Piotr BUDZYŃSKI[1], Mariusz KAMIŃSKI[1], Zbigniew SUROWIEC[2]

Faculty of Mechanical Engineering, Lublin University of Technology (1), Institute of Physics, Maria Curie-Skłodowska University (2)

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Microstructure and electrical conductivity of titanium irradiated with swift heavy ions

Abstract. *Radiation damage caused by heavy ions may induce changes in the surface topography, crystalline structure, and electrical conductivity of irradiated materials. Titanium was irradiated with 130 MeV xenon ions and 260 MeV krypton ions at a fluence of 1, 3, and 5×10^{14} ions/cm^2. The irradiation altered the surface topography of the sample, caused transformation of the α phase into the fcc phase, and reduced electrical conductivity with the increasing irradiation fluence.*

Streszczenie. *Uszkodzenia radiacyjne wywołane ciężkimi jonami mogą powodować zmiany topografii powierzchni oraz zmiany w strukturze krystalicznej i przewodności elektrycznej napromieniowanych materiałów. Tytan napromieniowano jonami Xe o energii 130 MeV i jonami kryptonu o energii 260 MeV. Doza napromieniowanych jonów była równa 1, 3 i 5×10^{14} jonów/cm^2. Napromieniowanie zmienia: topografię powierzchni próbki, powoduje transformacje fazy α w fazę fcc oraz obniża przewodność elektryczną wraz ze wzrostem dozy napromieniowania. (**Mikrostruktura oraz przewodność elektryczna tytanu po napromieniowaniu jonami wysokoenergetycznymi**).*

Keywords: ion implantation, swift heavy ions, microstructure, electrical conductivity.
Słowa kluczowe: implantacja jonowa, jony wysokoenergetyczne, mikrostruktura, przewodność elektryczna.

Introduction

Titanium is commonly used as a construction material in aviation as well as nuclear and thermonuclear reactors. It is necessary to investigate changes in its mechanical and electrical properties caused by heavy ion irradiation with energy of several hundred MeV generated through uranium nuclear fission and by high-energy ions in the upper atmosphere. Titanium has several allotropic phases. It is interesting to examine the impact of a crystalline structure on titanium conductivity in the different allotropic phases. At room temperature and under normal pressure, commercially pure titanium is a mixture of two allotropic phases: α (hexagonal close packed - hcp) and β (body centre cubic – bcc). The proportion of these phases depends on the temperature, sample preparation methods, and possible admixtures stabilising the phases. α is a low-temperature phase and β is a high-temperature phase. Under high pressure exerted on the sample, transformation into the ω phase (simple hexagonal structure) is possible [1,2]. The authors of papers [3-5] demonstrated that the hcp phase can be transformed into the fcc (face centred cubic) phase in thin-layer systems (a few nanometres) and in multilayers. Paper [6] shows that the allotropic hcp-fcc transformation in titanium takes place during the milling process. The transformation is endothermic and begins at a temperature of 321°C. It was interesting to explore the changes induced by irradiation of titanium with a swift ion beam in a range of 10÷20 μm. Such transformation was observed after irradiation with a 136 MeV xenon ion beam [7]. The authors of paper [1] reported transformation of the hcp phase into the ω phase after irradiation with a 2.2 GeV uranium ion beam at a temperature of 20K. The changes in the sample surface topography, crystalline structure, and electrical properties of titanium occurring after irradiation with swift krypton and xenon ions are interesting to explore. Changes in the mechanical properties of titanium were analysed in paper [8].

