Nitriding of VT3-1 titanium alloy in a glow discharge with hollow cathode

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Abstract. The paper presents research results of VT3-1 titanium alloy samples subjected to nitriding in glow discharge with a hollow cathode. The influence of working gas composition on the thickness of a hardened layer at nitriding in glow discharge with a hollow cathode was studied. It was found that the increase of argon up to 80-90% in gaseous mixture during nitriding in glow discharge with a hollow cathode leads to the increase of the hardened layer thickness to ~ 90 microns and surface microhardness to ~ 850 HV₀.₁, and contributes to a smaller gradient of the surface layer microhardness. The hardened layer on the surface of VT3-1 titanium alloy obtained upon nitriding in glow discharge with a hollow cathode is less fragile and resists plastic deformation more efficiently in comparison with the hardened layer obtained via traditional ion nitriding.

1. Introduction
At present titanium alloys are widely used in aviation industry, which is specified by unique combination of their physical, mechanical and technological properties: rather low density, high strength-to-weight ratio, corrosion resistance, thermal integrity. However their application is limited by a number of disadvantages related to surface properties of titanium and its alloys. Low hardness and, as a result, wear resistance of a surface, tendency to sticking, high friction coefficient alongside with overwhelming amount of materials limit the use of titanium alloys in production of components subject to intense friction and surface wear [1, 2].

The paper suggests using glow discharge with hollow cathode to form high density plasma [3–6] that ensures efficient ion etching of a nitride film formed on a surface of a treated titanium alloy that in its turn leads to the intensification of ion nitriding. The purpose of this paper is to study the influence of nitriding in glow discharge with a hollow cathode on the surface structure and properties of VT3-1 titanium alloy.

2. Experimental approach
Figure 1 shows the layout of an upgraded set-up ELU5-M used for titanium alloy nitriding in glow discharge with a hollow cathode. In order to form a cathode void over the sample a screen from titanium alloy with holes was installed and placed above the same current potential (Fig. 1). As a result high density plasma is formed in a cathode void, which leads to intense nitrogen saturation of a surface.

Two-phase VT3-1 titanium alloy (Ti–6Al–2Mo–1Cr), which is used in production of build-in and stamp-welded components, is able to operate long times at 400–500 °C. The samples were subject to preliminary quenching and aging.
Samples were studied via the scratch test using CSM Scratch Tester to define the influence of nitriding in glow discharge with a hollow cathode on properties of a surface layer of VT3-1 titanium alloy. Diamond pyramid hardness was used as an indenter. The applied load was changed with the linear movement of indenter from 0.03 N to 30 N at the speed of 12 N/min. The path length of the indenter was 5 mm, the speed of its movement was 2 mm/min.

![Diagram of experimental set-up](image)

**Figure 1.** Experimental set-up for sample nitriding in glow discharge with hollow cathode

### 3. Results and discussion
To define the influence of gaseous mixture composition on the thickness of a hardened layer of VT3-1 titanium alloy, samples were subjected to nitriding in glow discharge with a hollow cathode at a temperature of 800 °C during 4 hours in gaseous mixtures with various contents of nitrogen and argon. Figure 2 shows microstructures of sample sections from VT3-1 titanium alloy obtained using an optical microscope.

![Microstructures](image)

**Figure 2.** Microstructures of sample sections from VT3-1 titanium alloy subjected to nitriding in glow discharge with a hollow cathode at 800 °C during 4 hours in gaseous mixtures with various contents of nitrogen and argon

The analysis of the obtained microstructure images showed that samples with light poorly etched layer that were nitried in gaseous mixture with higher content of argon have higher thickness. The thickness of a light layer of samples nitried in gaseous mixture containing 80% Ar and 20% N₂ makes about 70 microns; then the layer is smoothly transformed into the microstructure of a basis while
samples nitried in gaseous mixtures with higher content of Ar and N\textsubscript{2} are presented as films with the thickness of several microns.

