Nitriding of Titanium Alloy VT-16 in Plasma of Glow and Arc Discharges

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Abstract – Results of experiments on nitriding of titanium alloy VT-16 (Ti-3Al-4.5V-5.0Mo) in plasma of non-self-sustained glow and arc discharges are presented. Nitriding process was held in helium-nitrogen mixture in non-self-sustained glow discharge plasma at 550 and 450 °C and in self-sustained arc discharge plasma at 550 °C, and in commercially pure nitrogen in non-self-sustained arc discharge plasma at 450 °C. Treated samples were investigated by microhardness measurements. It was found that nitriding of titanium alloy VT-16 in the plasma of glow and arc discharges at 550 and 450 °C provides the increase of surface microhardness up to 1.5–2 times.

1. Introduction
Small relative density, high corrosion resistance and biological compatibility of the titanium and its alloys have led to its wide use in medicine and aerospace industry. However, low hardness and, accordingly, low wear resistance of these materials are the reasons restricting their wider use.

In most cases, it is enough to raise hardness not of all the material, but only of its surface by coating or diffusion penetration of alloying elements [1–2]. Now various surface modification methods – vacuum-arc coating, ion nitriding and etc are used.

In present work, for surface modification of titanium VT-16 alloy (Ti-3Al-4.5V-5.0Mo) samples the nitriding method in the plasma of glow and arc discharges [3–4] has been chosen.

2. Material and a research technique
As a material for investigation the titanium alloy VT-16 preliminary exposed to water quenching from temperature 880 °C was chosen. Then samples with dimensions 10×15 mm and thickness ~1.5 mm were cut from the bulk material. After that, samples were ground and polished.

Treated samples were investigated by the method of optical metallography (optical microscope MMR-4), microhardness measurements (microhardness tester PMT-3).

3. Nitriding in non-self-sustained glow discharge with a hollow cathode
Nitriding of titanium VT-16 alloy was carried out in non-self-sustained glow discharge with a hollow cathode, which provides relatively high current densities (at some mA/cm²) at low (at some Pa) work gas pressures.

The experiment scheme of nitriding in non-self-sustained glow discharge plasma is presented in Fig. 1.

The nitrogen was supplied by two-channel automatic system SNA-2.

The nitriding of samples was carried out in a helium-nitrogen mixture at working pressure ~1.1 Pa. The gas was supplied by two-channel automatic system SNA-2.

The temperature of samples was measured by chromel-alumel thermocouple installed directly on the back (hidden from plasma affecting) side of sample through a quartz glass.

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During the nitriding, one part of the samples was mounted under negative voltage bias, and the other part was mounted under floating potential (on the isolated holder in the hollow electrode 2). The nitriding temperature was \(-550\) and \(-450^\circ\text{C}\) for the samples fixed on the holder. The temperature of the samples under floating potential was not measured. Duration of the nitriding process was 3 hours.

4. Nitriding in self-sustained arc discharge plasma

At nitriding we used plasma generator with cold hollow cathode, where arc discharge operates in crossed electric and magnetic field. At work in a wide pressure range \(p = (10^{-2}–10) \text{ Pa}\) generator at discharge voltage \((30–60) \text{ V}\) provided discharge current up to \((10–100) \text{ A}\) and plasma density in vacuum chamber \(n = (10^7–10^{10}) \text{ cm}^{-3}\).

The experiment scheme of nitriding in the plasma of self-sustained arc discharge is presented in Fig. 2.

![Fig. 2. The experiment scheme of nitriding in the plasma of self-sustained arc discharge: 1 – holder; 2 – additional two plates holder; 3 – sample; 4 – arc arrester; 5 – short magnetic coil; 6 – hollow cathode; 7 – trigger electrode; 8 – gas inlet; 9 – arc discharge power supply; 10 – negative voltage power supply](image)

The nitriding of samples was carried out in a helio-nitrogen mixture \((\text{He}:\text{N}_2 = 40:60\%)\) at working pressure \(\sim 1.1 \text{ Pa}\). The sample was fixed between two plates made of titan VT1-0 so that the plasma was not able to penetrate between them. As a result of such fixing, samples were not exposed to an ion bombardment, and there was only an interaction of neutral molecular or atomic components of the plasma on the sample surface.

