

Microstructure and Wear Resistance of Titanium Subjected to Severe Plastic Deformation and Ion-Beam Nitriding

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Abstract – The effect of combined processing including intensive plastic deformation and ion beam nitration on structure and tribological behavior of titanium is investigated. It is shown that intensive plastic deformation of titanium results in formation of submicrocrystalline structure and increases its hardness in 50–60%, but doesn't affect tribological properties during dry sliding. The subsequent implantation of nitrogen ions leads to the formation of solid solution of nitrogen in the α -matrix. It leads to the increase of the microhardness of the modified layer up to 3700 MPa and wear resistance in 25–30 times and reduces friction coefficient in 1.5–2 times.

1. Introduction

Titanium-, zirconium-, niobium-based alloys are the most promising alloys among the present biomaterials [1] because of their relatively low density and elastic modulus and high level of biocompatibility. In spite of that, these materials possess low durability characteristics and wear resistance. That's why the problem of improving their strength and tribological properties while saving biocompatibility is very important.

Combined high-energy methods provide new possibilities in this field. In particular, intensive plastic deformation is very promising method for improving strength properties of materials [2, 3]. Nitrogen ion implantation is known to highly increase wear resistance of materials [4, 5].

So, in this paper the influence of combined treatment including intensive plastic deformation by equal channel angle pressing of titanium with the subsequent ion implantation of nitrogen on the structure composition and tribological behavior of titanium was investigated.

2. Experimental details

Titanium specimens with the dimension of 14×14×160 were prepared for intensive plastic deformation. Intensive plastic deformation was performed by equal channel angular pressing (ECAP) technique developed in Physical Technical Institute of the National Academy of Science of Belarus. ECAP regimes with 1–6 cycles provided real logarithmic deformation of 0.88–2.49 [2].

Ion beam processing was performed on the device with the source of self-contained electron drift [5]. The energy of nitrogen ions was 3 keV with the current density of 2 mA/cm². The titanium sample temperature during ion-beam nitration varies in the range 620–820 K.

The depth of the ion modified layer was measured using optical microscopy on previously etched titanium specimens in solution containing 2 ml HNO₃, 2 ml HF and 96 ml H₂O.

Hardness was measured using Vicker's indenter at 30 kg load. Microhardness was defined with the load 50 g. Ten indentations were made on each titanium specimen so statically significant values could be obtained.

The structure of deformed and ion implanted titanium was investigated using conventional X-ray diffraction technique with CoK α radiation focused by Bragg–Bretano.

Tribological testing of titanium was conducted in the dry condition using machine described in [6]. The prismatic titanium samples (8×6×3 mm) were placed in a special specimen-holder and were moved back and forth along the steel plate with the hardness HRC62. The speed of titanium specimen movement was kept near 0.1 m/s. The tribological testing was carried out with initial pressure 1 MPa. The friction coefficient was recorded on the computer during the experiment. The total friction path for both implanted and deformed titanium specimens was about 200 m. To obtain the wear rate progress the experiment was interrupted after 15, 30, 60, 120, and 180 m of friction path. The weight loss was measured using a precision automatic balancer with an accuracy of $5 \cdot 10^{-5}$ g.

3. Structure and hardness

As-received titanium has hexagonal crystal lattice with parameters $a = 0.2951$ nm, $c = 0.4689$ nm, and $c/a = 1.589$. Mean grain size is about 30 μ m. Before intensive plastic deformation titanium has relatively low amount of linear defects such as dislocations and dislocation agglomerations. This results in low hardness (1630 MPa) and low level of diffraction line broadening of α -matrix (Fig. 1).

Intensive plastic deformation by ECAP technique leads to severe structural changes of titanium samples.

Figure 1 shows the function of broadening of diffraction lines 002, 004, 101, and 202 for titanium subjected to Intensive plastic deformation.

