Investigation of low-temperature ion nitriding technology of titanium alloy Ti-6Al-4V

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Abstract. The paper presents the results of low-temperature nitriding in non-self-maintained high-current arc discharge and in glow discharge. Measurement results of microhardness across the depth of a nitrated layer are given. It is found that the depth of a nitrated layer depends on treatment temperature. The temperature increase from 450 to 600 °C during nitriding in non-self-maintained high-current arc discharge leads to the increase in the depth of a nitrated layer from 10 to 24 μm, and from 10 to 22 μm when treated in a glow discharge. The paper also describes data on residual stress in a surface layer of samples after low-temperature ion nitriding.

1. Introduction
Compressor blades of a gas-turbine engine (GTE) are classified as critical parts of an engine and determine its lifetime and operational integrity. Nowadays, the aircraft engine industry faces a problem of increasing the strength properties of compressor blades [1].

Reliability and durability of blades significantly depend on physical-chemical and structural-phase composition, microgeometry and residual surface stress. The residual stress in a surface layer has a significant influence on fatigue resistance within parts operating under static and fluctuating loads. The compressive residual stress positively influences the fatigue resistance, while the compressive residual stress of a surface layer is caused by formation and development of fatigue crack growth and, hence, the decrease in lifetime of blades [2, 3].

The formation of compressive residual stress is solved through ion implantation. However, due to its characteristics ion implantation cannot be applied for treatment of monowheels since it may cause interblade area overlap (only treatment of single blades is possible).

Gas nitriding does not seem possible since treatment is carried out at 800–900 °C which leads to the growth of grains and deterioration of material properties. The authors suggest low-temperature ion nitriding having the following advantages: monitoring of treatment parameters in a variety of operating modes (temperature, pressure, ion current density, particle energy); high saturation rate, high degree of fineness; high performance of parts made from hard titanium alloys [4, 5].

2. Methodology
The research was carried out using a two-phase titanium alloy Ti-6Al-4V widely applied in the aviation industry. Two types of samples were used in the experiment: disc-shaped 80×10×1 mm plates of 20 mm in diameter and 5 mm thickness.
A series of experiments were performed at various modes on low-temperature ion nitriding of Ti-6Al-4V titanium alloy in non-self-maintained high-current arc discharge generated by plasma source and in a glow discharge. Different modes of low-temperature ion nitriding are shown in table 1.

**Table 1. Modes of low-temperature ion nitriding.**

| № modes | Temperature, °C | Duration, h | Operation environment composition |
|---------|-----------------|-------------|-----------------------------------|
| Nitriding modes in non-self-sustaining high-current arc discharge | | | |
| 1       | 450 ± 10        | 1           | 100 % N₂                           |
| 2       | 600 ± 10        | 1           | 100 % N₂                           |
| Nitriding modes in glow discharge | | | |
| 1       | 450 ± 10        | 1           | 85 % Ar + 15 % N₂                  |
| 2       | 600 ± 10        | 1           | 85 % Ar + 15 % N₂                  |

The inclined microsections with an angle of 6° to the monitored surface were prepared to measure the microhardness along the depth of the diffusion layer of nitrided samples. 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 static load applied to the diamond tip for the period of 10 s was 490 [MN] (50 g). The depth of a hardened layer was determined from the hardness distribution curve to the value of hardness of the basic material.

The microstructure of a hardened layer was studied using optical microscope Olympus GX-51. To determine the influence of the nitriding mode on the residual stresses, a method for precise measurement of changes of the interplanar distances determined from the displacement of the diffraction line was used. The measurement was carried out using the DRON-4 X-ray diffractometer.

**3. Results and discussion**

Measurements of surface microhardness showed that after nitriding there is an increase in surface microhardness which is caused by the formation of a golden nitride film and the presence of a diffusion layer [6]. Figure 1 shows variation in microhardness with respect to the depth of the diffusion layer.

![Figure 1](image-url)

**Figure 1.** Dependences of microhardness variation of the nitrided layer depth of Ti-6Al-4V alloy: (a) – basic and modified by non-self-sustaining high-current discharge; (b) – initial and modified in glow discharge.

The diagrams show that as the distance between the surface increases, the microhardness on the samples decreases smoothly, which indicates the presence of an extended layer with increased hardness.

