ION-PLASMA HARDENING OF A TITANIUM ALLOY OT4-1

ИОННО-ПЛАЗМЕННОЕ УПРОЧНЕНИЕ ТИТАНОВОГО СПЛАВА ОТ4-1

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Abstract: *The paper is devoted to studying the influence of technological process parameters of deposition on the composition, structure and strength characteristics of TiN coatings on titanium alloy OT4-1. The TiN coatings were deposited by a vacuum-arc evaporator. In addition, the influence of the type of layers formation on microhardness and wear resistance of TiN layers and a metal substrate is presented.*

***KEYWORDS**: ARC EVAPORATOR, LAYERS, TIN, ALLOY, MICROHARDNESS, WEAR RESISTANCE*

1. Introduction

A vacuum and plasma technology of hard coating deposition on different details of machines and parts of constructions allows to strengthen their surface. Protective and decorative titanium nitride layers are applied on various materials and alloys. The most widely used is wear-resistant coating of metal-cutting tools [1, 2]. Decorative coatings with high anticorrosive and wear-resistant properties are formed on the surface of various steels, such as 12X18H10T for example [3]. The properties of coatings are determined by technological process parameters since the operating properties of deposited layers depend on the degree of crystal structure's perfection. The paper is devoted to studying the influence of these parameters on the composition, structure and strength characteristics of TiN coatings made of OT4-1 titanium alloys.

2. The experimental part

Initial materials.

The sheets of titanium alloy OT4-1 were used as a material on which wear-resistant titanium nitride coatings were formed. OT4-1 titanium alloy (Ti—Al—Mn system) with a tensile strength of $s_в$ < 700 MPs relates to alloys of low strength and high plasticity. It is a pseudo α - alloy with a small amount of β -phases ($K\beta$ < 0,25) due to insignificant alloying of α- (Al) and β- (Mn) - stabilizers. It is characterized by high ductility both hot and cold that allows to receive all types of semi-finished products: foil, tape, sheets, plates, forgings, stampings, profiles, pipes and so on.

Methods of forming a coating.

In fig.1 a VU-1M vacuum installation's scheme is illustrated. To form a coating a solid (cathode) 7 is evaporated in vacuum by a low-voltage DC electric arc. By evaporation of the substance in a vacuum chamber 1 its highly ionized plasma is formed there. Between the cathode 7 and an electrode 8 a controlled potential difference appears. In the electric field charged plasma particles have been accelerated to achieve the energy proportional to the potential difference and run towards the auxiliary electrode 8, on which the processed details 2 are located, forming on a surface of dense and uniform coating thickness. The cathode material was "VT-1-0" titanium with a diameter of 60mm. Nitrogen gas of high purity had been used as reaction gas. Limiting residual pressure in the vacuum chamber was $6.6x10^{-3}$ Pa. The coating mode provided uniform layers with thickness in the range of 1,0-4,5 mcm. Studies were conducted on the samples made of titanic alloy OT4-1. Pre-ionic surface treatment of the samples was carried out in a glow nitrogen discharge.

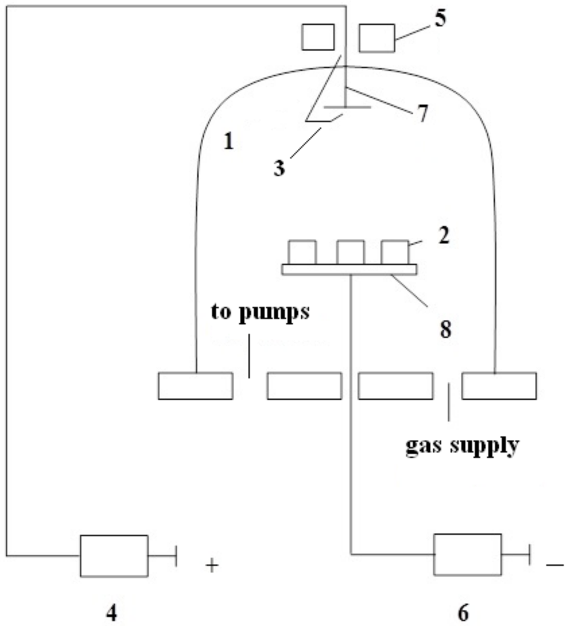

Fig.1. Schematic illustration of installation VU-1M:
1-vacuum chamber, 2- processed details, 3- igniter electrode, 4- power supply, 5- focusing coil, 6- power supply, 7- cathode, 8- auxiliary electrode

Research methods.

