Low-energy plasma-immersion implantation of nitrogen ions in titanium by a beam with ballistic focusing

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Abstract. The results of experiments on low-energy implantation of nitrogen ions into VT1-0 titanium alloy are presented. Processing was performed by a nitrogen ion pulsed beam obtained using a ballistic ion focusing system. An ion source was a nitrogen plasma of the non-self-sustained gas arc discharge with a thermionic cathode. It has been shown that when the specimens are processed in such a system, hardness of the surface increases from 1.5 to 2.5 times. In addition, the surface of the specimens undergoes ion etching which causes the formation of an etching cavity whose profile depends on the ion effect parameters.

1. Introduction

Increasing the working properties (hardness, wear resistance, etc.) of products made of titanium and its alloys is an important scientific and technical issue, in the solution of which aircraft and engine technology, automotive industry, space industry, etc. are interested. At the moment, one of the most popular methods for improving properties of the products is surface modification which involves changing surface properties of the product without changing its bulk properties. This approach is often sufficient and cheaper. Methods that involve changes in the elemental and phase composition of near-surface layers of materials are frequently used to modify the surface by putting additional substances into the surface. Among them, ion-plasma methods that use substances in the ionized state are more attractive. An ion implantation method has been widely used. It is realized by transferring added energy (tens or hundreds of kiloelectronvolts) to ions into the surface of the substance [1]. However, this method is limited by the magnitude of the ion projection distance in the surface (tens of monolayers). Another widely used method for changing elemental and phase composition of the surface is a diffusion saturation method [2]. It involves heating the workpiece to temperatures of 400-800°C at which diffusion of the atoms of the added substance ensures the production of modified layers in tens–hundreds of micrometers per ones-tens of hours. Diffusion saturation process of the surface with nitrogen atoms is called nitriding and is widely used in industry. Ion-plasma nitriding of titanium and its alloys makes it possible to increase the hardness of its surface by several times. However, substrate temperatures should be higher than 700°C [3]. At such temperatures, titanium loses its strength due to the growth of the grains. Thus, an important task for realizing the processes of obtaining modified nitrogen-containing near-surface layers in titanium substrates is a decrease in the temperature of nitriding process. In [4], it is shown that an increase in the ion current density causes an intensification of the nitriding process, and at the same time, transfer of energy (several hundred of electronvolts) to ions makes it possible to carry out both effective ion-beam cleaning of the surface from the oxides formed on it and realize their low-energy implantation [5] .To increase an ion current pulse density, a ballistic focusing system of an ion beam in a spherical configuration was proposed [6]. The use of this system for AISI 5140 steel processing showed a good result. The disadvantage of this system is a small processing area (approximately 2 cm^2). Accordingly, a similar system of cylindrical configuration has been developed, which allows processing surfaces of a larger area.

In this paper, the results of experiments on low-energy implantation with nitrogen ions of titanium (VT1-0) at a temperature of 600°C in the ballistic focusing system of an ion beam from a non-self-sustained arc gas discharge with a thermionic cathode are presented.

2. Experimental setup

The experiments were performed on a plant with dimensions of a vacuum chamber of 900×900×1100 mm, which was pumped out by a turbomolecular pump with a capacity of 1000 l/s to a pressure of not higher than 5×10^{-2} Pa. Gas plasma generation was carried out by the PINK source in the axial configuration [7]. The ballistic focusing system of the ion beam was a box-shaped housing with a rectangular base of 120×240 mm, one side of which was covered by a metal grid with a cell of 0.5× 0.5 mm and a geometric transparency of approximately 53% which was a part of the cylinder of radius 75 mm. The focusing system was located at a distance of 180 mm from the end of the hollow cathode of the PINK generator (figure 1). The specimens of 45×20×3 mm, made of VT1-0 titanium were pre-wiped with benzine and placed on a collector of the ballistic focusing system whose temperature was measured by a chromel-alumel thermocouple. The collector of 45×90 mm was electrically isolated from the housing of the ion focusing system, which allowed to measure the incoming ion current separately. The plasma generator was powered by a welding transformer with an output voltage of up to 70 V and a current of up to 200 A. Heating of the tungsten thermionic cathode of the source was carried out by a transformer with a thyristor current regulation and a frequency of 50 Hz. Accelerating voltage pulses were fed by a specially developed source with an output voltage of up to 2 kV, a current of up to 3 A with an adjustable frequency of up to 50 kHz and a pulse duty factor of 15 to 80%.

