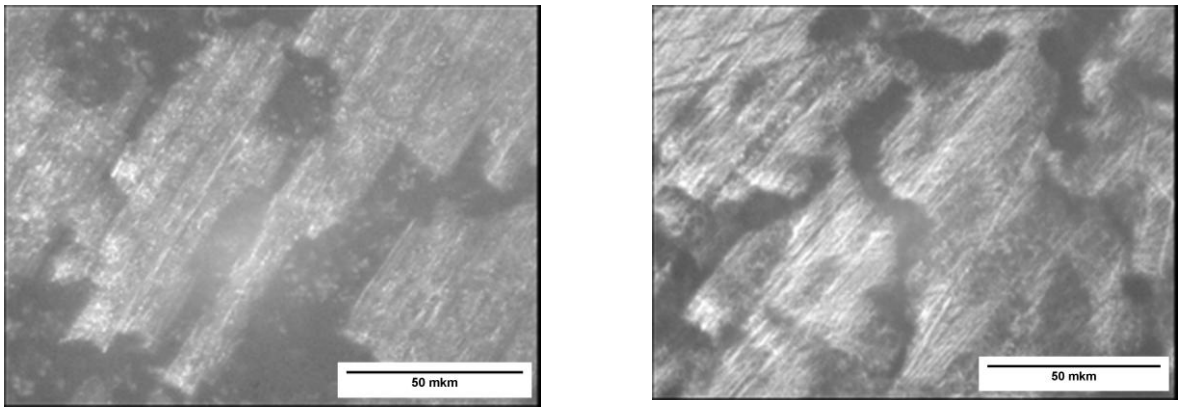


Fig.9. Samples of alloys D16_(N+X)_X and D16_(N+X)_X_U12 after exposure to the test solution for the control time. On the left is sample D16_(N+X)_X. On the right is sample D16_(N+X)_X_U12.

Micrographs at 160x magnification (Fig. 10) showed that the corrosion process created narrow zones of continuous damage between undamaged areas. The total area of the surface damaged by corrosion is 23% of the total surface area (Figure 10, a), which is 2.3 times less than the area of corrosion damage on the surface covered with chromium nitride and not subjected to ozonation. At a magnification of 160, a pattern of surface corrosion damage similar to that of chromium-nitrogen layer corrosion is observed, but the width of the damaged areas is significantly smaller. Thus, a decrease in damage to the micro- and macrostructure of the surface of D16 alloy samples coated sequentially with a layer of chromium nitride and a layer of chromium was observed when these samples were placed in the test solution. That is, additional spraying of a 0.5-micron-thick chromium layer onto the D16_(N+X) sample increases the corrosion resistance of D16 samples with a pre-applied chromium-nitrogen coating by 1.65 times (by 40%). At the same time, the final surface hardness of the D16_(N+X)_X samples is 1.57 times greater than HV=236 (36% greater) than the surface hardness of the D16 alloy samples HV=150. Also, the surface hardness of D16_(N+X)_X samples is 3 times greater (by 200%) than the surface hardness of D16_X.



a) b)

Fig.10. Micrographs of the surface of samples D16_(N+X) _X and D16_(N+X)_X_U12 after exposure to the test solution for the control time. a) CD16_(N+X)_X; b) D16_(N+X)_X_U12. Magnification 160.

Based on the results of our experiments, we can conclude that in order to create an anti-corrosion coating of chromium with high surface hardness, it is necessary to place a layer of a substance with high hardness between the surface of the D16 alloy product and the top layer of chromium. The surface structure is as follows:

1) the bottom layer of D16 alloy with a hardness of HV=150;

2) the second layer of a substance with high hardness, in our case a layer of chromium nitride with a hardness of HV=655;

3) the third layer of chromium applied over the chromium nitride layer to protect the chromium nitride layer from corrosion. The final surface hardness is HV=240.

Products made of D16 alloy, coated sequentially with layers of chromium nitride and chromium (D16_(N+X)_X), can be used in atmospheres containing abrasive particles and atmospheres containing halogen ions, for example in seaports.

Considering that the AOS surface treatment of D16_X samples resulted in a significant increase in corrosion resistance (Table 3), the surface of D16_(N+X)_X samples was also treated with AOS for 12 hours. As a result of surface oxidation, an increase in the hardness of the D16_(N+X)_X coating to HV= 350 was recorded, and the hardness under low loads increased by 1.47 times.

However, a significant increase in surface brittleness was also observed: under loads exceeding 50 grams, cracks formed around the indentation zone. This fact prevents the use of this coating in cases of high loads, but does not prevent its use in atmospheres containing abrasive particles, such as sand particles in the desert.

