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Low temperature ion nitriding of Ti-6Al-4V alloy

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Abstract. The paper presents the results of nitriding the titanium alloy VT6 (Ti-6Al-4V) in a non-self-maintained high-current discharge and in a glow discharge in different regimes. Nitriding was conducted in pure nitrogen and in the mixture of nitrogen and argon. It is shown that microhardness of the samples increased; however, the efficiency of surface modification depends on the modification conditions.

1. Introduction
Currently, titanium alloys play an important role in aviation and space industry due to heat resistance, high specific strength, high corrosion resistance, low density and non-magnetization.

However, along with the positive characteristics such alloys also demonstrate disadvantageous properties: low surface hardness and poor tribological characteristics [1, 2].

Surface modification can improve physical and mechanical properties of titanium alloys. The use of traditional gas nitriding in this case is impossible because this type of treatment requires temperatures in the range of 800-900 °C. Ion nitriding is one of the most efficient methods of surface treatment that increase hardness, wear resistance, and corrosion resistance. The temperature of nitriding of titanium alloys can be reduced in glow discharge and non-self-maintained arc discharge plasmas. Low temperature nitriding can preserve high quality of surface and exclude deformation of final products [3,4,5].

The aim of this work is to study the impact of low temperature ion nitriding on the microhardness and depth of the hardened Ti-6Al-4V layer.

2. Experimental approach
The Ti-6Al-4V alloy with chemical composition presented in Table 1 was used as the initial material for the investigations.

| Table 1. Chemical composition of steel [wt%] |
| Fe | C | Si | V | N | Ti | Al | Zr | O | H |
|---|---|---|---|---|---|---|---|---|---|
| 0.3 | 0.1 | 0.15 | 3.5–5.3 | 0.05 | 86.485–91.2 | 5.3–6.8 | 0.3 | 0.2 | 0.015 |

Literature analysis has shown that gas composition influences the hardness of a surface [5]. Nitriding of the titanium alloy was carried out in the mixture of argon and nitrogen of different concentrations. It is also known [1-5] that the depth of a nitried layer is substantially impacted by temperature and time of exposure.
In the study, several samples of VT6 titanium alloy (Ti-6Al-4V) were subjected to nitriding in a non-self-maintained high-current arc discharge using PINK plasma source [6] with an incandescent cathode installed in NNV-6.6-I1 installation [6] under the following conditions (Table 2):

| Sample No. | Temperature of treatment [°C] | Treatment time [h] | Composition of the working fluid |
|------------|-------------------------------|-------------------|----------------------------------|
| 1          | 550                           | 3                 | 60% Ar, 40% N₂                  |
| 2          | 500                           | 1                 | 20% Ar, 80% N₂                  |
| 3          | 400                           | 1                 | 20% Ar, 80% N₂                  |
| 4          | 500                           | 3                 | 100% N₂                         |
| 5          | 400                           | 1                 | 100% N₂                         |

The other samples were subjected to nitriding in a glow discharge on modernized ELU-5M installation under the following conditions (Table 3):

| Sample No. | Temperature of treatment [°C] | Treatment time [h] | Composition of the working fluid |
|------------|-------------------------------|-------------------|----------------------------------|
| 1          | 500                           | 3                 | 85% Ar, 15% N₂                  |
| 2          | 500                           | 3                 | 85% Ar, 10% N₂, 5% H₂            |
| 3          | 500                           | 1                 | 85% Ar, 10% N₂, 5% H₂            |
| 4          | 450                           | 1                 | 85% Ar, 15% N₂                  |

The initial microhardness of the VT6 specimen surface subjected to quenching and aging was 330 kgf/mm². The uniformity of microhardness distribution over the surface was determined by measuring the microhardness diagonally at regular intervals with PMT-3M microhardness tester. The depth of the modified layers was measured in cross sections at angle α ≈ 6°. A visual analysis of the samples was used to investigate color uniformity against the initial state in order to identify possible surface defects.

