

Formation of Nitride by Electron-Beam Irradiation of Titanium in Nitrogen Media

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Abstract

This paper demonstrates the possibility of nitriding of titanium by a forevacuum electron source. We present an original design of the experimental setup and the measurements of the tribological properties and elemental composition of the subsurface layer of the titanium experimental sample. Raster electron microscopy analysis of the nitride sample have demonstrated that titanium and nitrogen are found to be the main chemical elements; oxygen and carbon also present, though their total concentration does not exceed 6 wt. %. The thickness of the modified layer after a 75-minute long process of nitriding was about 8 μm . Wear resistance test of have shown that the nitrided sample has a 500 times less loss of the material as compared with the original titanium sample, meaning a times-fold increase of resistance to wear. Copyright © VBRI Press.

Keywords: Plasma, titanium nitriding, electron beam, forevacuum pressure range.

Introduction

The nitriding of metals is a widely known method of improving their operational properties [1]. It is currently generally accepted that the main effect exerted by an electron beam on a solid body, when it is irradiated in high vacuum, consists in heating [2]. At the same time, as our investigations carried out over the last years have shown, the picture can become more complicated in case of the electron beam interacting with an insulated or dielectric target under the middle vacuum conditions [3]. In such circumstances, the beam plasma and the plasma particles hitting the sample may acquire a significant role. The specifics of interaction between the solid sample processed by the beam of the charged particles and the plasma forming within its vicinity will determine the character of changes occurring in the surface layer of the sample. The method of plasma ion nitriding is used to form thin micrometer layers of transition metal nitrides on the surfaces of steel parts. It is widely used to improve hardness and durability of various parts in machine building, heavy, and aerospace industries.

In the present work, we performed nitriding of titanium using a forevacuum electron source.

Experimental

Setup for electron beam nitriding and plasma analysis

The experimental setup for nitriding of titanium is shown in Fig. 1. The plasma source was placed on the

flange of the vacuum chamber. The plasma electron source was fed by constant-voltage sources connected to the discharge and accelerating gaps. Upon applying the voltage between anode and cathode, a glow discharge is ignited.

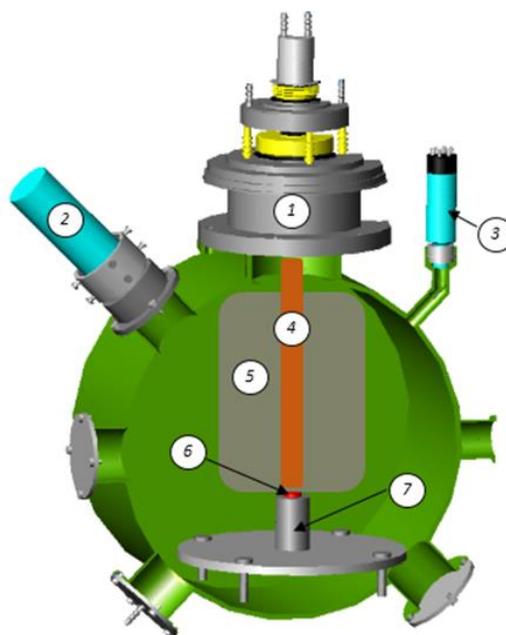


Fig. 1. Experimental setup: 1 – electron gun; 2 – optical pyrometer; 3 – vacuum meter sensor; 4 – electron beam; 5 – beam plasma; 6 – titanium sample; 7 – tantalum crucible.

The electrons were extracted through a perforated plate. In the anode-extractor gap, upon application of the corresponding accelerating voltage, the electrons were accelerated and the initial formation of the beam occurred. The electron beam formed by the source entered into the magnetic field of a short-focused magnetic coil, where the final formation of the beam took place. When propagating to the working sample, the electron beam generates the beam plasma. As a result of interaction between the electron beam and the sample surface, the sample heats up.

The chamber was pumped out to a pressure of $5 \cdot 10^{-3}$ Pa by the turbomolecular pump nEXT300D at the speed of 300 l/s, followed by nitrogen puffing up to the pressure of 8 Pa. During the process of nitriding, the beam current and the accelerating voltage were respectively 100 mA and 6 kV, and the time duration was 75 minutes. The sample temperature, monitored by a remote optical pyrometer Raytek, was 900 °C.

