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doi:10.15199/48.2018.07.34

Mechanical and electrical properties of the titanium surface layer irradiated with 168 MeV ^{136}Xe ions

Streszczenie. *Niniejsza praca zawiera wyniki badań przewodności elektrycznej oraz właściwości tribologicznych technicznie czystego tytanu, który został napromieniowany jonami ksenonu z energią 168 MeV z dozami 1÷5×10^{1}□ Xew/cm^2. Przedstawione zostały pomiary przewodności elektrycznej, wyniki analizy GXRD oraz wyniki pomiaru współczynnika tarcia oraz zużycia tribologicznego.* ***(Mechaniczne i elektryczne właściwości warstwy wierzchniej tytanu napromieniowanego jonami ^{136}Xe z energią 168 MeV)***

Abstract. *This paper contains the results of electrical conductivity and tribological properties tests of technically pure titanium, which was irradiated with xenon ions with energy 168 MeV with doses of 1÷5×10^{1}□ Xe/cm^2. They were presented a results of electrical conductivity, results of GXRD analysis and the results of friction coefficient and tribological wear measurements.*

Słowa kluczowe: tytan, implantacja jonowa, przewodność elektryczna, zużycie tribologiczne
Keywords: titanium, ion implantation, electrical conductivity, tribological wear

Introduction

Besides the substantial density of defects, high energy deposited in metals by swift heavy ions induces a number of specific effects: formation of tracks, local melting and amorphization of the target material, and transformation of phases [1, 2]. The most important and interesting effect is the formation of a specific spatial distribution of defects along the ion trace with a diameter of several nanometres and a length of several micrometres called tracks. It was interesting to check the changes caused by the irradiation of titanium with swift xenon ions. Titanium is a material used for construction of instruments working in space, aviation, and nuclear reactors, where it is bombarded with high-energy ions. Therefore, exploration of changes caused by swift ion radiation is essential. Titanium has several allotropic phases. The fcc phase (face centred cubic phase) is of special interest; it is observed during mechanical processing of the surface layer of titanium elements and upon irradiation of thin titanium films with swift xenon ions with energy of several hundred MeV [3, 4]. We focused our attention on changes in the mechanical and electrical properties caused by irradiation of 2 mm thick titanium samples at a temperature value close to room temperature (allowable operating temperature). These elements can be applied in practice as construction elements in devices working at high radiation, e.g. in nuclear reactors or space devices etc.

Materials and methods

Samples of commercially pure titanium (Grade2) were ground and polished to obtain a mirror-quality surface ($R_a \leq 0.04$ μm). They were not heated in order to retain the surface structure produced by the mechanical processing. Irradiation was carried out with a Xe ion beam with energy 168 MeV at fluences of 1×10^{14}, 2.2×10^{14}, and 5×10^{14} (Xe/cm^2) using an IC-100 cyclotron (ZIBJ Dubna). The projected range of xenon ions calculated using the SRIM program [5] was R_p = 11 μm – Figure 1.

Friction and wear coefficients were measured during a tribological test in the conditions of technically dry friction on a custom-made pin/ball-on disc stand [6]. A Si_3N_4 ball with a diameter of φ = 2 mm with 392 mN (40 g) load was the counter-sample. The measure of wear was the cross-sectional area of the track made by the ball. The wear traces were measured with the use of a Form Talysurf Intra Taylor Hobson profilometer.

Measurements of electrical conductivity were performed using a Sigmatest 2.069 meter calibrated with a 4.4 MS/m conductivity standard.

GXRD measurements were performed using a PANalytical Empyrean X-ray diffractometer. The GXRD diffractograms were measured at a glancing angle of Θ = 2°, which limits the X-ray radiation range in titanium to about 1 μm thick surface layer.

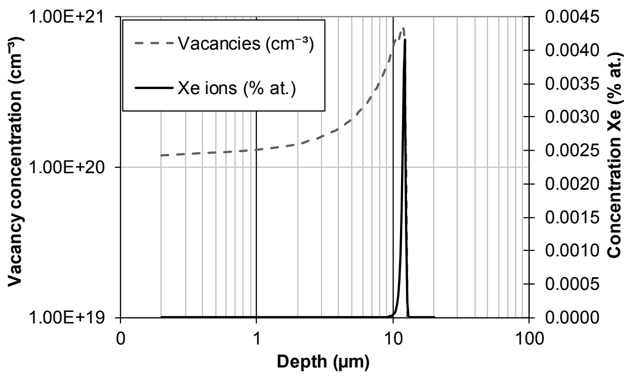

Fig. 1. SRIM calculations of Xe ion distribution profiles (168 MeV) and radiation defects in the titanium sample. The fluence in both cases was 2.2×10^{14} Xe/cm^2

