Представлені результати по взаємодії плівки оксиду алюмінію, нанесеної на технічно чистий титан марки ВТ1-0 методом магнетронного реактивного напилення, з атмосферою, яка містить водень. Необхідність проведення таких досліджень полягає в пошуку захисних покриттів, що перешкоджають проникненню водню у виріб. Дана система витримувалась у водневій атмосфері в інтервалі від 1–4 годин, при тиску $2 \cdot 10^5$ Па (2 атм) і температурі T=400 °C. Отримано дані щодо розподілу водню по товщині плівки і його вмісту в тонкоплівковій системі. Показано, що водень проникає в плівку і накопичується в ній аж до третьої години, і лише після починає проникати в підложку. Вдалося збільшити час витримки у воденьміскому середовищі і збільшити температуру нагрівання до стадії руйнування плівки. У разі вихідної плівки і після витримки протягом від 1 до 3 годин сила зчеплення плівки з підложкою зростає, очевидно, за рахунок утворення водневих зв'язків плівка-підложка. Адсорбція атомів водню на поверхні плівки Al_2O_3 супроводжується збільшенням її провідності не більше ніж на 4 % і зі збільшенням часу витримки. Така зміна провідності плівки Al_2O_3 може бути пояснена на підставі утворення зонної структури. Тонкі оксидні плівки можуть мати суцільну однобічну провідність, в разі ж, якщо плівка товста (від 0,5 мкм і вище), то говорити про однорідну провідність не можна. Отримані дані щодо впливу часу витримки у водневій атмосфері вказують на збільшення адгезійної міцності майже до 6 разів протягом 3-х годин і 2,5 раз після 4 годин. Певний коефіцієнт тертя плівки зростає не більше ніж в 2,5 рази. Вимірюючи електропровідність поверхні плівки, було виявлено, що вона зростає в міру збільшення часу витримки у водневій атмосфері. Така закономірність очевидно пов'язана зі створенням переходів p-n-типу в плівці оксиду алюмінію за рахунок іонів водню

Ключові слова: титан марки ВТ 1-0, метод магнетронного розпилення, оксид алюмінію, воднева атмосфера, адгезія, трибологія, електропровідність поверхні плівок

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INFLUENCE OF THE DURATION OF AGING THE SYSTEM Ti/Al_2O_3 IN A HYDROGEN ATMOSPHERE ON HYDROGEN SORPTION, ADHESION, TRIBOLOGY, AND ELECTRICAL CONDUCTIVITY OF THE FILM

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1. Introduction

Hydrogen energy possesses the potential to implement a sustainable society as it can produce energy from various sources and in so doing does not emit carbon dioxide. At present, however, it is necessary to solve many technical problems associated with the use of hydrogen, namely, the improvement of technologies of fuel cells, hydrogen production and storage, as well as the development of materials and systems, capable to withstand cyclic loads in a hydrogen

environment [1, 2]. When hydrogen is stored in metal collectors there occurs a metal's structure degradation due to the emergence of stretching and cyclic stress concentrators, which leads to hydrogen embrittlement [3–5]. While the atomistic processes of hydrogen embrittlement have not yet been fully understood, common sense implies that hydrogen penetrates the microstructure of alloys as a result of physical, chemical adsorption, and diffusion. The catalytic and thermal effects during operation of an article provide enough energy for adsorption, dissociation, and absorption at the newly formed (not oxidized) metal surfaces. Because hydrogen embrittlement is the process that starts at the surface of a material, it is advisable to employ methods of surface treatment in order to create protective barrier layers at the surface.