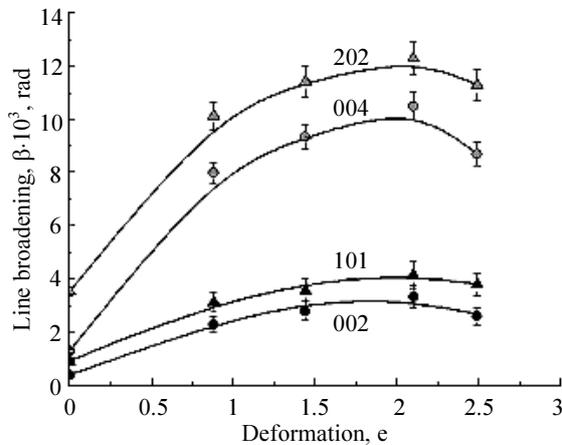


Fig. 1. Diffraction line broadening of titanium subjected to ECAP

It can be seen that line broadening increases rapidly on the early stages of deformation ($e = 0.88$ and $e = 1.44$). The ratio $\beta_{202}/\beta_{101} \approx \text{tg}\theta_{202}/\text{tg}\theta_{101}$ indicate the formation of evolved dislocation substructure with dislocations and their agglomerations randomly distributed [7]. As the result, the hardness reaches 2250 MPa after 2 cycles of ECAP deformation ($e = 1.44$) (Fig. 2).

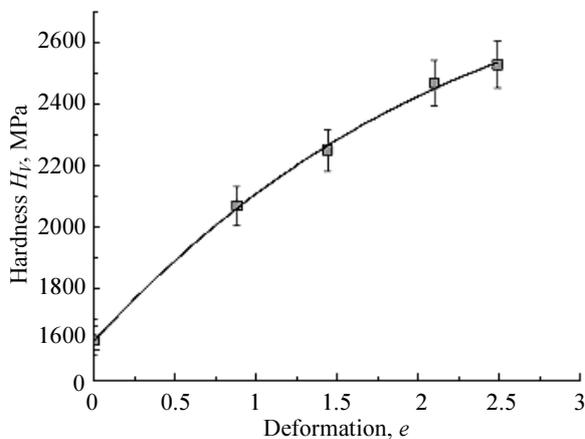


Fig. 2. Hardness of titanium subjected to ECAP

Figure 3, *a* shows the TEM image of titanium subjected to 4 cycles of intensive plastic deformation. It reveals submicrocrystalline grains of 0.1–0.3 μm in size with high dislocation density within the grains. Line broadening of titanium matrix diffraction lines reaches its maximum value $\beta_{202} \approx 12.3 \cdot 10^{-3}$ rad after 4 cycles of deformation ($e = 2.1$). The ratio β_{202}/β_{101} decreases witnessing the formation of transition state between the structure with the randomly distributed dislocations and the one with correlate distribution of dislocations which is approved by TEM observations (Fig. 3, *a*).

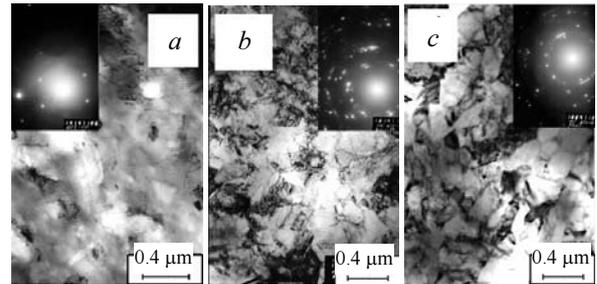


Fig. 3. TEM-images of titanium processed by various regimes: *a* – as-received; *b* – subjected to ECAP with $e = 2.1$; *c* – subjected to ECAP with $e = 2.49$

Further increase of the deformation level up to 6 cycles ($e = 2.49$) results in the significant reduction of the diffraction line broadening and β_{202}/β_{101} ratio witnessing the increase of dislocation distribution correlation and the formation of fine submicrocrystalline structure with grain size 0.1–0.3 μm .

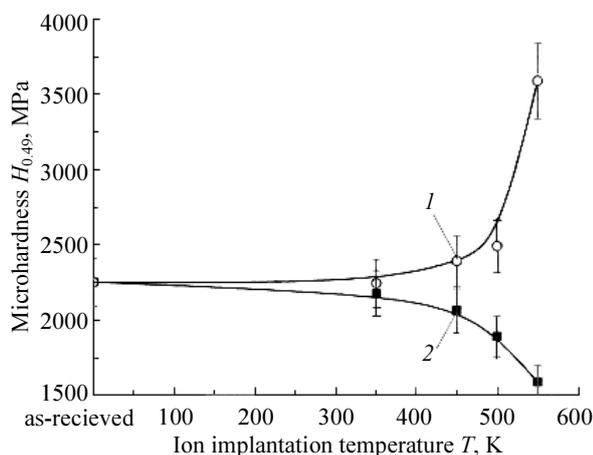
This is approved by TEM image of titanium subjected to 6 cycles ECAP deformation shown on Fig. 3, *b*. The formation of fine submicrocrystalline structure in titanium after severe (6 cycles) deformation level results in the increase in hardness up to 2500 MPa.