The temperature of samples was measured by chromel-alumel thermocouple isolated by means of a quartz glass, installed on the sample fixed between two plates of the holder.

5. Nitriding in non-self-sustained arc discharge plasma

The installation scheme (Fig. 3) and analyses of nitrided samples in the plasma of non-self-sustained arc discharge are considered in detail earlier in [4].

![Fig. 3. The experiment scheme of a nitriding in the plasma non-self-sustained arc discharge](image)

In present experiment, nitriding was carried out in a commercial nitrogen. Working pressure was 0.8 Pa. Samples were fixed on a surface of the rotating massive holder mounted in the centre of the chamber, and on the ceramic insulator placed on the holder. In the latter case, samples were mounted under the floating potential.

The temperature of samples was measured by chromel-alumel thermocouple isolated by means of a quartz glass, installed in a blind hole in the holder centre.

At shown key position, the ion regime was carried out. Changing the key position, the regime of heating by electrons was carried out.

The process of heating up to the nitriding temperature was taken place by switching the regimes of ion and electron affect at regular intervals within an hour. The nitriding temperature was \(\sim 450^\circ\text{C}\) for the samples fixed on the massive holder. The temperature on the samples mounted under the floating potential was not measured. Duration of the nitriding process was 2 h.

6. Results and discussions

Microhardness measurements on a samples surface were carried out using the loading on the indenter of 0.5 N and loading time about 10 s. Results of the measurements are presented in the Table.

Investigation of samples surface by the method of optical metallography has shown, that after nitriding of a sample in the non-self-sustained glow discharge at temperature 550°C with biasing there is an intensive sample surface etching mainly at the grain boundaries.

After the nitriding at temperature of 450°C etching of the biased sample surface, practically, was not ob-
Modification of Material Properties

served. On a sample surface which was mounted under the floating potential, grain boundaries which have kept the structure of quenched martensite (Fig. 4) are visible.

Table. Dependence microhardness of titanium VT-16 alloy on temperature and bias voltage

<table>
<thead>
<tr>
<th>Samples number</th>
<th>Treatment mode</th>
<th>T, °C</th>
<th>Ubias, V</th>
<th>HV0.5, GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>GD</td>
<td>550</td>
<td>−900</td>
<td>4.6</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>−</td>
<td>0</td>
<td>6.0</td>
</tr>
<tr>
<td>3</td>
<td>SAD</td>
<td>450</td>
<td>−650</td>
<td>5.7</td>
</tr>
<tr>
<td>4</td>
<td>NAD</td>
<td>−</td>
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<td>550</td>
<td>0</td>
<td>5.1</td>
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<tr>
<td>6</td>
<td></td>
<td>450</td>
<td>−450</td>
<td>6.5</td>
</tr>
<tr>
<td>8</td>
<td></td>
<td>−</td>
<td>0</td>
<td>3.5</td>
</tr>
</tbody>
</table>


![Photo of sample surface](image)

Fig. 4. The photo of the sample surface after nitriding under the floating potential

After the nitriding process in both non-self-sustained glow discharge and self-sustained arc discharge in the conditions when samples were mounted under the floating potential at 550 and 450 °C during 3 h titanium nitrides were formed on samples surface. It was confirmed by yellow surface color. At the same time the microhardness of treated samples surfaces increased to ~1.5–2 times in comparison with the initial hardness.

At nitriding in the plasma of non-self-sustained arc discharge with the supplying of negative potential to the samples the surface etching is observed. While on the sample, which was mounted under the floating potential, essential changes of surface structure were not observed. During of electron heating, owing to bad thermal contact and the high power input, the sample that had holder potential was heated up to ~600-700 °C. Probably, it also has served as the reason of hardness increase.

7. Conclusion

The nitriding in the plasma of non-self-sustained glow discharge allows to carrying out low temperature nitriding (up to 450 °C) of titanium alloy VT-16. Thus, there is an increase in microhardness of treated samples surfaces in ~1.5–2 time in comparison with the initial hardness.

The increase of nitriding temperature by the increase of bias voltage leads to etching of the sample surface.

When the samples are mounted under the floating potential during the nitriding in plasma of the non-self-sustained glow discharge and the self-sustained arc discharge it allows to carry out the saturation of titanium alloy VT-16 by nitrogen effectively without etching of surfaces by accelerated ions.

References