2. Literature review and problem statement

New technologies in the field of metallurgy cannot ensure complete protection against the penetration of hydrogen into the depth of a material. Therefore, in order to protect structural and functional materials against hydrogen embrittlement, it is a relevant task to design technologies for forming the thin-film coatings. Thus, paper [1] considers the application of composite films as more erosion resistant for protecting structural materials made from tungsten, applied in nuclear reactors. The thin-film system must have high corrosion resistance and heat-resistance. Such properties were discovered in experiments [2] for aluminum oxide films. They were deposited by plasma spraying onto substrates made from various grades of stainless steel and titanium. The highest barrier properties were demonstrated by films of aluminum oxide on a titanium substrate. Film thickness was about 1 μm, while the coefficient of permeability was reduced to 10^4 mol-$H_2m^{-1}s^{-1}Pa^{-0,5}$. Authors of work [3] explored the hydride phases, the orientation and impact of a hydride orientation on mechanical properties and the absorption of hydrogen by zirconium alloys. Paper [4] compares the physical-mechanical characteristics of the two thin-film systems Al_2O_3 and NiAlSi deposited on a steel substrate. A coating with the aluminum oxide film has no cracks, but it has high porosity, which is why it is recommended for use in the military and mining industries. A study into corrosion resistance and tendency to embrittlement in screws made from alloys of the system Ti-10Mo-8V-1Fe-3.5Al was reported in [5]; the results showed that the main cause of destruction of screws made from titanium alloys was the internal defects rather than hydrogen embrittlement. There has recently been an increase in failures at power plants related to the destruction of welding joints T24 at temperatures ranging from 200 °C. Authors of [6] conducted a series of experiments that linked this phenomenon to the penetration of hydrogen, which leads to cracking of welding joints. And it was established in [7], when determining the mechanical properties, that the hydrogen dissolved in a β-titanium alloy reduces yield limit of samples and reduces stress to destruction.

Butt and arc welded joints of gas pipes made from alloys of the system 7CrMoVTiB10-10, prone to cracking, were improved in terms of their operational characteristics following the specialized heat treatment after welding [8]. Most research on the Al_2O_3 films were conducted in a hydrogen environment at pressures <100 kPa and it was found that these layers are an effective barrier at the surface of articles against hydrogen penetration [4, 5, 7]. Fragmented information that is found in the scientific literature about the properties of dissolved hydrogen in metals is explained by that its behavior depends on certain factors, namely the nature of a metal, the degree of its purity, concentration of the alloying element, stresses, and others. In the indicated papers, authors examine the temperature-concentration dependences of diffusion coefficients, the causes of solubility and penetration of hydrogen into metals. Cracking starts at temperatures above 200 °C. It is natural that in order to work out effective measures to remove hydrogen from metallic products (or prevent them from gas saturation) and, therefore, to improve their operational properties, it is necessary to receive reliable qualitative and quantitative information on the temperature and concentration dependences of diffusion coefficients.

Given the above, it is necessary to undertake a study into the interaction between hydrogen and the Al_2O_3 film, applied onto technically pure titanium of grade V-T 1-0 using a magnetron sputtering method, where the film acts as a "barrier" preventing the penetration of hydrogen into a titanium alloy.

3. The aim and objectives of the study

The aim of this study is to examine the effect of hydrogen on the thin-film system "aluminum oxide – titanium", namely, to investigate the distribution of hydrogen, as well as the film's adhesive and sorption properties, and a change in friction coefficient.

To accomplish the aim, the following tasks have been set:

– to prepare the examined samples (surface treatment of titanium VT1-0 and application of the Al_2O_3 thin-film coatings by the method of magnetron sputtering, saturation in a hydrogen atmosphere);

– to determine the distribution of chemical elements in the system Al_2O_3/Ti;

– to investigate the effect of hydrogen on the mechanical and electrical properties of the Al_2O_3 thin film.

4. Materials and methods of research

The examined objects were the samples of titanium of grade VT1-0 with the Al_2O_3 (Al_2O_3/Ti). Titanium samples the size of 20×20×1 mm^3 were fabricated by a spark cutting method. The surface of the samples was mechanically ground and polished prior to saturation with hydrogen.