Microhardness of the created layers had been determined by microhardness tester PMT-3. The load on a diamond pyramid was 50 or 20 g.

Microstructure and thickness of the samples were examined also by microhardness tester PMT-3 with increase x130 and x500.

Wear resistance in abrasion was conducted with MI-2 machine according to GOST 11626-75 by repeated multiple sliding of the sample on one and the same track of the counter body plane on an abrasive cloth under the load of 68,6 N (7kgs). The sliding speed of the samples on the sandpaper's plane was 40 rpm or 0.28±0.05 m/sec. The loss of the sample's mass or the time of removing the cover during testing is a criterion of wear depreciation. The weight loss of the samples when sliding on Abrasive was evaluated by weighing the samples on analytical balance VLA-200. The weighing error was ±0,0002. Abrasive on a fabric basis with electro corundum and granularity numbers 40, 10 GOST 6456-79, GOST 5009-76 was used in the work

3. Results and discussion

The process of forming a coating may be divided into three steps. The first step is making a plasma stream of evaporated substance's particles, herewith the electrode 3 ignites the arc and evaporating of the cathode material starts (fig. 1). The peculiarity of the arc with cathode spots is that the current at the cathode is concentrated on one or a few merging, rapidly disappearing and randomly moving spots. a cathode zone the emission process is occurring which ensure the value of the electron current from the cathode which is close to the total discharge current. In addition, the thermal, auto- and thermoautoelectronic emission processes are prevailing. When the cathodes receive a large amount of energy from current, cathodes strongly heat up and evaporate, and most often there is their erosion - destruction of an electrode to ablation of weight. Therefore, arc discharges usually to burn in the vapor of the evaporated metal [4]. Feature of arc with cathode spots is seen in the fact that the current at the cathode is concentrated in one or more emerging, the fast disappearing and randomly moving spots. The current density in those cathode spots reaches 10^7 A/cm^2. The second step is ionic bombing or ionic cleaning of the surface and has a very important role in the formation. As a result of the bombardment physical and chemical cleaning of the surface in high vacuum and significant warming of the surface occur while the bulk sample remains relatively cold. In turn, this causes considerable compressive stress that heals surface micro-cracks and improves the working characteristics of the surface. Finally, the bombing causes grasping the sprayed particles by the purified and heated surface and the thinnest layer between the substrate and the coating is formed. The third step is in coating a predetermined thickness onto the surface - this is carried out with a small U bias potential between the cathode and the auxiliary electrode (not more than 200 V). During the coating process, the surface is periodically short-ionic cleaned to maintain the desired temperature.

Microhardness layers of TiN.

Due to anisotropy of mechanical properties of titanium nitride TiN in different crystallographic directions one should expect increase in operating properties of coating along one of them. If the layer is texturised along the direction [111], the strength characteristics will be maximal as the plane (111) in a face-centered cubic unit cell is the most populated with atoms. The spatial group is Fm3m, parameters of crystal cell are: a=0,422-0,424 nm. The lattice type is NaCl.

Figure 2 shows the influence of technological parameters (the potential bias) and the type of layers formation on the microhardness of TiN layers and a metal basis.

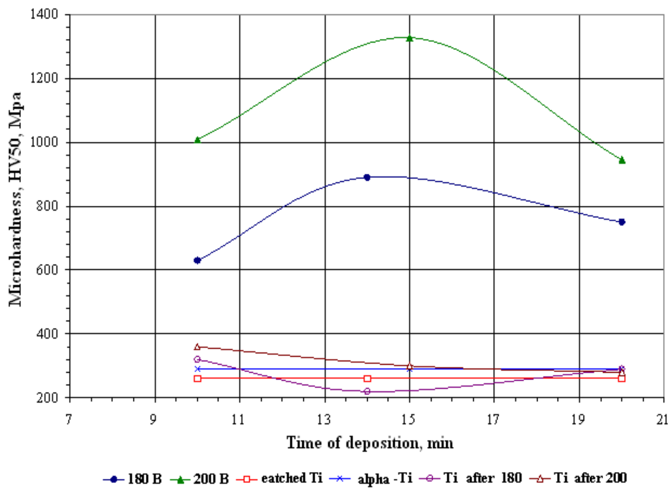

Рис.2. Influence of the potential of displacement on microhardness of TiN layers

The initial microhardness of titanic alloy OT4-1 varied from HV50=150 to HV50=290 MPa in accordance with the surface preparation before spraying. Such considerable difference in values of HV50 is explained by different composition of surface layers. The initial sample was not heat-treated or alpha case. Heat-treatment was made to increase the alloy plasticity and to protect from oxidation. The samples' etching (removing of the alpha case layer) was carried out traditionally in water solution containing 100-120 g/l of sulfuric (H2SO4), 120-130 g/l of nitric (HNO3) and 30-40 g/l of hydrofluoric (HF) acids.