Low-energy high-current ion implantation of nitrogen at relatively low temperatures ($T = 620\text{--}720$ K) leads to the formation of modified layer consisting of solid solution of nitrogen atoms in hexagonal α -Ti without formation of brittle titanium nitrides. According to data obtained by optical microscopy the depth of nitrated layer is 1–2 μm . This results in faint increase of microhardness of the surface layer (Fig. 4, *a*). At the same time low temperatures of ion-beam processing doesn't activate recrystallization processes of the hard submicrocrystalline structure in titanium saving high level of strength properties of substrate.

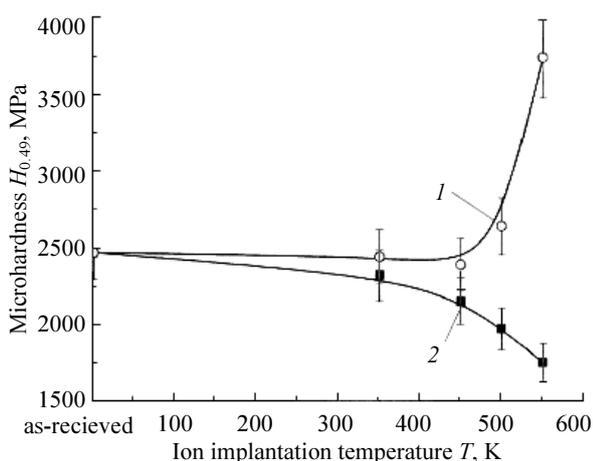
While increasing the temperature of ion-beam processing up to 770–820 K the depth of modified layer increases up to 5 μm . Figure 4, *b* shows X-ray diffraction pattern of titanium sample subjected to intensive plastic deformation and subsequent ion implantation. Asymmetrical diffusion distribution which is connected with solid solution formation of nitrogen in α -Ti matrix can be seen from the left side (higher interplanar spacing) of diffraction lines. Nevertheless, asymmetrical diffusion distribution isn't seen near the line 100. It means that the parameter c of crystal lattice of solid solution increases from 0.4689 to 0.4718 nm and parameter a remains the same. The microhardness of the nitrated layer increases up to 3500 MPa. It should be mentioned that recrystallization processes activate in deformed titanium at the temperatures 770–820 K. This results in significant decrease of substrate hardness (Fig. 4, *b*).

Thus low-energy high-current ion-beam processing of titanium at temperatures 620–820 K leads to formation of solid solution of nitrogen in titanium. Besides there are no evidence of presence of nitride phases.

It was shown that the depth of modified layer increases with temperature of ion implantation resulting in higher values of surface microhardness. But increasing the temperature of ion nitriding leads to activation of recrystallization processes of submicrocrystalline structure in titanium substrate formed by ECAP resulting in decrease of strength properties.



a



b

Fig. 4. Microhardness of deformed titanium samples subjected to ion implantation: a – deformation with $e = 1.44$; b – deformation with $e = 2.1$

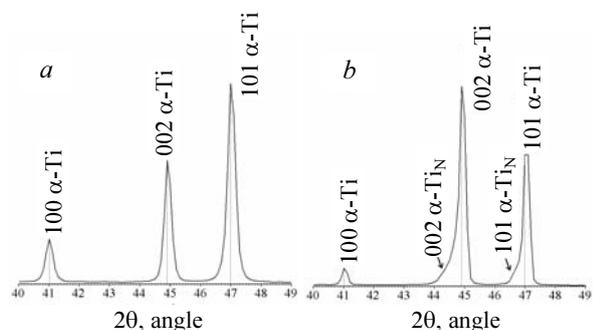
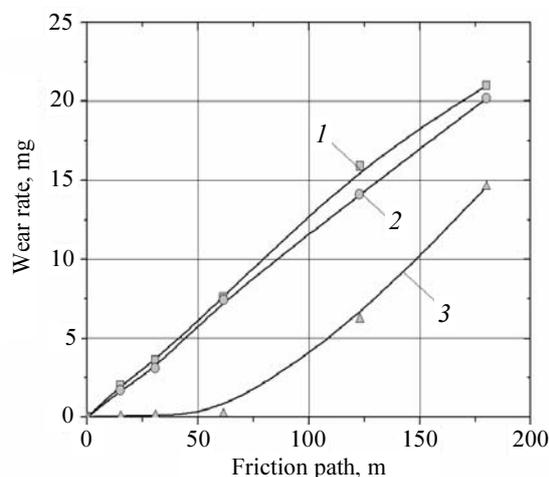


