**Kinetic Regularities of Interaction of Sintered Titanium with Molecular Nitrogen at Atmospheric Pressure**

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Abstract — The kinetics of gas nitriding of the sintered titanium at a temperature of 800 °C was investigated. Parameters of surface roughness and surface microhardness were evaluated.

Key words — VT1–0 titanium alloy, sintered titanium, surface microhardness, roughness, gas nitriding, kinetics of gas nitriding, powder metallurgy.

I. Introduction

The products and billets of titanium alloys, in particular VT1-0, are widely used in engineering, medicine, chemical and food industries, etc. However, their use is limited by the tendency to sticking, seizure in friction pairs with other metals, and also high-value of products. Nitriding and oxidation are the effective methods to strengthen the surface layers and improve the tribotechnical properties of titanium. The method of powder metallurgy allows the bring down the price of products obtained from the titanium alloys. Meanwhile the utilization factor of material can reach 95%. The structure of the products obtained by powder metallurgy differs from the compact (produced by the traditional technology) by the randomly distributed pores, mainly irregular shape [1-4].

The purpose of this work is to study the kinetics of gas nitriding of sintered VT1-0 titanium by nitrogen and evaluate the strengthening effect of nitriding.

II. Materials and methods of research

The samples of c.p. titanium VT1-0 produced by the traditional technology and powder metallurgy (sintering) were nitrided. The sintered samples were produced from PT5-1 titanium powder. The powder was pressed in a hydraulic press at a pressure of 700 MPa. The billets were sintered in a vacuum furnace at the temperatures of 1200 ... 1300 °C for 3 h. Then they were cooled with the furnace. A vacuum was 13,3 Pa. The prismatic billets were cut by the electroerosive method on the samples of 10 × 15 × 2 mm. The surface of the samples was grinded and polished.

The thermodiffusion saturation of the samples was performed in a nitrogen-containing environment at the atmospheric pressure. For nitriding, nitrogen gas of technical purity was used (GOST 9293-74). The kinetics of nitriding was studied at the temperature of 800 °C for 5, 10 and 20 h.

The change of weight of the samples after saturation was evaluated by the gravimetric method on an OHAUS Voyager balance with an accuracy of ± 0.1 mg. The surface roughness was measured by a 170621 roughness indicator determining the mean arithmetic deviation of the surface profile Rₐ, µm. The surface hardening was determined by measuring the microhardness with a PMT-3M microhardness tester under a load on an indenter of 0,981 N. The depth of nitrided layer was evaluated by the microhardness gradient.

III. Results and discussion

The golden and dense nitride film is formed on the surface of the sintered and compact samples as the result of the isothermal exposure in a nitrogen atmosphere [5]. The increase of the isothermal exposure at 800 °C from 5 to 20 h causes an increase of the intensity of the golden color. It was established that the intensity of the interaction of samples, produced by powder metallurgy, by the nitrogen, is much higher than samples produced by the traditional technology (Fig. 1). Thus, the weight increment of the compact samples at 10 h exposure is 0,537 mg/sm², while for sintered samples of titanium is 6,25 mg/sm².

![Fig. 1. Dependence of change of weight titanium produced by different technologies on the duration of saturation in the nitrogen of an atmospheric pressure at the temperature of 800 °C.](image)

It is known that nitrogen atoms are the interstitial atoms, so the defects in the crystal lattice (vacancies and grain boundaries) play a significant role in the mechanism of nitriding [5]. Since the defect density of crystal lattice in the porous material is much larger than in the compact material, so the intensity of saturation of the samples produced by powder metallurgy by nitrogen is higher. Thus, the pores, which come to the surface of the sample, play the decisive role since the clusters of defects in the crystal lattice are near pores and grain boundaries. In addition, at the presence of open porosity the nitrogen transfers to the inner layers of the sample and the nitriding process occurs in the sample.

Nitride film which is formed on the surface of the samples after thermodiffusion saturation has a distinctive...
relief. The increase of the duration of nitrizing process causes an increase of surface roughness as both the compact samples and the sintered samples (fig. 2, a). In this case the worsening of surface quality is less noticeable on the sintered samples.

The surface microhardness shows the hardening effect of the surface of the titanium alloy after thermochemical treatment. In the initial state the hardness of the compact and sintered samples is the same ≈2 GPa (fig. 2, b). With increasing isothermal exposure it was observed a general tendency to increase the surface microhardness, and therefore to a higher strengthening level of the titanium regardless of technology.

![Graph showing surface roughness and microhardness](image)

The nitride layer which includes δ-nitride TiN_x and ε-nitride Ti_xN is formed on the surface of the compact and sintered titanium after the thermodiffusion saturation. The increase of duration of the nitrizing process leads to an increase of the thickness of this layer as both the compact samples and the sintered samples. The diffusion sublayer of the solid solution of nitrogen in α–titanium is formed under the surface phase film as a result of diffusion of nitrogen into titanium matrix. So, the depth of diffusion layer at the isothermal exposure of 5 h is ≈ 50 μm (fig. 3). On the titanium produced by the traditional technology, the reinforced layer has the structure with increased α-grains which is little etched at the same time on titanium produced by powder metallurgy the layer structure is little observed.

It is difficult to determine the depth of the diffusion layer of sintered titanium. During thermodiffusion saturation by nitrogen the atoms penetrate into the material deep, that causes a sharp increase of hardness as both the surface layers and a matrix of samples. The hardness of the matrix of the sintered samples is from 3,8 to 2,5 GPa (fig. 3).

![Graph showing influence of thermodiffusion saturation by nitrogen](image)

Fig. 3. Influence of thermodiffusion saturation by the nitrogen at the isothermal exposure of 5 h on the microhardness distribution of surface modified titanium layers produced by different technologies.

#### Conclusion

It was determined that the intensity of the interaction of the titanium samples produced by powder metallurgy with nitrogen is much higher than the samples produced by traditional technology. The weight increment of the compact samples at the 10 h exposure is 0,537 mg/sm², while the samples sintered from powder titanium PT5 -1 is 6,25 mg/sm². The surface microhardness of the sintered samples is the same as the compact samples regardless of isothermal exposure. Thermodiffusion saturation by nitrogen strengthens the surface layers of the sintered titanium and increases the hardness of the titanium matrix. The surface quality of the titanium is deteriorated regardless of the production technology.

#### References