The sample with the maximum microhardness of 1330 N/mm^2 was textured by a plane (111). The X-ray phase analysis of the coverings deposited at various nitrogen pressures showed that the maximum value of microhardness corresponds to the smallest width of diffraction lines. As appears from fig. 2, microhardness along with the influence of potential bias depends on spraying duration. Increasing the thickness of coating (spraying duration) causes an increase in microhardness. This fact is not new. We have observed a decrease in microhardness with increasing the duration from 15 to 20 minutes. It may be connected with the fact that a TiN film begins to pulverize having reached thickness of 5-6 microns under the influence of the plasma column, and its surface becomes more defective. As a consequence there is a decrease in microhardness.

Fig. 3 shows the dependence of coating thickness on the time of deposition. The results were obtained by processing the X-ray pattern of system "substrate-film" in view of the X-ray absorption coefficient calculated for the stoichiometric composition of titanium nitride.

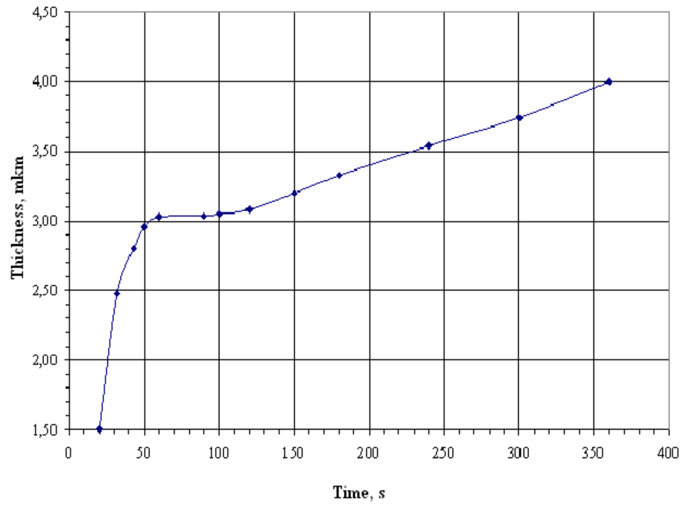

Fig.3. The thickness of film as a function of the time deposition ($p_{N2}=10^{-2}$ Pa)

The dependence course of "thickness-time of deposition" may be explained on the basis of the island model of films' growth. At the initial moment of growth the film is formed in the form of islands while the X-ray diffraction intensity of the reflection from the substrate is weakening slightly. With a further growth the islands begin to merge, and the proportion part of the coated surface is greatly increased. This causes sharp changes in the intensity ratio of diffraction peaks on the substrate material before and after coating. The growth can occur upto several layers of a film, and then the dependence of coating thickness on the application time varies slightly. One of possible reasons for this are equally probable processes of deposition and sputtering. Thus, according to X-ray investigations under these conditions the coating thickness intensively increases during the first 1.5-2 min.

Since the microhardness of a metal base depended on the type of training the original surface (fig. 2), we tried to do a comparative analysis of microhardness according to the conditions of layer formation (tab. 1).

The greatest microhardness of HV_{50}=2000 MPa was observed in the double-layer sample formed in conditions of maximum HV_{50} of TiN monolayer (current of the arc discharge 80A, time of deposition 15 min, potential of displacement 200V). On the etched surface of a titanium alloy a layer of the titan then titanium nitride were put. TiN layer upon gas-saturated surface of alloy OT4-1 had HV_{50}=1080 MPa, but was more rough.

Table 1

Microhardness of TiN layers depending on the type of layer formation

Formation conditions of TiN layer	HV_{50}, MPa
eatched Ti	150
alpha Ti	290
alpha Ti/ Ti layer /TiN	2000
alpha Ti./TiN	1080
eatch.Ti /TiN/Ti layer	440
12X18H10T	250

Wear resistance of TiN coatings.