Application of the film was performed using a magnetron reactive sputtering method. The coating application technology implied the following: samples made from titanium were put onto a movable table, and the chamber was pressurized to a vacuum of $P=4\cdot10^{-3}$ Pa followed by the subsequent ion cleaning (Ar), at the following parameters of the beam: U=2.5 kV, I=0.2 A. When the target was cleaned from the non-desired oxide film, oxygen was added to the chamber with the magnetron system entering the operation mode: $P=2\cdot10^{-1}$ Pa, U=0.8 kV, I=10 mA. Upon stabilization of discharge parameters, we used the magnetron sputtering of the aluminum oxide film. Coating thickness is ~400 nm.

We aged the titanium VT1-0 and the system Al_2O_3/Ti in a hydrogen atmosphere at the installation "Gas Reaction Controller" at the following parameters: hydrogen pressure

in the chamber P=2·10^5 Pa (2 bar), temperature T=400 °C, over 1, 2, 3 and 4 hours. The hydrogen content in the examined samples was determined as the duration of aging in a hydrogen atmosphere, based on a change in the pressure in the working chamber, as well as at the hydrogen analyzer RHEN-602, and using a thermally stimulated gas release method (TGSR).

Distribution of chemical elements in the system Al_2O_3/Ti was examined at the glow discharge spectrometer GD-PROFILER 2; adhesion strength of coatings was determined at the Micro-Scratch Tester MST-S-AX-0000. The following adhesion test modes were employed: load F=20 N, load rate v=19.99 N/min, length of the scratch L=10 mm.

Friction coefficient of the system Al_2O_3/Ti was determined at a high temperature tribometer. The following test modes were employed: radius of the ball d= 2.98 mm, linear velocity is 3 cm/s, normal load N=1 N, temperature T=23 °C (room).

We measured surface conductivity using a method of "overhead electrodes" (made from copper). The voltage magnitude varied in the range of 0.01–0.09 V, the lower limit is selected based on the generator capacity, the upper limit – to avoid the breakdown of the film due to the high electric field strength. Based on the acquired voltampere characteristics (VAC), we determined surface conductivity from formula

$$\sigma = \frac{I}{U} \cdot \frac{L}{d}, \quad (1)$$

where σ is the conductivity of the film surface (siemens), U is the value for the applied voltage, I is the measured current strength, d is the distance between the electrodes (d=2 mm), L is the size of the electrode (L=7 mm).

4. 1. Determining the content of hydrogen

Table 1 gives values for the mass content of hydrogen in the system Al_2O_3/Ti depending on aging duration in a hydrogen atmosphere. With an increase in the aging duration in a hydrogen atmosphere from 1 to 3 hours the hydrogen content in samples increases insignificantly (except for data acquired at the "Gas Reaction Controller"), and after 4 hours there is a dramatic increase in the hydrogen content. Such a difference may be due to the fact that with an increase in the aging duration in a hydrogen atmosphere the film is modified under the action of hydrogen and the hydrogen penetrates through the film. Work [9] demonstrated that following the saturation in a hydrogen environment the aluminum oxide film undergoes cracking, however, its protective properties in terms of hydrogen penetration are retained.

Fig. 1 shows dependences of intensity of the H_2 release from the system Al_2O_3/Ti, aged in a hydrogen atmosphere over 1, 2, 3 and 4 hours. The heating rate was 1 deg./s. Based on the spectra of the thermally stimulated hydrogen release, we calculated data on the cumulative gas release (Table 1).

Dependence in Fig. 1 shows that an increase in the aging duration in a hydrogen atmosphere leads to an increase in the concentration of hydrogen in the system Al_2O_3/Ti.

It is also worth noting that at an increase in aging duration we observed the offset of the temperature peak towards the region of lower temperatures, Table 2. This shift is predetermined by the different values of activation energy of hydrogen desorption. The presence of two temperature peaks after 4 hours of aging in a hydrogen atmosphere is associated with the formation of titanium hydrides and their subsequent decomposition during thermal heating.