Results

The results of the measurements of the friction coefficient and means wear were shown in Figure 2. The titanium irradiation decreased the friction coefficient. At the beginning of the test, its value was the lowest, i.e. ~0.28. For the first time, we observed two distinct stages in the change in the friction coefficient of the irradiated layers. In the first stage, the friction coefficient of the irradiated samples persisted at a level of about 0.28 and then increased to 0.6. In the second stage, the friction coefficient was at the level of 0.6 and increased to the value characteristic for a non-irradiated sample. The length of both these stages depends on the irradiation fluence.

Figure 3 shows the profilograms obtained after the first and second stages of changes in the friction coefficient. It can be assumed that the thickness of both layers worn during the first and second stage is about 2 μm and slightly increases with the increasing irradiation fluence.

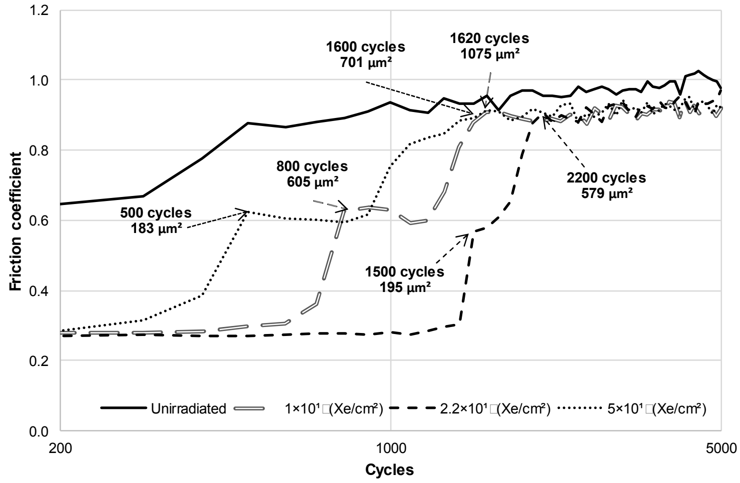

Fig. 2. Change in the titanium friction coefficient and tribological wear depending on the number of tribological test cycles

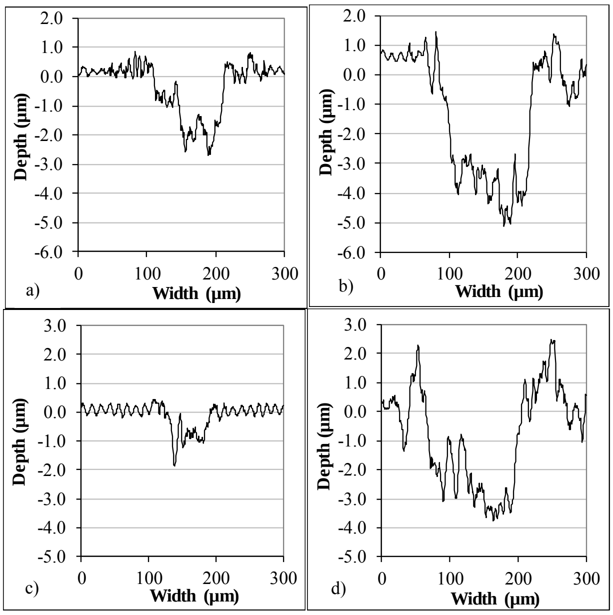

Fig. 3. Profilograms traces worn on irradiated titanium samples a) at a fluence of $1{\times}10^{14}$ Xe/cm^2 – stage 1, b) – stage 2, c) at a fluence of $5{\times}10^{14}$ Xe/cm^2 – stage 1, d) – stage 2.

The two stages of changes in the friction coefficient of the irradiated samples (Fig. 2) are probably related to the core of the trek and the surrounding thermal spike. The core of the trek ends closer to the surface than the thermal spike. Full interpretation of changes in the friction coefficient and magnitude of wear requires consideration of changes in the energy deposited by xenon ions at different depths of the sample and the related various defects in the phenomenological version of this model [7]. Theoretical calculations are quite difficult, which makes quantitative interpretation impossible at present. Another limitation is the absence of a specific trend (increase or decrease) in the sample wear depending on the irradiation fluence after 5000 cycles. After 5000-measurement cycles, the trace depth was about 6 µm (fig.4), which is a lower value than the xenon ion range (11 µm) calculated using the SRIM program [5].