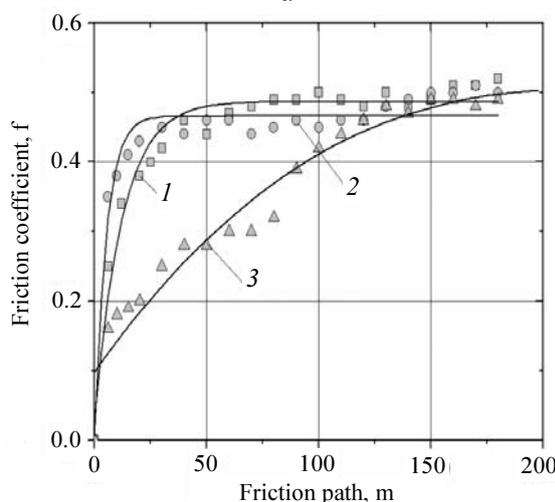
Fig. 5. X-ray patterns of as-received titanium (a) and subjected to intensive plastic deformation ($e = 2.1$) with subsequent ion implantation at $T = 770$ K (b)

4. Tribological properties

Figure 6, a shows wear rate of titanium in initial state (line 1), after intensive plastic deformation with $e = 2.1$ (line 2) and ion implantation (line 3), and Fig. 6, b shows friction coefficient of the investigated samples.



a



b

Fig. 6. Wear rate (a) and friction coefficient (b) of as-received titanium (line 1) subjected to ECAP with $e = 2.1$ (line 2) and subsequent ion implantation at $T = 820$ K

It can be seen that friction coefficient and wear rate of as-received titanium and titanium subjected to ECAP deformation are almost the same and reach the values of 0.11–0.12 mg/m and ~ 0.5 , respectively. Severe plastic deformation of near-surface layer occurs during dry sliding of titanium. Since the degree of deformation reaches the value 10^2 – 10^3 percent [8] which is comparable with deformation level after intensive plastic deformation the structure formed during friction of titanium in initial state is believed to be similar to that obtained by ECAP.

So, activation energy of the destruction is believed to be the same both for submicrocrystalline and large-grain titanium.

Ion implantation affects greatly the tribological properties of near-surface layer of titanium. It can be seen that ion implanted titanium has low wear rate 0.004 mg/m and friction coefficient 0.2–0.3. As the modified layer destroys with the increasing friction path wear rate and friction coefficient of ion implanted titanium reaches the values corresponding to unimplanted initial state of material.

It should be mentioned that friction coefficient of unimplanted titanium during dry sliding is significantly lower than that of metal with cubic lattice [9] probably because of lower shear strength of densely packed basis plane (0001). Due to location of implanted nitrogen in octahedral pores of hexagonal crystal lattice during ion-beam processing of titanium, as it was mentioned earlier, parameter c of crystal lattice increases and parameter a remains the same. Increase of interplanar spacing between basis planes (0001) leads to decrease of shear strength in them resulting in the decrease of friction coefficient of ion implanted titanium to half of that of unimplanted material. On the other hand impinging nitrogen enhances atomic bonds leading to the increase of microhardness and wear resistance of implanted material.

5. Conclusion

Intensive plastic deformation of titanium by ECAP leads to formation of great amount of randomly distributed dislocation on the early stages of deformation ($e = 0.88$, $e = 1.44$) and structure refinement and formation of submicrocrystalline structure with correlate dislocation distribution on the later stages resulting in significant increase of hardness in 50–60%.

Subsequent ion implantation of nitrogen ions in titanium causes the formation of nitrated layer of several

micrometers consisting of solid solution of nitrogen in α -Ti matrix resulting in increase in surface microhardness up to 3700 MPa.

Intensive plastic deformation of titanium does not affect its tribological properties, but subsequent ion implantation leads to enhancing wear resistance in 25–30 times, and halve friction coefficient.

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