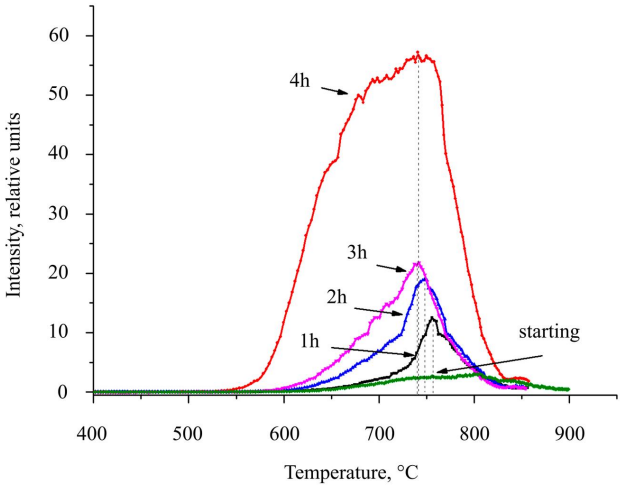

Fig. 1. Thermal-desorption spectra of hydrogen release depending on aging duration in a hydrogen atmosphere (1–4 hours)

Table 1

Mass content of hydrogen in the system Al_2O_3/Ti

Aging duration in a hydrogen atmosphere, h	starting	1	2	3	4
Hydrogen content, % by weight		0.032	0.113	0.610	0.750
Mass content of hydrogen based on the hydrogen analyzer RHEN-602, ppm	85.5	140.3	212.8	344.3	1,030.8
Integrated hydrogen release based on data from TGSR, relative units	468	821	1,524	2,007	8,807

Table 2

Desorption activation energy

Aging duration in a hydrogen atmosphere, h	starting	1	2	3	4	
Temperature peak, °C	805	757	748	743	695	743
Desorption activation energy, eV	4.58	4.38	4.34	4.31	4.12	4.32

4. 2. Distribution of chemical elements for depth

Fig. 2 shows results of the distribution of chemical elements of the film and substrate for depth, acquired at the glow discharge spectrometer GD-PROFILER 2. One can see (Fig. 2, *a*) that the starting film originally contained hydrogen, which does not contradict the data given in Table 1. The presence of hydrogen in the film is due to its penetration during the process of application and formation of the film. Comparing the content of H_2 in Fig. 2, *a*–*e*, one can see that an increase in aging duration (from 1 to 4 hours) leads to an increase in the hydrogen concentration in a hydrogen atmosphere. Low oxygen content in the film is due to the low sensitivity of the device for oxygen and nitrogen.