Irradiation increased titanium wear over the full cycle (5000 rotations), in comparison with the non-irradiated sample - figure 5. Only the sample irradiated at a fluence of $2.2{\times}10^{14}$ Xe/cm^2 exhibited lower wear than that in the non-irradiated sample.

To acquire more information about the changes in titanium irradiated with swift xenon ions, we analysed the crystallographic structure with the GXRD method. The results of the analysis of the diffractograms are presented in table 1.

After irradiation, the sample conductivity was reduced (Fig. 6) due to radiation damage and reduction of the crystallite size. The increase in the grain number reduces the conductivity of the irradiated samples with the increasing fluence.

The irradiation of titanium reduces the crystalline lattice parameters in both the hcp and fcc phases, decreases the sizes of crystallites, and alters microstrain within the domains in both phases. The most substantial changes are induced by irradiation at a fluence of $1{\times}10^{14}$ Xe/cm^2: the contribution of the hcp lattice decreases by 7.4%, a and c lattice constants as well as crystallite sizes (S) are reduced, and the microstrains within phase hcp domains increase.

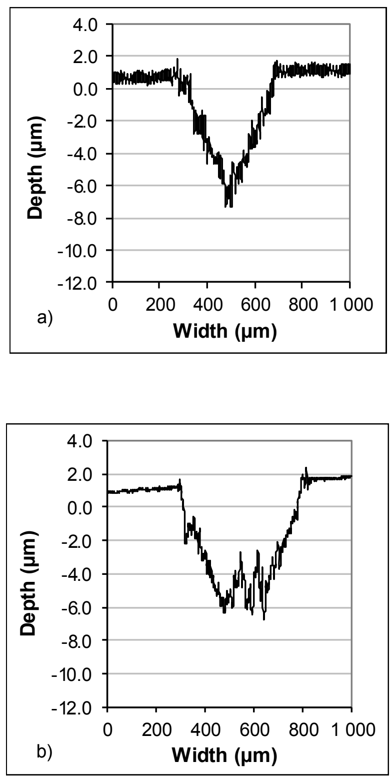

Fig. 4. Profilograms traces worn on irradiated titanium samples a) at a fluence of $2.2{\times}10^{14}$ Xe/cm^2, b) at a fluence of $5{\times}10^{14}$ Xe/cm^2

Table 1. Results of GXRD measurements of titanium samples: a, c – lattice constant, S – crystallite size, ε – microstrains

Parameter	Phase hcp				Phase fcc			
	Unirradiated	1×10^{14} Xe/cm^2	2.2×10^{14} Xe/cm^2	5×10^{14} Xe/cm^2	Unirradiated	1×10^{14} Xe/cm^2	2.2×10^{14} Xe/cm^2	5×10^{14} Xe/cm^2
Content [%]	96.7(3)	89.3(5)	90.5(4)	95.9(5)	3.3(2)	10.7(4)	9.6(1)	4.1(7)
a (nm)	0.29574(2)	0.29518(1)	0.29502(1)	0.29399(1)	0.44132(7)	0.43978(3)	0.43972(3)	0.43827(8)
c (nm)	0.46891(4)	0.46858(2)	0.46843(2)	0.46678(2)				
S (nm)	208.33(2)	193.71(2)	61.57(2)	57.44(2)	2791.3(8)	2471.5(8)	118.1(2)	57.68(2)
ε (%)	0.180(2)	0.241(2)	0.197(2)	0.153(2)	0.292(2)	0.27(1)	0.28(1)	0.181(2)