In this work we used an accelerated method of wear resistance tests. An abrasive grain rotating at a speed of 0.285 m/s cuts the coating layer from the sample surface. Therefore in this case grinding should be considered as super-fast cutting (scratching) surface layers of the sample by a large number of the smallest grinding grains (cutters) cemented into one sheaf [5]. We have found that TiN coating's wear resistance in conditions of accelerated sliding on an abrasive cloth doesn't depend on the size of grains while the wear pattern is rather complex. One can see on the wear curves the first (lapping) wear of the sample, then there comes a time the TiN film slipps on the electrocorundum's abrasive grain (90-95% Al2O3) and after the film destruction the loss of metal substrate's mass takes place. Fig. 4 shows the wear resistance of titanium alloy samples OT4-1. For comparison here is the wear of steel 12X18H10T.

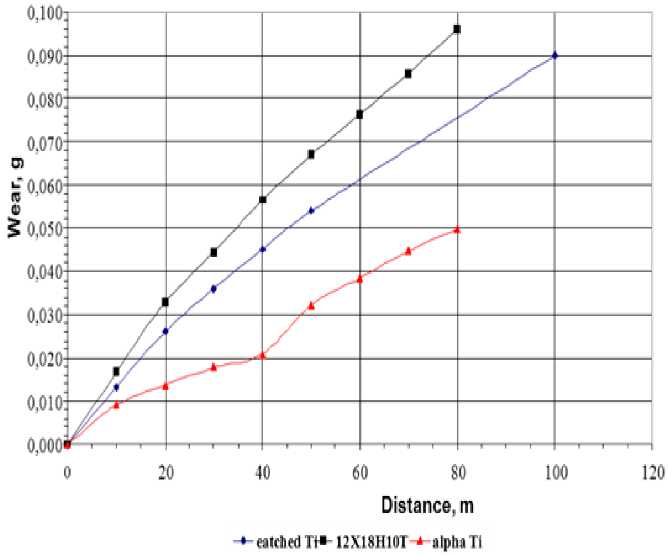

Fig.4. Wear resistance of initial samples

As follows from fig. 4, replacement of steel 12X18H10T with the titanium alloy OT4-1 allows to reduce the wear almost twice (At l=30 m the wear of m = 0,044 g (12X18H10T), for gas-saturated OT4-1, m=0,023 g). As well as in a case with a microhardness, a wear of a titanium alloy depends on the surface state. A gas-saturated (alpha) layer protects a titanic alloy against abrasion. The alpha layer's wear pattern is complex because the layer on the sheet's surface is uneven, friable and has a large surface roughness

Investigation of wear resistance of TiN layers revealed its increase in comparison with a metal substrate in all the studied samples. The TiN layer's wear pattern depends on microhardness, an original surface state and the method the coating is formed.

Figure 5 shows changes in the wear pattern according to technological parameters of the coating process (time-thickness, the potential bias). With increase in the layer's thickness (spraying duration) the wear resistance and microhardness also increase.

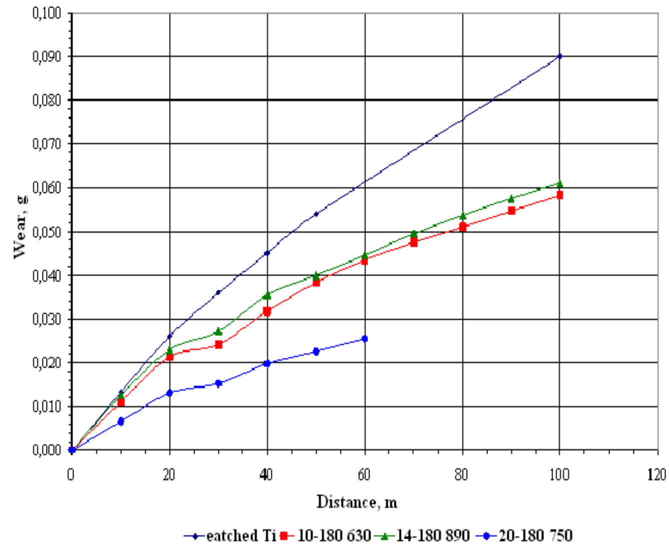

Fig.5. Wear resistance of TiN layer

But the most interesting is the wear resistance of multilayer coatings (fig. 6). TiN layers were formed either on etched or on alpha surface. Microhardness of these layers is presented in table 1. In all the samples studied an increase in the wear resistance was observed. The greatest wear was seen in etch. Ti/Ti/TiN sample with the maximum microhardness of HV50=2000 MPa. The sample was double-layer: the first layer contained plastic Ti and the second layer had solid TiN. The layer thickness was 9,5 microns, however the wear of this layer was observed at the first meters when it was sliding on a grinding skin number 10.