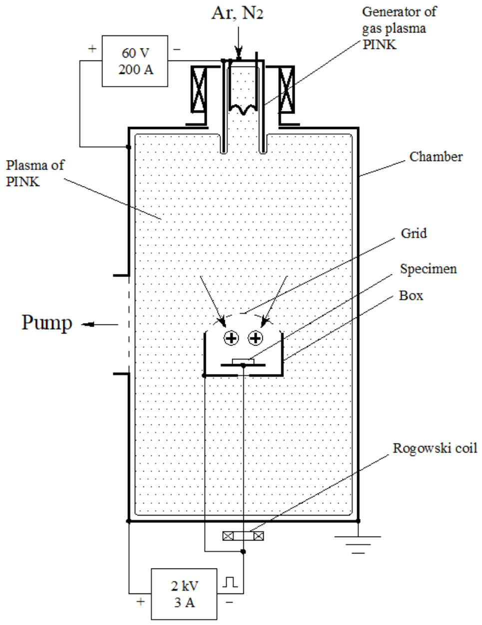

Figure 1. Experimental scheme.

The specimens were preliminary cleaned and heated with argon ions with an energy of 1 kV for 20 minutes. Implantation was carried out at nitrogen operating pressure of 0.5 Pa for 1 hour. After processing, the specimens were cooled in the working chamber to a temperature of not higher than 70°C with a chamber pressure of not higher than 5×10^{-2} Pa.

The measurement during the experiments were carried out by the LeCroy waveRunner 6050 oscillograph, total current of the accelerating voltage source and collector current were fixed on the Rogowski coils. The shapes of voltage pulses were fixed too.

Studies of the specimen relief were carried out using a STIL 3D Micromesure optical profilometer. The surface hardness was measured by a PMT-3M microhardness tester.

3. Results and discussion

All three specimens were processed at the same ion energy, which was set by the pulse bias voltage (1400 V) and different voltage pulse duty factors. Repetition rate of the voltage pulses remained unchanged at 40 kHz. To maintain the same temperature of the specimens (600°C), plasma density was varied by adjusting the discharge current of the plasma generator. The processing modes are given in table 1. The fixed mean values of the ion current arriving at the collector are also presented in this table.

Table 1. The Specimen treating regimes.

Specimen number	Duty factor, %	Discharge current, A	Collector average current, мA	Collector average current per pulse, мA	Total ion average current, мA
1	40	50	145	250	830
2	60	28	135	210	740
3	80	20	95	120	610

It can be seen, that as the discharge current of the plasma generator decreases, both total ion current and current to the collector decrease, but this does not cause a decrease in the measured specimen temperature. It can be explained by an improvement in ion beam focusing with increasing cathode layer width. The increase in the cathode layer width, under the conditions of a constant accelerating voltage, is caused by a decrease in the plasma density with a decrease in the discharge current of the plasma generator, and reduces the form-factor effect of the woven accelerating grid. The temperature was measured by a thermocouple mounted on the back of the specimen, in the middle of the collector. An improvement in the ion beam focusing increases power density directly opposite the thermocouple. The total power delivered by the ion beam to the entire specimen surface decreases. It should cause an increase in the temperature gradient from the center of the specimen to its edges.

Studies of the specimens surface profiles showed that during processing the specimens were subjected to ion etching with different intensities (figure 2). Against the background of the initial curvature of the specimens, an etching cavity of different depth and width was observed at their center (in the region of the focus line). When pulse duty factor increases, the depth of the cavity grows and the width decreases. It can be seen that the depth of the cavity grows from approximately15 μm on specimen 1 to approximately 25 μm on specimen 3. However, it is impossible to quantify the increase in the depth of the cavity because different regions of the specimens are subjected to different etching intensity. However, analyzing a cavity shape it can be stated that ion current density in the ion beam focus region increased with the improvement of the focusing conditions and it caused an exacerbation of the cavity. After processing, the appearance of grains on specimens 2 and 3 in the focusing region of the ion beam (width of 4–6 mm) was observed. This indicated a local overheating of the specimens in this region.