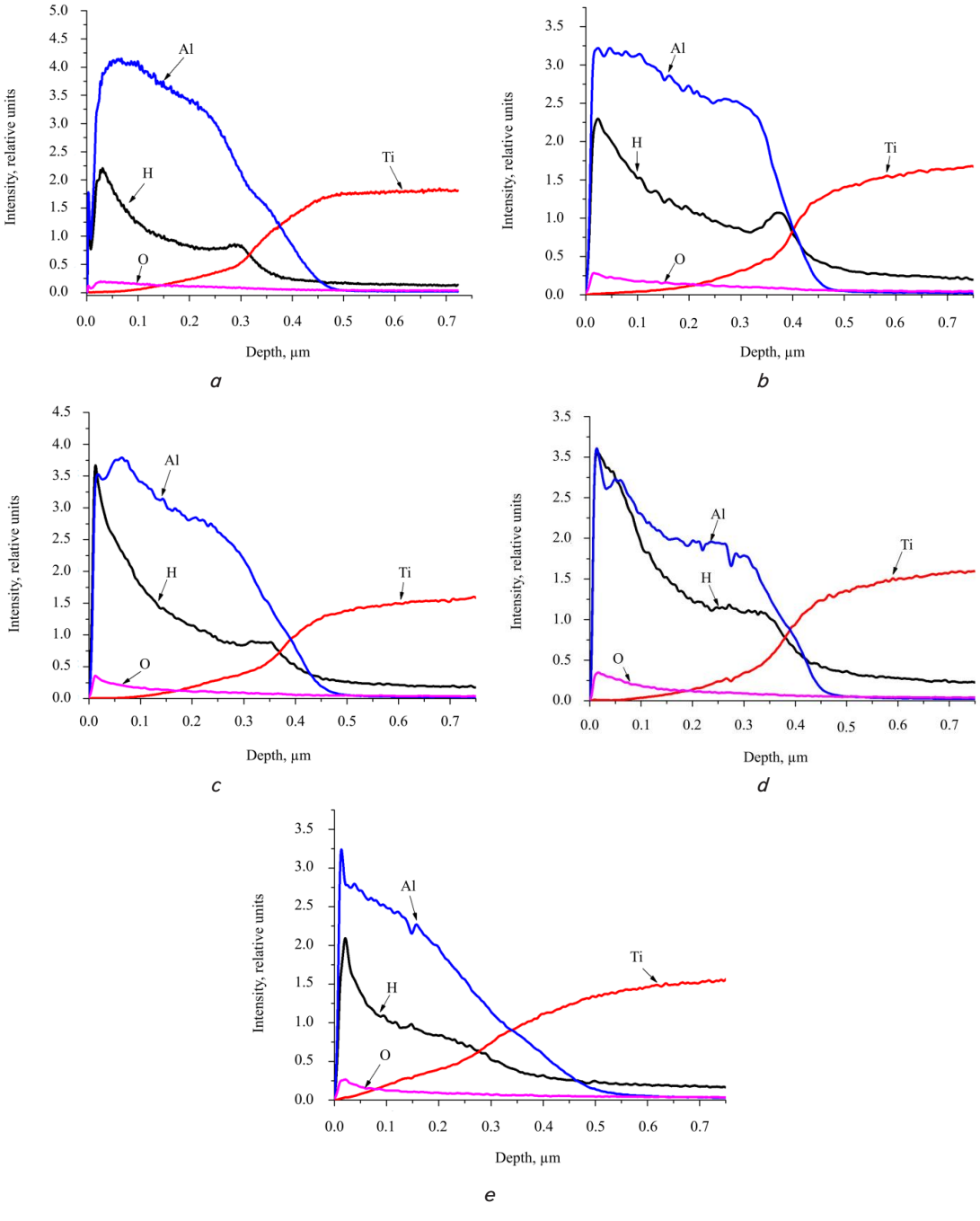

Fig. 2. Distribution of chemical elements in the system Al_2O_3/Ti: *a* — starting film; *b* — aged for 1 h in a hydrogen atmosphere; *c* — aged for 2 h in a hydrogen atmosphere; *d* — aged for 3 h in a hydrogen atmosphere; *e* — aged for 4 h in a hydrogen atmosphere

4. 3. Tribology and adhesion

Fig. 3 shows results of tribological testing of the Al_2O_3/Ti coatings on a titanium substrate. A given dependence demonstrates that the lowest friction coefficient (~0.1) characterizes the samples after 1 hour of aging in a hydrogen atmosphere; an increase in aging duration leads to its growth for up to 3 hours. After 3 and 4 hours of aging in a hydrogen environment, we observe equal friction coefficients (~0.16), indicating the accumulation of hydrogen in the film.

A change in the properties of the film relates to the diffusion of atomic hydrogen through it. Free hydrogen atoms are adsorbed at the film's surface and diffuse it into the near-surface layers; this leads to the modification of the coating. At this stage, there is an increase in the dispersion of the microstructure of the coating, with its surface lacking any visible damage. When increasing the aging duration, hydrogen through modified layers easily penetrates sites at the surface of the substrate, with an increase in its diffusion rate. This is the stage of the film destruction – the instantaneous destruction of the surface layer of the film. Hydrogen penetrates the microvoids inside the film, where it is mobilized. The voids "close" and this leads to an increase in the pressure of molecular hydrogen on the walls of microcracks; there occurs the instantaneous destruction of the film along all previously emerged microcracks with an increase in friction coefficient.