Dependences of microhardness of the depth of the nitrided layer show that the temperature of ion nitriding, both in non-self-sustained high-current arc discharge and glow discharge, has a significant effect on the nature of nitrogen diffusion into the interior of titanium alloy during low-temperature
nitriding. For example, in case of nitriding in non-self-sustaining high-current arc discharge, an increase in temperature from 450 to 600 °C leads to an increase of the nitrided layer depth from 10 to 24 μm. In case of nitriding in glow discharge, an increase in temperature from 450 to 600 °C also leads to an increase of the nitrided layer depth from 10 to 22 μm. Figure 2 shows the structure of Ti-6Al-4V titanium alloy samples after low-temperature ion nitriding.

Figure 2. Microstructure of Ti-6Al-4V titanium alloy samples after nitriding: (a), (b) – after nitriding in non-self-maintained high-current arc discharge; (c), (d) – after nitriding in glow discharge.

The microstructure of samples after various surface treatment modes is mainly presented by equiaxial α-phase grains with boundary separation of drop-, acicular, and prolate shape β-phase (figure 2). At the same time the average size of α-and β-phase grains in their initial state makes 7.5 and 2.1 μm correspondingly.

Treatment at 450 °C does not change microstructural parameters of samples and grain phases remain equiaxial. At 600 °C there is a growth of separate crystallites which illustrates the beginning of secondary recrystallization. Microstructural parameters of a boundary layer are similar to the parameters of a material.

Residual stress in a surface layer after low-temperature ion nitriding was determined for disc-shaped samples. The values of the residual stresses after ion modification are presented in table 2. The presence of compressive stresses on the surface of the initial sample of 7.1 kgf/mm² could be bound to preliminary machining (turning, grinding, etc.), which usually leads to the residual stress in a thin surface layer of materials.

After nitriding in non-self-sustained high-current arc discharge, tensile stresses arise on the surface of samples, which can be the cause of accelerated generation and development of cracks. The results presented in table 2 show that the temperature affects the magnitude of the residual stress. Long-time holding at a temperature of 600 °C leads to an increase of tensile stresses from 5.7 to 12.4 kgf/mm², which is unacceptable for key workpieces. High temperature and prolonged exposure, apparently, leads to the degradation of structure and appearance of an α-layer. The decrease in temperature leads to the decrease in surface tensile stress.
Table 2. Residual stress values on the samples.

| № modes                                      | Residual stress values $\sigma$, kgf/mm$^2$ |
|----------------------------------------------|---------------------------------------------|
| Initial                                      | $+7.1 \pm 1$                                |
| After treatment in non-self-sustained high-current arc discharge |                                          |
| 1                                            | $+0.5 \pm 1$                                |
| 2                                            | $+12.4 \pm 1$                               |
| After treatment in glow discharge             |                                            |
| 1                                            | $-23.7 \pm 1$                               |
| 2                                            | $-8.3 \pm 1$                                |

Compressive stresses appear on the surface of samples treated in glow discharge, possibly due to formation of phases with an increased specific volume. Moreover, a decrease in temperature and exposure time of treatment leads to an increase in the compressive residual stresses and amounts to $-23.7 \pm 1$ kgf/mm$^2$.

4. Conclusion

Increase in microhardness of Ti-6Al-4V titanium alloy samples is observed after low-temperature ion nitriding both in non-self-maintained high-current arc discharge and in glow discharge.

It is found that the depth of a nitrated layer significantly depends on treatment temperature. The temperature increase from 450 to 600 °C during nitriding in non-self-maintained high-current arc discharge leads to the increase in the depth of a nitrated layer from 10 to 24 μm, and from 10 to 22 μm when treated in a glow discharge.

The study revealed that nitriding at 450 °C does not result in the change of sample structure and at 600 °C the growth of separate crystallites is observed.

It was defined that the temperature affects the sign and magnitude of residual stresses. Thus, in case of ion nitriding in non-self-sustained high-current arc discharge at a temperature of 600 °C, the residual stress was presented at the range of $+12.4 \pm 10$ kgf/mm$^2$. When nitriding is performed in glow discharge at 450 °C, the residual stress amounted to $-23.7 \pm 10$ kgf/mm$^2$.

References

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