Since the sample surface hardening is connected with the presence of nitrogen atoms near the work piece being processed, we performed the mass-charge analysis to determine the constituent composition of the beam plasma ions in order to find optimal parameters of the electron beam for nitriding. The mass-charge analysis of the plasma ions was carried out using the modified quadrupole analyzer of the residual atmosphere RGA-100 located directly near the nitriding sample. The scheme for the measurement of the ion plasma constituents and the particular changes introduced in the analyzer design are discussed in detail in [4]. Since the process of nitriding supposes a long and intensive heating of the sample, and hence of the nearby region, the analyzer was removed for the duration of the process to avoid possible damage to its sealing materials and the mass-separator insulation.

Nitrided sample diagnostics

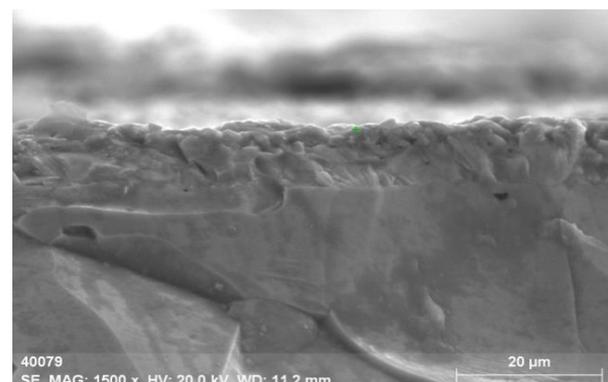
For the sample, we used sheet titanium VT-1, 4-mm thick, from which an element of the dimensions 10×10 mm was cut out. The sample was placed on the tantalum crucible and was grounded. Nitrided sample chip and the characteristic radiation spectrum of the nitrided sample was investigated shot by the scanning microscope Hitachi S3400N coupled to EDS Bruker X'Flash 5010. The X-ray phase analysis (XPA) of the modified layer was performed using a Shimadzu XRD-6000 diffractometer.

Results and discussion

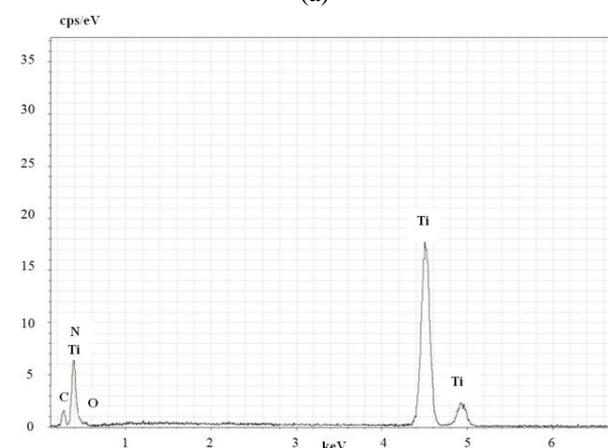
The method of raster electron spectrometry was used to determine the chemical composition of the modified layer (Fig. 2).

Titanium and nitrogen are found to be the main chemical elements; oxygen and carbon also present, though their total concentration does not exceed 6 wt. %. The minor concentration of oxygen and carbon in the modified layer is due to their presence in the

surface of the titanium sample itself and, as shown in [5], such quantities do not lead to degradation of the nitride layers. The thickness of the modified layer after a 75-minute long process of nitriding was about 8 μm .



(a)



(b)

Fig. 2. Picture of the cross-section chip (a) and the spectrum of characteristic radiation of the nitrided sample (b).

The X-ray analysis did not reveal any presence of carbide phases in the nitrided layer. However, it was detected a small concentration of the oxide phase corresponding to TiO_2 и Ti_2O_3 (Fig. 3).