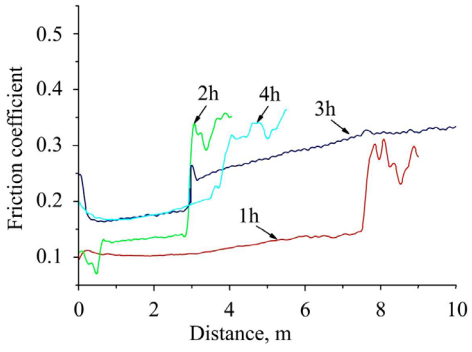

Fig. 3. Dependence of friction coefficient of the aluminum oxide film on titanium on aging duration in a hydrogen atmosphere

Table 3 shows adhesion strength of the aluminum oxide film on the titanium VT1-0. It follows from these data that after 4 hours of aging in a hydrogen atmosphere the film starts to flake off (a decrease in adhesion strength); this may be due to the accumulation of hydrogen at the film-substrate interphase boundary and, possibly, to the formation of hydrides in titanium [9]. In the case of the starting film, and after aging duration of 1 to 3 hours, adhesion strength between the film and substrate increases, apparently due to the formation of hydrogen bonds film-substrate.

Table 3

Adhesion strength of the aluminum oxide film on titanium

Aging duration in a hydrogen atmosphere, h	0	1	2	3	4
Adhesion strength, N	1.2±0.3	4.9±0.1	5.5±0.4	7.8±0.7	3.1±0.7

4. Electrical conductivity of films' surface

Table 4 gives data on voltage and current, based on which we calculated electrical conductivity of the surface of the Al_2O_3/Ti film on the titanium VT1-0 from formula (1) after different aging duration in a hydrogen atmosphere. The dependences derived are shown in Fig. 4. One can see that, depending on the time of aging in a hydrogen atmosphere, electrical conductivity of the film's surface films increases. It should be noted that a given method produces a qualitative dependence because the estimation does not take into account the geometrical dimensions of plates and the edge effects that occur on the plates-electrodes.

Table 4

Current and voltage values on the Al_2O_3 film

Voltage, V	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
Current (starting), A·10^{-8}	1.3	2.23	3.14	4.05	4.9	5.85	6.74	7.66	8.56
Current (1 hour), A·10^{-8}	1.37	2.28	3.18	4.09	4.99	5.89	6.77	7.62	8.59
Current (2 hours), A·10^{-8}	1.47	2.37	3.26	4.16	5.06	5.96	6.85	7.46	8.66
Current (3 hours), A·10^{-8}	1.53	2.43	3.32	4.23	5.12	6.02	6.9	7.8	8.7
Current (4 hours), A·10^{-8}	1.6	2.5	3.4	4.3	5.2	6.1	7	7.9	8.8

Fig. 4 shows that the adsorption of hydrogen atoms at the surface of the Al_2O_3 film is accompanied by an increase in its conductivity not exceeding 4 % and an increase in aging duration. Such a change in the conductivity of the Al_2O_3 film's surface can be explained based on the formation of a zone structure. Thin oxide films may possess continuous one-side conductivity; in the case when the film is thick (above 0.5 μm), then it does not make sense to argue about the homogeneous conductivity.