Experimental studies

The irradiation with 260 MeV heavy krypton ions and 130 MeV heavy xenon ions was performed with the cyclotron U-400 at the LRJ ZIBJ (Laboratory of Nuclear Reactions, Joint Institute of Nuclear Research) in Dubna. The fluences applied in both cases were 1, 3, and 5×10^{14} ions/cm^2. To ensure better than 5% uniformity of the implanted fluence, the ion beam was swept over the sample horizontally and vertically. The intensity of the ion beam was limited to $(2\div8)\times10^{12}$ ions/(m^{2}s) in order to obtain beam power in the range of (80÷310) W/m^2. Moreover, the sample was placed in an aluminium holder partly located outside a vacuum chamber cooled with an air stream. In these conditions, the temperature of the probe did not exceed 50°C. The surface topography was analysed under an atomic force microscope (AFM) Nanosurf Easy Scan2. The results were analysed using the program [9]. X-ray and GXRD diffractograms were measured with CuK_α (α = 0.1542 nm) radiation. Measurements of electrical conductivity were performed using a Sigmatest 2.069 meter calibrated with a 4.4 MS/m conductivity standard.

Fig.1a shows the surface of titanium after irradiation with xenon ions. There is a visible track of the beam along the diagonal of the photo. Along the track, hillocks with a ~10 nm height and a ~100 nm diameter are formed. Irradiation also leads to formation of voids/craters, which are clearly discernible in Fig.1a. After irradiation at a fluence of 5×10^{15} Xe/cm^2, almost the entire surface layer of the sample was locally melted – Fig.1b.

The surface of titanium irradiated with krypton ions at a fluence of 1×10^{15} Kr/cm^2 is presented in Fig.1c. The hillocks have a 20÷60 nm height and a 160÷380 nm diameter. The number of hillocks increases proportionally to the fluence, while their size remains constant.

The differences in the changes in the krypton- and xenon-implanted titanium surface are related to magnitude of energy loss of swift ions moving in the titanium surface layer and the ion range. The energy loss of krypton and xenon ions amounts to 15 and 22 keV/nm, respectively, and the most probable ion ranges are 19.5 and 10.2 μm, respectively.

Diffractograms of the titanium surface layer are presented in Fig.2. The surface layer is composed of the α phase with a ca. 8% admixture of the β phase. After irradiation, peaks originating from the reflections of the α phase, i.e. (002), (102), (110), (200), (112), and (004) disappear. Peaks (220) and (202) characteristic of the fcc phase appear. The high-intensity peak of the fcc phase (111) occupies the position of the reflection peak (110) of the α phase. The fcc phase peaks (200) and (311) are located in the position occupied by the α phase peaks (101) and (103) before the irradiation. The total change in the appearance of the spectrum and the emergence of peaks characteristic of the fcc phase imply transformation of the α

phase into the fcc phase after irradiation with the xenon ion beam. Transformation occurs at the fluence of $1 \div 5 \times 10^{14}$ ions/cm^2 and is enhanced along the increasing fluence. After irradiation with 5×10^{14} Xe/cm^2, the entire α phase is transformed into the fcc phase.

