Effect of low-temperature ion nitriding on microhardness, roughness and residual stresses in the surface layer of Ti-6Al-4V

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Abstract. The paper presents the results of two experimental series with low-temperature nitriding of VT6 titanium alloy in a non-self-maintained high-current arc discharge and in a glow discharge in different regimes. It is shown that microhardness and depth of a surface layer are significantly impacted by the temperature of treatment. The value of surface roughness of the samples is also affected, with a temperature rise from 450 to 600°C resulting in a surface roughness increase by 2–2.5 times. The temperature of nitriding affects the sign and magnitude of residual stresses in a surface layer.

1. Introduction
In modern engineering, structural titanium alloys are of particular importance, having a unique combination of high specific strength and corrosion resistance, which makes them highly attractive for wide use in aviation, automotive and chemical industry, medicine and other fields [1–3]. The development of aviation technology imposes a number of increased requirements for the efficiency of parts thereby challenging the improvement of their physical and mechanical properties [4].

Damages are mostly initiated by macro- and microdefects, residual stresses on the surface, mechanical and physical properties of a surface layer, corrosion, fretting corrosion, high-temperature gas corrosion, erosion, wear, etc. The efficiency of parts under operating conditions is ensured through the selection of appropriate specific operating conditions of materials and surface treatment technology (mechanical, thermal, chemical-thermal, etc.) [5]. There is a variety of ways to improve physical and mechanical properties of titanium alloys. The most common include various surface modification methods. Nitriding is one of the most effective methods to ensure surface treatment of titanium alloys, which increase their surface hardness, wear resistance, corrosion and erosion resistance. However, relatively high temperatures (800–900°C) are primarily applied for nitriding titanium alloys, which in turn entails significant changes in the structure and properties of the initial material and can even change the surface microgeometry [6–9].

Therefore, active work is currently underway both abroad and in Russia, to study the low-temperature ion nitriding of titanium alloys. Particularly relevant is the reduction in the temperature of nitriding when treating complex, thin-walled or critical parts of titanium alloys. Conducting low-temperature nitriding makes it possible to maintain a high surface finish and avoid the deformation of products [9, 10].
2. Experimental approach
The samples of Ti-6Al-4V titanium alloy that looked like 80×10×1 mm³ plates were chosen as the object of study. Ion nitriding was carried out in the temperature range of 450–600°C for 60 minutes in a nitrogen-argon gas mixture (15%N₂ + 85%Ar).

By way of comparison, some samples were treated with a non-self-maintained high-current arc discharge using PINK plasma source with an incandescent cathode installed in NNV-6.6-I1 installation, while others were treated in a glow discharge on modernized ELU-5M installation.

The study was conducted against the original samples, the microhardness of which was 350 kgf·mm⁻². The uniformity of microhardness distribution over the surface was determined by measuring the microhardness diagonally at regular intervals. To measure the depth of a nitrated layer, inclined microsections were prepared with an angle of 6° to the monitored surface. The depth of a modified layer was determined by the hardness distribution curve to the value of initial hardness. Microhardness measurements were carried out applying the method of reconstructed imprint in accordance with the Russian State Standard GOST 9450-76 using the Struers Duramin-1/-2 microhardness tester. The value of static load applied to a diamond indenter for 1 second was 490 [mH] (50 g).

Roughness of the samples before and after nitriding was measured using the MarSurf PS1 roughness tester, while surface residual stresses were measured on the DRON-4 X-ray diffractometer.

3. Results and discussion
A visual analysis after ion nitriding in a non-self-maintained high-current arc discharge revealed that the samples appeared dull with a barely noticeable golden hue. The samples treated in a glow discharge acquired a golden hue, which indicated the existence of a thin layer of titanium nitride on the surface [6].

Surface microhardness measurements showed that after nitriding the microhardness increased both in the non-self-maintained high-current arc discharge and in the glow discharge as compared to the initial value.

Surface microhardness of the samples subjected to low temperature ion nitriding is shown in table 1.

| T (°C)          | Microhardness before nitriding (kgf·mm⁻²) | Microhardness after nitriding (kgf·mm⁻²) |
|-----------------|-------------------------------------------|------------------------------------------|
| After nitriding in non-self-maintained high-current arc discharge |                                            |                                          |
| 450             | 350±10                                    | 437±10                                   |
| 500             |                                             | 453±10                                   |
| 550             | 465±10                                    | 628±10                                   |
| 600             |                                             |                                          |
| After nitriding in glow discharge |                                            |                                          |
| 450             | 428±10                                    |                                           |
| 500             | 432±10                                    |                                           |
| 550             | 441±10                                    |                                           |
| 600             | 462±10                                    |                                           |

An increase in surface microhardness after ion nitriding is related to a thin film of titanium nitride formed on the surface of the samples [6]. Thus, the maximum surface microhardness after ion nitriding in the non-self-maintained high-current arc discharge was found on a sample treated at...
\[ T = 600°C \] and amounted to 672 kgf-mm\(^{-2}\), while the minimum – on a sample treated at \( T = 450°C \) with a value of 437 kgf-mm\(^{-2}\).

After ion nitriding in the glow discharge at \( T = 600°C \), surface microhardness was 462 kgf-mm\(^{-2}\). The values on the surface of the samples treated at temperatures of 450, 500, 550°C remained approximately the same.