Surface microhardness studies for each specimen were carried out at the center of the etching cavity and at a periphery of 6 mm from it at two loads on the indentor of 50 and 100 g (table 2).

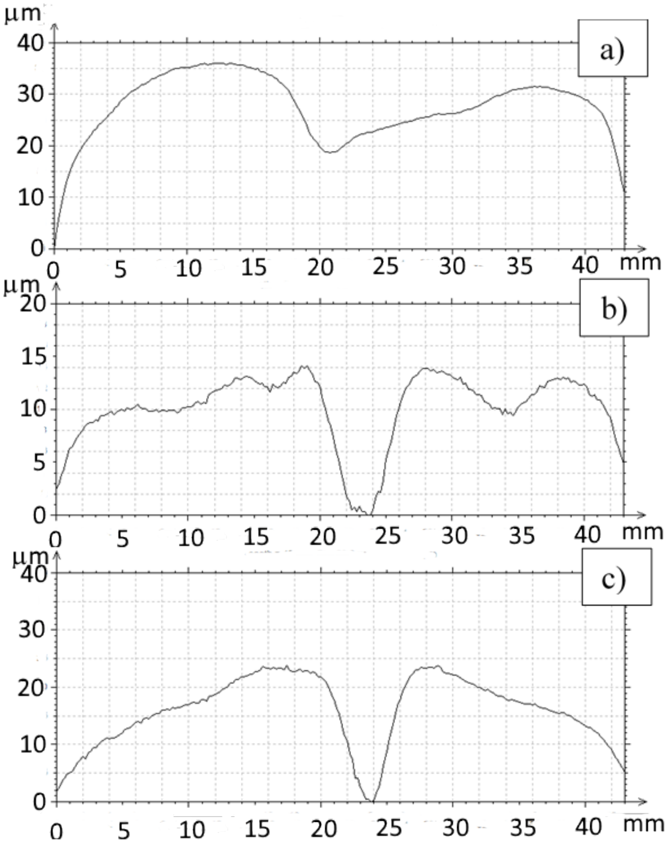

Figure 2. Specimen surface profiles: a) - specimen 1; b) - specimen 2; c) - specimen 3.

It can be seen (table 2) that the hardness increased both at the center of the etching cavity and at its periphery, with the greatest hardness observed on specimen 2. The lower hardness at the center of the etching cavity on specimen 1 can be due to the lower density of the ion current in this location and, as a consequence, a lower local surface temperature. A slight decrease in the hardness on specimen 3 can be explained by local overheating of the etching cavity region. The hardness at the periphery of the etching cavity on specimen 2 is also the largest, which can be explained by the fact that at this point there were optimum local temperatures (higher than for specimen 1 because of better ion beam focusing and local overheating of the etching well) and ion current density (greater than for specimen 3 because of the worst ion beam focusing). However, further studies are necessary to confirm it. The substantial difference in hardness at different loads on the indenter indicates that the thickness of the modified layer is not large and the penetration depth of the indenter does not exceed 10% of it. Thus, the thickness of the modified layer does not exceed 30 µm, or it significantly decreases in depth.

Table 2. The specimen surface microhardness.

Specimen number	Indenter load 50 g				Indenter load 100 g			
	Center		Periphery		Center		Periphery	
	Hardness, GPa	Penetration depth, µm	Hardness, GPa	Penetration depth, µm	Hardness, GPa	Penetration depth, µm	Hardness, GPa	Penetration depth, µm
1	3.8	2.2	3.8	2.2	3.4	3.3	2.9	3.6
2	4.3	2.1	5.8	1.9	3.5	3.3	3.8	3.2
3	4.2	2.2	4.0	2.2	3.0	3.5	3.3	3.4

4. Conclusion

1) The processing of VT1-0 titanium by low-energy ion-beam nitrogen implantation method was performed.

2) It is shown, that microhardness of the surface increases by 2.5 times for one hour. The surface is subjected to intense ion etching, with the formation of the etching cavity.

3) It is shown that maximum hardness of the surface is achieved not at the center of the etching cavity but at its periphery, which can be due to the factors including both local ion beam density and local specimen surface temperature.

4) Estimates of the modified layer depth show that its thickness does not exceed 30 μm, or the hardness of this layer has a strong gradient deep into the surface.

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