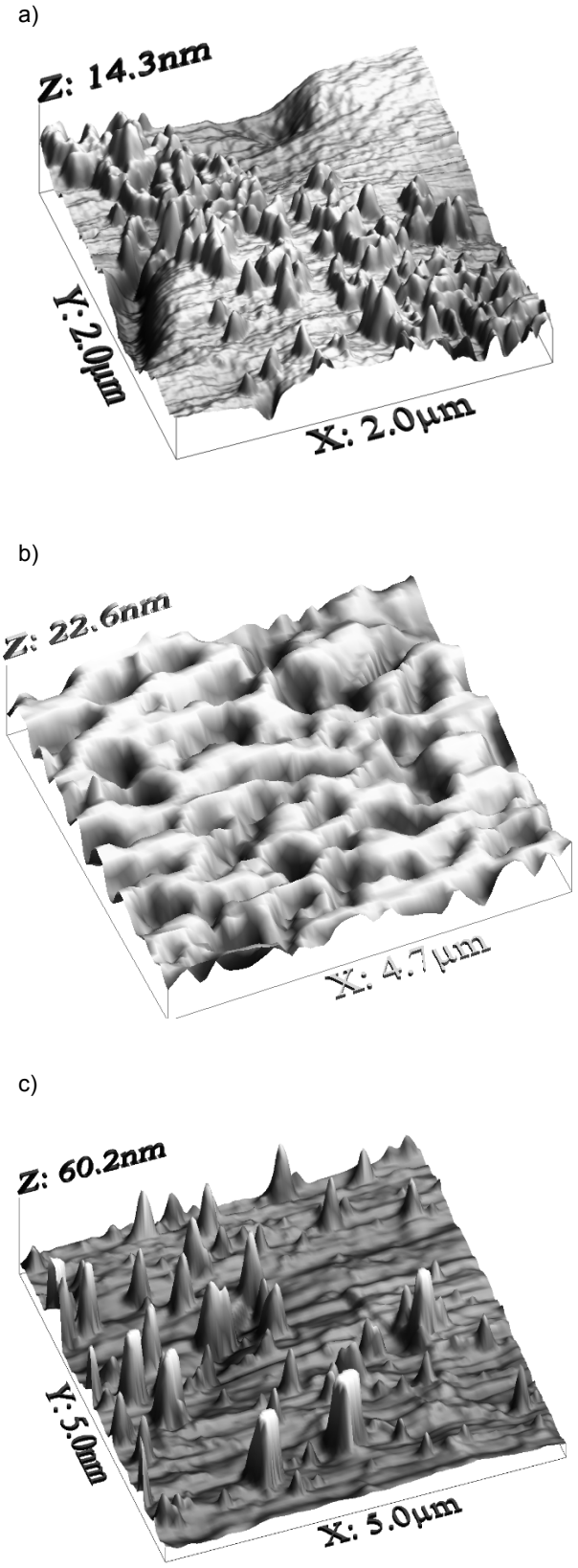

Fig.1. Titanium surface irradiated with 130 MeV xenon ions at a fluence of *a*) 1×10^{15} Xe/cm^2, *b*) 5×10^{15} Xe/cm^2, *c*) 260 MeV krypton ions at a fluence of 1×10^{15} Kr/cm^2

Before irradiation, the sample was characterised by 1.784 MS/m conductivity, which declined after irradiation – Fig.3. Slightly greater changes were induced by irradiation with the xenon rather than krypton beam.

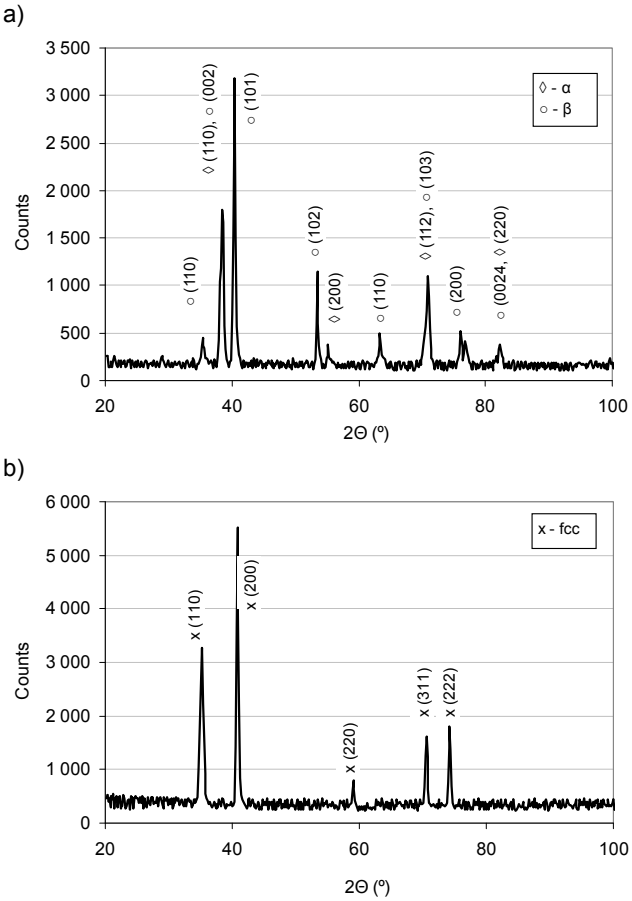

Fig.2. X-ray diffractograms for titanium; *a*) before irradiation, *b*) after irradiation with a xenon ion beam at a fluence of 5×10^{14} ions/cm^2