Figure 1 shows the dependences of a microhardness change over the depth of a nitrated layer.

The analyzed dependences showed that the temperature of ion nitriding, both in the non-dependent high-current arc discharge and in the glow discharge, has a significant effect on the depth of the nitrided layer. Thus, after ion nitriding in the non-self-sustained high-current arc discharge at a temperature of 450°C, the depth of the hardened layer was \( \sim 10 \) µm. With a temperature rise up to 600°C, the depth of the nitrided layer increased by 3 times and was \( \sim 30 \) µm. The depth of the modified layer after treatment at temperatures of 500 and 550°C was 14 and 20 µm respectively.

When nitriding was conducted in the glow discharge, a temperature rise from 450 to 600°C brought about an increase in the depth of the nitrided layer from 8 to 24 µm.

To reveal the effect of low-temperature ion nitriding on surface roughness, the roughness value of the original sample was measured before processing and appeared to be \( Ra = 0.337 \) µm. The results of measuring the surface roughness of the samples are shown in table 2.

After ion nitriding, both in the non-self-sustained high-current arc discharge and in the glow discharge, there was a deterioration in the surface quality of the samples. Moreover, the higher the temperature of treatment, the worse the surface quality. With a temperature rise from 450 to 600°C, the roughness value of the surface being treated increased by 2–2.5 times.

The minimum increase in roughness arose after ion nitriding in the glow discharge at temperatures of 450 and 500°C. At low temperatures, the surface quality was attributable primarily to its relief caused by the volume expansion due to the formation of nitrides. Moreover, surface roughness of the samples after processing at higher temperatures was mainly due to the effects of its expansion and sputtering coupled with the repeated precipitation of nitrides [11].

| \( T \) (°C) | Initial roughness, \( Ra \) | Roughness after processing in arc discharge, \( Ra \) | Roughness after processing in glow discharge, \( Ra \) |
|---|---|---|---|
| 450 | 0.337 | 0.457 | 0.357 |
| 500 | | 0.51 | 0.414 |
| 550 | | 0.721 | 0.507 |
| 600 | | 0.745 | 0.622 |
When nitriding in the arc discharge, the treated surface is bombarded with ions of the working gas, thus leading to ionic cleaning from oxide and nitride layers. However, it is known that when a sample is bombarded with ions of the working gas, selective etching occurs, which contributes to an increase in surface roughness [11].

The authors [12] argue that a surface layer is crucial for fatigue resistance within parts. Its quality can be characterized by roughness, physical state of the material, and above all residual stresses. Moreover, compressive residual stresses contribute to a fatigue crack growth, whereas tensile stresses – to its decrease.

Table 3 presents the measurements of residual stresses in a surface layer of the samples.

| Temperature of treatment | Residual stress values $\sigma$ (kgf-mm$^{-2}$) |
|--------------------------|-----------------------------------------------|
| initial                  | $+7.1 \pm 1$                                  |
| after treatment in non-self-sustained high-current arc discharge | $+0.5 \pm 1$                                  |
| 450 °C                   | $+4.9 \pm 1$                                  |
| 500 °C                   | $+5.7 \pm 1$                                  |
| 550 °C                   | $+12.4 \pm 1$                                 |
| 600 °C                   | $-23.7 \pm 1$                                 |
| 500 °C                   | $-19.7 \pm 1$                                 |
| 550 °C                   | $-8.4 \pm 1$                                  |
| 600 °C                   | $-8.3 \pm 1$                                  |

After nitriding in the non-self-maintained high-current arc discharge, tensile stresses arise on the surface of the samples, which can later cause accelerated generation and development of cracks. The results presented in table 3 show that a decrease in the temperature of treatment led to a decrease in the magnitude of surface residual tensile stresses.

On the surface of the samples treated in the glow discharge, compressive residual stresses arose, and a decrease in temperature led to an increase in their magnitude. After processing at a temperature of 450°C, the value of residual stresses was $-23.7 \pm 1$ kgf-mm$^{-2}$.

4. Conclusion

After low-temperature ion nitriding in non-self-maintained high-current arc discharge, the samples appeared dull with a barely noticeable golden hue. The samples treated in glow discharge acquired a golden hue, which indicated the existence of a thin layer of titanium nitride on the surface.

After nitriding, the distribution of microhardness over the surface and the depth of the samples is fairly uniform, without sharp transitions to the core.

The temperature of treatment has a significant effect on surface microhardness and depth of a nitrated layer. Thus, after ion nitriding in non-self-maintained high-current arc discharge at a temperature of 450°C, the depth of a modified layer was $\sim 10$ μm. Increasing the temperature to 600°C resulted in an increased layer by a factor of 3 and amounted to $\sim 30$ μm.

When conducting the nitriding in glow discharge, a temperature rise from 450 to 600°C also led to an increase in the depth of a nitrided layer from 8 to 24 μm.

The temperature of ion nitriding affects the surface quality of the samples. Thus, with a temperature rise from 450 to 600°C, the roughness value of the surface being treated increased by 2–2.5 times.

It was defined that after low-temperature ion nitriding in non-self-maintained high-current arc discharge, tensile residual stresses were generated on the surface, and after processing in glow discharge – compressive. The temperature of nitriding affected the sign and magnitude of surface residual stresses.
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