Considerable wear was also observed for the layer TiN, formed on an alpha case surface. The sliding distance was not more than 20 m. For a double-layer coating of etch.Ti/TiN/Ti we recorded the lowest point of microhardness HV50=440 MPa, but the wear (0,018 g) and destruction were found while sliding 36 meters. An alpha case layer on a titanium plate has the maximum stability (40 m slip) and thus the greatest wear (0,020 g).

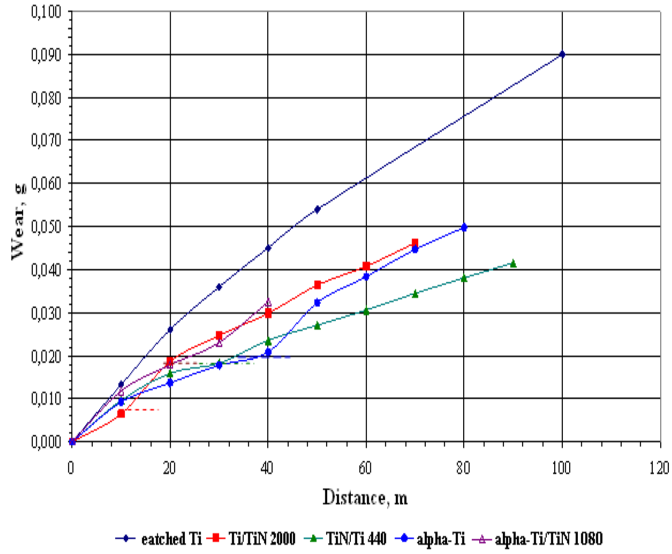

Fig.6. Wear resistance of TiN layer

The results of abrasive wear of the samples for which we have found the maximum wear resistance are given in fig. 7.

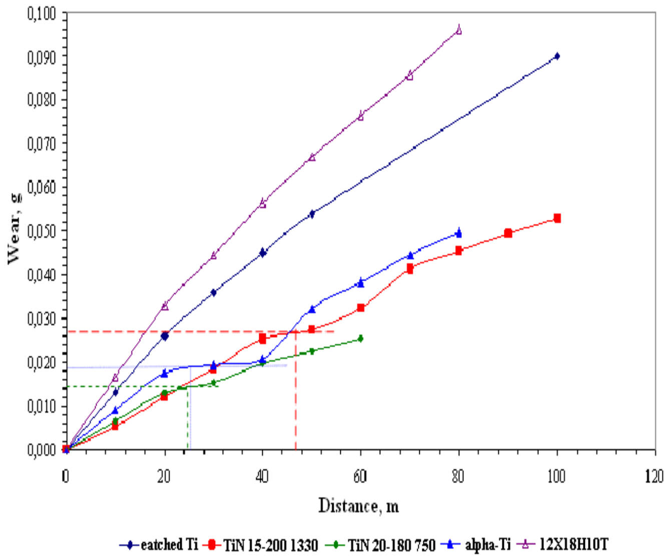

Рис.7. Wear resistance of TiN layer

For performing a comparative analysis it is necessary, in our view, to analyze the region of the «shelf» though even the grinding area indicates various wear patterns of a TiN layer, an alpha case layer and an etched surface of the titan. The calculated coefficients of friction also show different wear patterns of nitride layers formed under different conditions.

Type of material	Coefficients of friction
12X18H10T	0,87
eatch. Ti	0,57
alpha condition Ti	0,52
TiN (15-200-1330)	0,51
TiN (20-180-750)	0,50

4. Conclusion

The samples of OT4-1 alloy both in etched and in an alpha states are more abrasive wear- resistant as compared with the steel alloy 12X18H10T. Alpha layers's wearing out starts at a distance of 24 m while sliding on Abrasive number 10. TiN layer's destruction with the maximum microhardness starts when it is sliding for 23 m, but its wear is less than for alpha titanium alloy's case layer. A TiN layer having microhardness HV50> 1330 MPa starts to break down when sliding distance is 45 m and has the same wear as the titanium alloy the sliding distance of which gets 20 m. Thus as a result of ion-plasma deposition of TiN layers on a titanium alloy OT4-1 is observed surface hardening, reducing wear and friction coefficient.

5. Acknowledgments

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