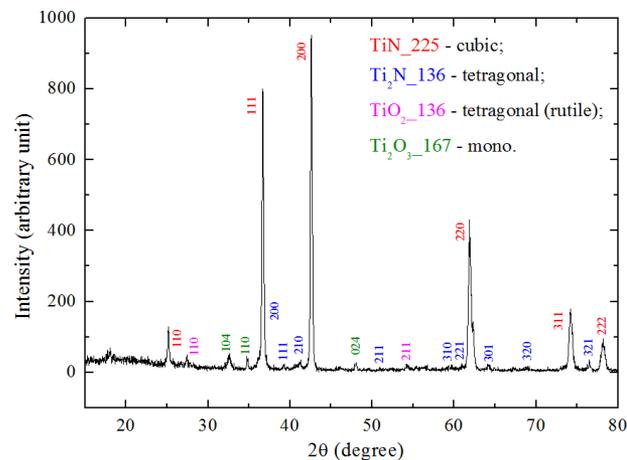


Fig. 3. Results of the X-ray analysis.

It follows from the above that the oxide layer is thin and takes up an insignificant portion of the bulk volume. Its presence is due to its being present in the original sample. The X-ray data completely corroborates the data obtained by the raster electron microscopy. The tribological characteristics were measured at room temperature using the PC-operated high temperature tribometer. This device implements the 'sphere-on-disk' measuring technique. The sample being tested is subject to exertion of a spherical tip with the force of 2 N. The tip is mounted on a rigid lever, which is fact a frictionless force sensor. The coefficient of friction is determined during the test by measuring the deflection of the elastic lever. The material wear is determined by measuring the track (Fig. 4) left as a result testing.

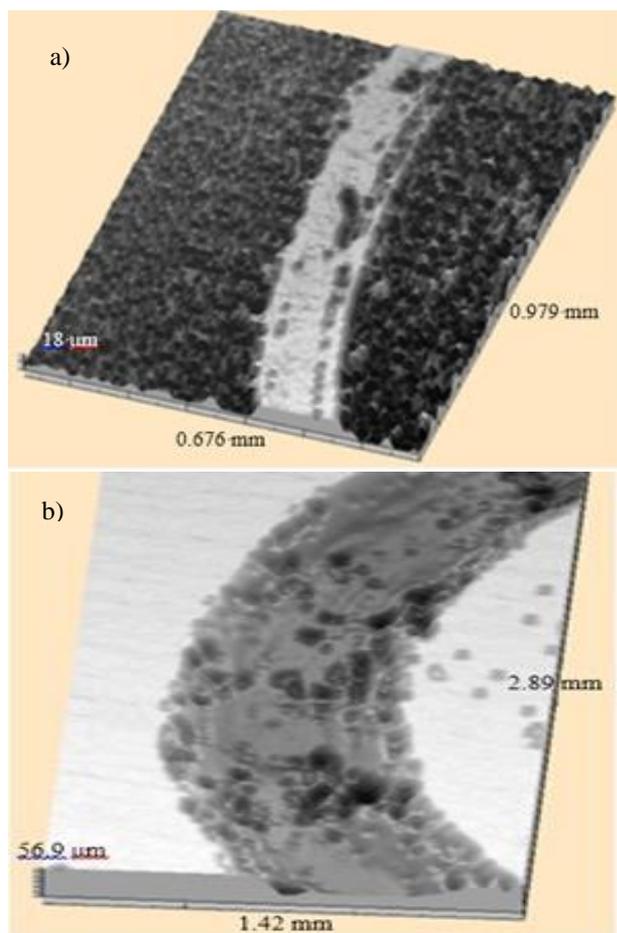


Fig. 4. Picture of the track left during tribological measurements of the initial (a), and nitride sample (b).

The nitrided sample has a 500 times less loss of the material as compared with the original titanium sample, meaning a times-fold increase of resistance to wear.

Conclusion

The experimental results on nitriding of titanium by fore-vacuum electron source, presented in this paper, demonstrate an effective modification of the surface

layer of the titanium sample, of the order of 8 μm for 75 minutes, and times-fold increase of resistance to wear as compared with the original sample. X-ray analysis have shown the presence of significant amount of nitrides (TiN , Ti_2N), small amount of oxides (TiO_2 and Ti_2O_3), and no carbides, in the nitrided layer. This result was achieved without any heating of the sample by additional heat source. Further research will be devoted to the optimization of sample temperature regime during nitriding.

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Author's contributions

Conceived the plan: EM, DA, SM;
 Performed the experiments: AV, YuG, DB;
 Data analysis: AV, YuG;
 Wrote the paper: AV, YuG, DB. Authors have no competing financial interests.

Supporting information

Supporting informations are available from VBRI Press.

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