Corrosion resistance testing showed a significant reduction in the corrosion process of D16 alloy samples coated with chromium nitride plus pure chromium, subjected to prolonged exposure to active forms of oxygen (12 hours): samples D16_(N+X)_X_U12. A mass loss of 1.86% was recorded for the sample as a result of exposure to the test solution during the control test. Before oxidation, there was a 2.49% loss in the surface mass of the D16_(N+X)_X samples (Table 3). That is, additional oxidation of the surface of the D16_(N+X)_X samples increased the corrosion resistance of the surface by 1.34 times (by 25%).

Visually, after the control time in the test solution, a small amount of fine pitting is present on the surface of sample D16_(N+X) _X_U12 (Figure 9, b). The colour of the sample surface has not changed, and no reddish tint has appeared. Outside the corrosion pits, the microstructure of the surface of sample D16_(N+X)_X_U12 did not change (Figure 10, b). The total area of corrosion damage on sample D16_(N+X)_X_U12 is no more than 12% of the total study area, which is 1.92 times less than the area of corrosion damage on the surface of D16_(N+X)_X. Compared to the surface of sample D16_(N+X)_X, the total area of affected areas on the oxidized surface of sample D16_(N+X)_X_U12 decreased by 48%.

In summary, the following conclusions can be drawn:

1. As a result of treating the surface of D16_(N+X)_X samples with air exposed to UV radiation, the surface hardness increased from HV=236 to HV=350 (1.48 times).

2. The corrosion resistance of the surface of samples D16_(N+X)_X as a result of AOS treatment increased by 1.34 times. The total area of corrosion damage as a result of surface treatment decreased by 1.7 times.

4. Conclusions

In summary, the following conclusions can be drawn:

1. Films of chromium and chromium nitride deposited by cathodic sputtering onto the surface of a D16 alloy specimen form a smooth, uniform surface with good adhesion to the D16 alloy surface. These films are free from cracks.

2. Spraying a layer of chromium onto the surface of alloy D16 significantly increases the corrosion resistance of the surface (by 39%), but at the same time significantly reduces the hardness of the surface (by 46%). The D16_X coating cannot be used in conditions where there are abrasive particles in the space surrounding the product. This D16_X, D16_X_U12 coating is acceptable for use in sea fog conditions.

3. Spraying a layer of chromium nitride onto the surface of the D16 alloy greatly increases the surface hardness – 4.3 times that of the D16 alloy surface.

At the same time, the corrosion resistance of the surface is significantly reduced (by 35%). Products with a D16_(N+X) chromium nitride coating cannot be used in sea fog conditions, but can be used in sandy desert conditions.

4. Treating the sputtered chromium nitride layer with air exposed to ultraviolet radiation significantly increases hardness by 41% and corrosion resistance by 62% compared to the untreated chromium nitride layer. These D16_(N+X)_U12 products are suitable for use in sea fog conditions and in the presence of abrasive particles in the atmosphere.

5. It was found that in order to increase the hardness of the chrome-coated surface, it is necessary to place a layer of a substance with very high hardness, for example, a layer of chromium nitride with a hardness of HV=655, between the surface of the D16 alloy and the chrome layer. This will increase the hardness of the surface layer of chrome by 3 times, from HV=80 to HV=236.

6. A layer of chrome sprayed over a layer of chromium nitride reduces the corrosion rate of the product D16_(N+X) by 38%.

7. Treating the chrome layer applied to the surface of the chromium nitride with air exposed to UV radiation increases the hardness of the top chrome layer by 32% to HV=350. Oxidation of the chrome surface also increases the corrosion resistance of the chrome layer by 25%.

8. In marine climates and in the presence of strong winds, it is recommended to use D16_(N+X) _X_U12 products made of D16 alloy, coated with a layer of chromium nitride, on top of which there is a layer of additionally oxidized chromium.

Conflict of interest statement

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

CRediT author statement

Guchenko S.A.: investigation, writing - original draft; **Seldyugaev O.B.:** conceptualization, writing - review & editing; **Karstina S.G.:** investigation, writing - review & editing; **Danilov M.B.:** investigation, formal analysis; **Afanasyev D.A.:** visualization, writing - review & editing. The final manuscript was read and approved by all authors.

Statement on the use of Artificial Intelligence.

The authors declare that no artificial intelligence tools were used to generate scientific content, results, or conclusions of this article.

Data Availability Statement

The data that support the findings of this article are openly available.

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