Data obtained as a result of the analysis allow to make a conclusion that nitriding in glow discharge with a hollow cathode in gaseous mixtures with low content of nitrogen in VT3-1 alloy leads to the formation of a distinct light diffusion layer of about 70 microns thick which is smoothly transferred into the microstructure of a basis. The increase of nitrogen in gaseous mixture of up to 60–80\% leads to the formation of a thin light non-etched film on the surface of alloys.

Microhardness profiles by layer depth obtained via measurements of microhardness on section surfaces were obtained in order to assess the thickness of the reached hardened layer after nitriding in glow discharge with a hollow cathode in gaseous mixtures with various contents of nitrogen and argon. Dependences of microhardness on distance from the surface of samples of VT3-1 titanium alloy obtained through measurements are shown in Figure 3.

![Figure 3](image)

**Figure 3.** Distribution of microhardness of VT3-1 titanium alloy surface layers nitried in glow discharge with hollow cathode in gaseous mixtures with various contents of nitrogen and argon within 4 hours

The analysis of obtained distributions showed that nitriding in glow discharge with a hollow cathode of both alloys in gaseous mixture with 20\% nitrogen allows to receive the biggest values of both the surface microhardness and thickness of the hardened layer. The thickness of this layer after nitriding during 4 hours amounts to 70-90 microns, microhardness smoothly decreases to values of microhardness of a basis that is one of the main requirements imposed on the nitried layer.

The obtained experimental data and its further analysis demonstrate that nitriding of VT3-1 alloy in glow discharge with a hollow cathode is the most efficient in gaseous mixtures with low (15-20\%) concentration of nitrogen. Despite a high saturant gradient, nitriding in mixtures with high content of nitrogen is less efficient as it leads to the formation of a nitride film thus creating a barrier for nitrogen diffusion deep into the material. High content of argon in gaseous mixture leads to intense etching of a nitride film and, as a result, to the formation of big diffusion layers. Besides, high content of argon in gaseous mixtures ensures more intense heating up to the working temperature and nitriding at lower burning voltage of the discharge.

Figure 4 shows dependences of acoustic emission on the imposed load obtained as a result of a scratch test applied for all samples. The analysis of obtained dependences showed that active breakdown of a surface layer of a sample nitried in traditional glow discharge begins at the smallest imposed loads while when the sample was nitried in glow discharge with a hollow cathode, the wave peaks of acoustic emission were registered starting from the imposed load of 1.8 N. This fact allows to make a conclusion that the surface of titanium alloys nitried in glow discharge with a hollow cathode...
has bigger hardness and abrasive wear resistance in comparison with the surface nitried in traditional glow discharge.

![Acoustic emission, %](image)

**Figure 4.** Acoustic emission obtained during a scratch test of VT3-1 titanium alloy samples nitrided in glow discharge

Figure 5 shows images of scratches of the studied samples obtained using an optical microscope.

![Images of scratches](image)

**Figure 5.** Images of scratches formed on VT3-1 titanium alloys following the scratch test, a – nitriding in traditional glow discharge, b – nitriding in glow discharge with hollow cathode

The analysis of images (Fig. 5) revealed chips at the edge of scratches of a surface nitride layer, typical for brittle failure, and the size of the width of such chips for samples nitried in glow discharge with a hollow cathode is smaller, which may indicate smaller fragility of the surface layer of these samples.

4. **Conclusion**

As a result of the research it was found out that the increase in argon content in gaseous mixtures of up to 80–90% during nitriding of VT3-1 titanium alloy in glow discharge with a hollow cathode during 4 hours at 800 °C leads to the increase in thickness of the hardened layer up to ~ 90 microns and surface microhardness up to ~ 850 HV$_{0.1}$, and contributes to the formation of a smaller microhardness gradient of a surface layer.
The hardened layer on the surface of VT3-1 titanium alloy obtained upon nitriding in glow discharge with a hollow cathode is less fragile and resists plastic deformation more efficiently in comparison with the hardened layer obtained via traditional ion nitriding.

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