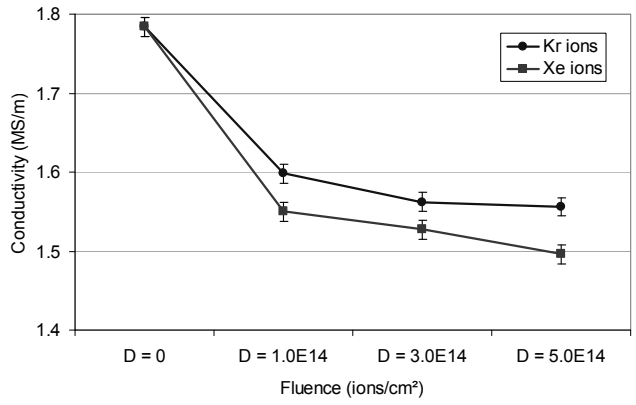

Fig. 3. Changes in titanium conductivity after irradiation with swift xenon and krypton ions

Conclusion

Irradiation of titanium with swift xenon and krypton ions leads to formation of hillocks. They are the dominant structure emerging after irradiation with krypton ions. Additionally, irradiation with xenon ions produces voids/craters. Irradiation of the titanium surface at a fluence of 5×10^{14} Xe/cm^2 causes its local melting. Swift ions induce transformation of the α phase into the fcc phase.

The change in the crystalline structure and the non-uniformity associated with the hillocks as well as voids and craters cause about 15% reduction of the conductivity of the modified surface layer of titanium. The transformation of the Ti α phase (hcp) into the fcc phase may have practical importance, as the latter phase is usually more ductile than the former, which is essential in the case of titanium.

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Authors: *dr hab. inż. Piotr Budzyński, Lublin University of Technology, Faculty of Mechanical Engineering, 36, Nadbystrzycka Str., 20-618 Lublin, Poland, E-mail: p.budzynski@pollub.pl; mgr inż. Mariusz Kamiński, Lublin University of Technology, Faculty of Mechanical Engineering, 36, Nadbystrzycka Str., 20-618 Lublin, Poland, E-mail: mariusz.kaminski@pollub.pl; dr Zbigniew Surowiec, Institute of Physics Maria Curie-Skłodowska University, 1, Maria Curie-Skłodowska Sk., 20 031 Lublin, Poland, zbigniew.surowiec@umcs.lublin.pl.*

REFERENCES

[1] Dammak H., Dunlop H.A., Lesueur D., Study of the irradiation-induced α→ω phase transformation in titanium: kinetics and mechanism, Philosophical Magazine A, 79 (1999) 147-166.

[2] Lutjering G., William J.C., Titanium, Springer, New York, (2007), 13-50.

[3] Tepper T., Shechtman D, van Heerden D., Josell D., fcc titanium in titanium/silver multilayers, Materials Letters, 33 (1997), 181-184.

[4] Banerjee R., Dregia S.A., Fraser H.L., Stability of f.c.c. titanium in titanium/aluminum multilayers, Acta Materialia, 47 (1999), 4225-4231.

[5] Kado T., Structure of Ti films deposited on MgO(001) substrates, Surface Science, 454-456 (2000), 454-456.

[6] Zhang D.L., Ying D.Y., Formation of fcc titanium during heating high-energy, ball-milled Al–Ti powders, Materials Letters, 50 (2001), 149-153.

[7] Sadi S., Paulenova A., Loveland W., Watson P.R., Greene J.P., Zhu S., Zinkann G., Surface morphology and phase stability of titanium foils irradiated with 136 MeV 136Xe, Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 328 (2014), 78-83.

[8] Budzynski P., Skuratov V.A., Kochański T., Surowiec Z., Titanium surface layers after irradiation with swift Kr and Xe ions, Vacuum, 83 (2009), 190.

[9] Horcas I., Fernandez R., Gomez-Rodriguez J.M., Colchero J., Gomez-Herrero J., Baro A.M., WSXM: A software for scanning probe microscopy and a tool for nanotechnology, Review of Scientific Instruments, 78 (2007), 013705.