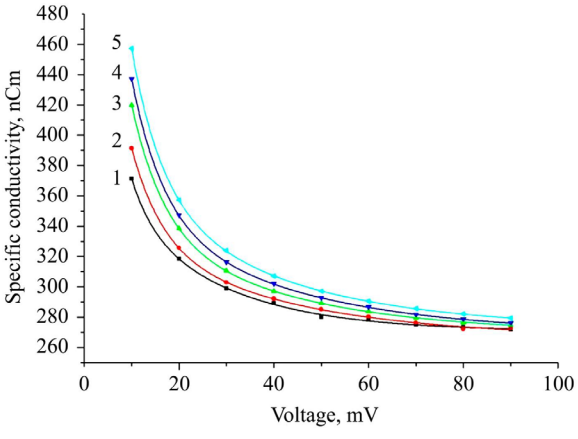

Fig. 4. Dependence graph of electrical conductivity of the surface of the Al_2O_3 film on titanium after different duration of saturation with hydrogen: 1 – starting, 2 – 1 h, 3 – 2 h, 4 – 3 h, 5 – 4 h

$A1_2O_{3+m}$ is a *p*-type hole semiconductor, and, at a missing electron $A1_{2+n}O_3$, – of the *n*-type. Thus, the formation process of the aluminum oxide film leads to the creation of the *r*-n-type junction with an intermediate layer of a dielectric. It is known that when exposed to a long time of passing electric current through the oxide film, the ions are redistributed, resulting in a partial destruction of the *p*-*n*-junction. The $A1_2O_{3+m}$ film turns into an electron semiconductor that contains the excess or shortage of aluminum ions, which also depends on conditions. A similar destruction of the *p*-*n* junction is observed when heating an oxide film, in this case, there is an inverse relationship, that is the concentration of oxygen ions decreases, resulting in the excessive amounts of aluminum ions. In the case of hydrogen interaction with an oxide film, binding the oxygen, it leads to the transformation of aluminum oxide into an electron semiconductor [11].

As is often the case when applying the films, there form the defective places, which possess high conductivity. The cause of defects could be a heterogeneous surface of the oxide (pore), which also affects the conductivity in films.

5. Discussion of results of studying the Al_2O_3 film

It follows from the results obtained after aging the films in a hydrogen atmosphere that hydrogen diffuses into the film and builds up inside it. Such an accumulation due to diffusion lasts for 3 hours at a temperature of 400 °C, which shows better results, since, prior to it, the destruction occurred at 200 °C [5, 6] and a pressure of $2 \cdot 10^5$ Pa. The further

increase in aging duration leads to that the film starts to break down [11], while hydrogen begins to accumulate in the substrate. Earlier work [11] reported a study into the aluminum oxide film, but on the alloy VT-6 substrate. In this paper, we used the alloy VT-1 substrate, as the most widely used construction material for nuclear reactors. In this case, we increased aging duration in a hydrogen-containing environment and obtained satisfactory results. Give this, such films could be used as hydrogen accumulators and as protective coatings for walls of nuclear reactors in hydrogen-containing media at pressures below atmospheric. To effectively saturate metals with hydrogen in a HF hydrogen plasma, the optimum absorbed power of an HF discharge (HFD) is 200 W at a sample's temperature of 500 °C. Efficiency of saturating the samples of titanium with hydrogen in a high-frequency hydrogen plasma, created in a quartz reactor, grows with an increase in the power, absorbed by the plasma of high-frequency radiation, up to the values of 200 values W. The higher power charges lead to the modification of the surface and oxygen enrichment with the formation of O–H bonds. In this case, oxygen is released from the walls of a quartz reactor under the influence of a high-frequency radiation.

6. Conclusions

1. An increase in aging duration in a hydrogen atmosphere from 1 to 4 hours leads to an increase in friction coefficient of the Al_2O_3 film by more than 2 times.

2. An increase in aging duration in a hydrogen atmosphere from 1 to 3 hours leads to an increase in the film's adhesion strength by more than 6 times compared to the starting one. After 4 hours of aging in a hydrogen atmosphere, the adhesion strength decreases by more than two-fold, compared to 3 hours; this is obviously due to the increase in hydrogen at the film-substrate interphase boundary.

3. An increase in aging duration in a hydrogen atmosphere leads to an increase in electrical conductivity of the film's surface not exceeding 4 %. This difference is due to that the passing of current through the oxide film redistributes ions in the film resulting in a partially destroyed *p-n*-junction.

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