3. Results and discussion
The visual analysis showed that after ion nitriding in a non-self-maintained high-current arc discharge, the samples appeared dull with a barely noticeable golden hue (Figure 1a). The samples treated in a glow discharge acquired a golden hue with slight color heterogeneity (Figure 1b).

Surface microhardness tests showed that the hardness increased in all samples in the range of 348-672 kgf/mm². Surface microhardness for the samples treated with non-self-maintained arc discharge and glow discharge is shown in Tables 4 and 5, respectively.

| Regime | Microhardness before nitriding [kgf/mm²] | Microhardness after nitriding [kgf/mm²] | Hardening [%] |
|--------|------------------------------------------|------------------------------------------|---------------|
| Sample 1 | 672                                      | 104                                      |               |
| Sample 2 | 528                                      | 60                                       |               |
| Sample 3 | 330                                      | 13                                       |               |
| Sample 4 | 493                                      | 49                                       |               |
| Sample 5 | 348                                      | 6                                        |               |
Figure 1. General view of the samples before and after ion nitriding: a) samples treated in non-self-maintained high-current discharge; b) samples treated in glow discharge.

Table 5. Surface microhardness of the samples before and after ion nitriding

| Regime  | Microhardness before nitriding [kgf/mm²] | Microhardness after nitriding [kgf/mm²] | Hardening [%] |
|---------|------------------------------------------|------------------------------------------|---------------|
| Sample 1 | 353                                      |                                          | 7             |
| Sample 2 | 389                                      |                                          | 17.8          |
| Sample 3 | 390                                      |                                          | 18            |
| Sample 4 | 389                                      |                                          | 17.9          |
| Sample 5 | 393                                      |                                          | 19            |
| Sample 6 | 395                                      |                                          | 20            |

The increase of the microhardness for the samples treated in glow discharge is less significant as compared to those treated in non-self-maintained arc discharge.

Changes in the profile along the microhardness depth of the samples processed in non-self-maintained arc discharge are shown in Figure 2. The maximum surface microhardness of nitrided samples of 672 kgf/mm² was found in Sample 1. The minimum surface microhardness of nitrided samples of 348 kgf/mm² was found in Sample 5. Microhardness in Samples 2, 3 and 4 was 528, 373 and 493 kgf/mm², respectively. Thus, the surface hardness and growth kinetics of the nitried layers were influenced by nitriding temperature at the greatest [7, 8]. The proportions of gas composition do not have any significant impact on hardness and depth of the nitried layers.

Hardness-depth changes of the samples after nitriding in a glow discharge are shown in Figure 3. The hardness on the surfaces of all samples that were ion nitrided in glow discharge was about 390 kgf/mm². The maximum depth of the hardened layer was found in Sample 2 and reached 17 μm. The dependencies in Figure 3 show that the temperature change of 50 °C had no significant effect on the surface hardness. Addition of hydrogen increases the depth of the modified layer. This is probably due to the fact that hydrogen reacts with oxygen, which results in reduced oxygen concentration in the surface layer and increased nitrogen adsorption sites on the surface. This in turn enhances nitrogen in the sample volume and increases the hardness of the surface layer [6, 9].
Figure 2. Hardness-depth changes of samples after nitriding in a non-self-maintained arc discharge.

Figure 3. Hardness-depth changes of samples after nitriding in glow discharge.

4. Conclusion
Samples that were subjected to ion nitriding demonstrate sufficiently even microhardness distribution on the surface and in the volume of the material without sharp transitions to the core.

The study showed that ion nitriding can be efficient in improving microhardness. The microhardness and depth of a surface layer are greatly influenced by temperature.

Visual study showed that non-self-maintained high-current discharge nitriding leads to only slight change of sample color. The samples processed in a glow discharge acquired a golden color due to the appearance of titanium nitride on their surfaces.
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