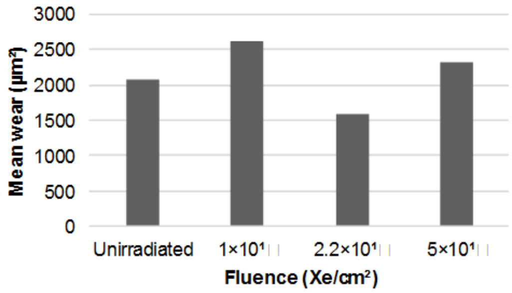

Fig. 5. Wear of samples examined after the 5000-cycle tribological test

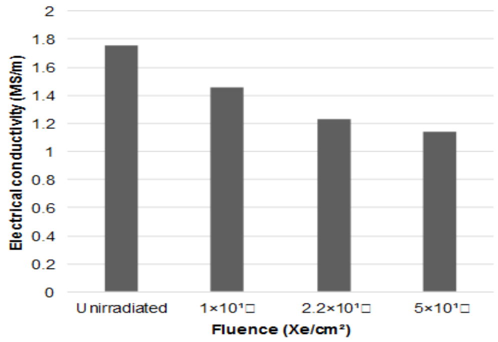

Fig. 6. Results measurements of electrical conductivity of titanium samples

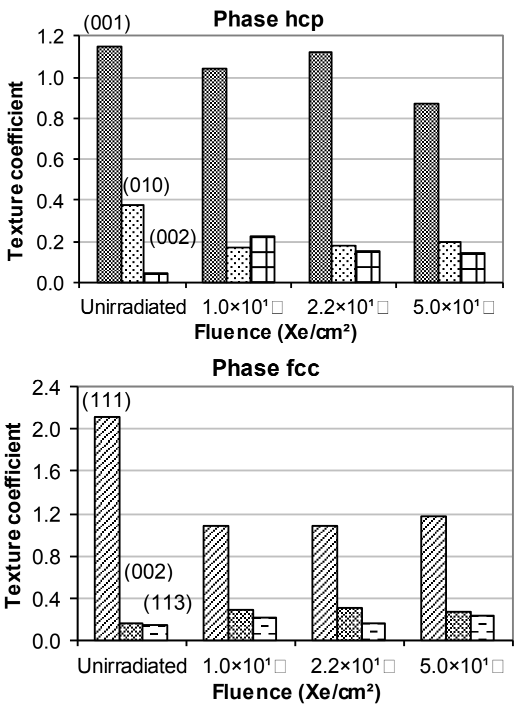

Fig. 7. Texture coefficients of titanium sample

The content of the fcc phase increases and the lattice constant declines. The crystallite sizes and microstrains in the fcc phase are comparable with those in the non-irradiated sample. Irradiation at higher fluences results in further reduction in the parameters of both lattices and crystallite sizes and leads to reduction of microstrains and restoration of the initial contribution of both phases. Irradiation has an effect on the texture of both phases determined with parameter Pk [8] – figure 7.

Conclusion

Titanium irradiation with xenon ions results in changes in the relative content of the hcp and fcc phases, i.e. reduction in the grain size as well as changes in stresses and crystalline lattice texture. Xenon ions produce a substantial number of defects in the crystalline lattice. The reduced grain sizes (increasing the number of grain boundaries) and defects decrease the conductivity of the irradiated titanium layer. It was found that titanium irradiation with xenon ions with energy 168 MeV resulted in formation of two ~2 μm thick layers. Interpretation of the changes in the friction coefficient along with the increase in the trace depth should take into account the decline in the quantity of energy deposited along the ion track, which alters the friction coefficient. The magnitude of wear of both layers (number of cycles required for their wear) depends on the irradiation fluence; however, there is no proportionality between these two parameters.

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REFERENCES

[1] Crespillo M.L., Agullo-Lopez F. Zycchiatti A. Cumulative approaches to track formation under swift heavy ion (SHI) irradiation: Phenomenological correlation with formation energies of Frenkel pairs. *Nucl. Instrum. Methods Phys. Res., Sect. B.* (2017) vol. 394 20-27
[2] Krauser J. Zollondz JH. Weidinger A. et al. Conductivity of nanometer-sized ion tracks in diamond-like carbon films. *J. Appl. Phys.* (2003) vol. 94 1959-1964.
[3] Sadi S., Paulenova A., Loveland W., Watson P.R., Zinkann G., Surface Morphology and Phase Stability of Titanium Foils Irradiated by 136 MeV 136Xe. *Nucl. Instr. Meth. in Phys.Res. B.* (2014) vol. 328 78-83.
[4] Budzyński P., Kamiński M., Surowiec Z., Microstructure and electrical conductivity of titanium irradiated with swift heavy ions. *Przegląd Elektrotechniczny*, (2016) No. 8 155-157.
[5] Ziegler J.F., SRIM-2003, *Nucl. Instrum. Methods Phys. Res., Sect. B.* (2004) vol. 219 1027-1036;
[6] Budzyński P., Tarkowski P. and Pękała P. Influence of nitrogen ion implantation on tribological properties of tool steel NC10, *Vacuum*, (2001) vol. 63 731-737.
[7] Wang Z., Jin Y., M.Hou., G.Jin Modeling of damage creation in metallic materials under swift heavy ion irradiation, *Nucl. Instrum. Methods Phys. Res., Sect. B*, (2000) vol. 169 98-105.
[8] Chawla V., Jayaganthan R., Chandra R., Structural characterizations of magnetron sputtered nanocrystalline TiN thin films, *Mater. Charact.* (2008) vol. 59 1015-1020