Deformation characteristics of the near-surface layers of zirconia ceramics implanted with aluminum ions

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Abstract. The effect of ion treatment on the phase composition and mechanical properties of the near-surface layers of zirconium ceramic composition 97 ZrO_2-3Y_2O_3 (mol%) was studied. Irradiation of the samples was carried out by accelerated ions of aluminum with using vacuum-arc source Mevva 5-Ru. Ion beam had the following parameters: the energy of the accelerated ions E = 78 keV, the pulse current density J_i = 4mA / cm^2, current pulse duration equal τ= 250 mcs, pulse repetition frequency f = 5 Hz. Exposure doses (fluence) were 10^{16} и 10^{17} ion/cm^2. The depth distribution implanted ions was studied by SIMS method. It is shown that the maximum projected range of the implanted ions is equal to 250 nm. Near-surface layers were investigated by X-ray diffraction (XRD) at fixed glancing incidence angle. It is shown that implantation of aluminum ions into the ceramics does not lead to a change in the phase composition of the near-surface layer. The influence of implanted ions on mechanical properties of ceramic near-surface layers was studied by the method of dynamic nanoindentation using small loads on the indenter P=300 mN. It is shown that in ion-implanted ceramic layer the processes of material recovery in the deformed region in the unloading mode proceeds with higher efficiency as compared with the initial material state. The deformation characteristics of samples before and after ion treatment have been determined from interpretation of the resulting P–h curves within the loading and unloading sections by the technique proposed by Oliver and Pharr. It was found that implantation of aluminum ions in the near-surface layer of zirconia ceramics increases nanohardness and reduces the Young's modulus.

1. Introduction

It is well known that the surface layer has a great influence on the physical and mechanical properties of solids. Surface modification of materials appears as the power and widely methodology, as it allows combining different properties of material bulk and material surface, hence designing of new materials characterized by unique properties. One of the most promising modern directions in modification of near-surface properties of materials is the treatment of accelerated ion beams.

Modification of the near-surface region of materials by use of energetic ion beams has been investigated extensively in recent years [1-6]. The energetic ion comes to rest by displacing atoms from their normal lattice sites by atom collisions thus producing a large number of radiation defects of different nature and complexity. The ion implantation allows one to introduce any element into the near-surface region of solids in a controlled and reproducible manner that is independent of most equilibrium constraints.

This may change not only the elemental composition of the surface, but as result atom rearrangement and accumulation of implanted ions new chemical compounds may be formed.

As a result structural-phase state of the material surface layers changes dramatically. Both radiation damage and implanted species themselves can introduce very large compressive stresses into near – surface layer and these can have marked effects on deformation of the surface layers, as well as the conditions of nucleation and propagation of cracks therein. All previously mentioned structure changes induced by the ion bombarding of materials must to lead to modification of near-surface layers properties.

Ion radiation treatment has been widely used to modify the mechanical properties of the surface of metals and alloys [1-3]. Recently new areas of application of ion implantation emerge, among them implantation into various types of ceramics [3-6]. Zirconium oxide is the most widely used oxide ceramic for functional applications and in particular as structural ceramics in mechanical engineering. The full exploitation of this material requires a fine modification of its properties. The use of ion beams represents one effective approach to achieve this goal. The interaction processes of ion beam with ceramics yet are understood not well. They are different from those observed in metals and alloys. Alterations to the structure and properties of ceramics are complex due to the range of bonding types encountered and the necessity for maintaining local charge balance.

As rule the state of implanted ions, their the reaction with host atoms, the phase evolution and the properties of implanted ceramics cannot be predicted accurately. Structural-phase state and properties of the modified surface layer ceramics depend on the conditions of ion treatment (nature of the implanted ions, ion beam power, radiation dose). By varying of these parameters we can form near-surface layers in ceramics with the most diverse properties. Our studies have shown [7] that processing of zirconium ceramics by powerful pulsed beam of accelerated carbon ions causes a decrease in hardness and increases plasticity of its of surface layers.

In the present work the effect of irradiation with accelerated aluminum ions on mechanical properties of the surface layers of zirconium ceramics is studied.

2. Experimental technique

The object of this study was (mol %) 97ZrO2-3Y2O3 zirconia ceramics sintered from ultrafine powders synthesized by plasma-chemical method. Sintering was conducted in air in a resistance furnace at T = 1400 ° C for 3 hours. The samples were made in the form of pellets with the diameter of 10 mm and thickness of 3 mm. Their density was equal to ρ= 5.7 g/cm^3, and the porosity was 6.5%. After sintering the samples were polished and then subjected to normalizing annealing at T = 1000 ° C for one hour. Aluminum ions were implanted into the ceramics. For generation of metal ion beam we used ion source Mevva-5 Ru based on vacuum arc discharge [8]. Ion beam had the following parameters: the energy of the accelerated ions E = 78 keV, the pulse current density J_i = 4mA / cm^2, current pulse duration equal τ= 250 mcs, pulse repetition frequency f = 5 Hz. Fluences (f) of ion irradiation were equal to 10^{16} and 10^{17} ion / cm^2. Samples were analyzed using technique of glancing incidence X-ray diffraction (GIXRD). The GIXRD was performed using Cu K_α X-rays. X-ray diffractometer ARL X’tra was used in the experiments. The elemental composition of the implanted layers was studied by SIMS with mass spectrometer PHI6300.

The study of mechanical properties of thin modified near-surface layers of specimens was performed by the method of nanoindentation using small loads on the indenter. It consisted in a continuous hardness indentation, with the load on the indenter varying linearly over time. This was also accompanied by the measurement of indentation depth (*h*), and load (*P*). The method makes it possible to measure in one cycle the depth of nonrecovered h_m and recovered (plastic) h_0 indents, Young’s modulus *E* (from the tilt of the linear section of the unloading curve), and material hardness *H*. By applying this method we can follow the deformation process dynamics in a material microvolume and obtain more complete information on its mechanical characteristics. To this end, in this work we used a CSEM Nano Hardness Tester. Indentation tests were performed using a Vickers pyramid. About 8 – 10 indents were applied to the specimen surface.

The spacing between the dips of the indenter exceeded 20 μm. The indenter load P was 300 mN. The statistical error was within 6 – 10 %.

3.Experimental results

TRIM code and method SIMS are used to estimate the depth profile of the implanted ions in ceramics. The results are shown in Figure 1.

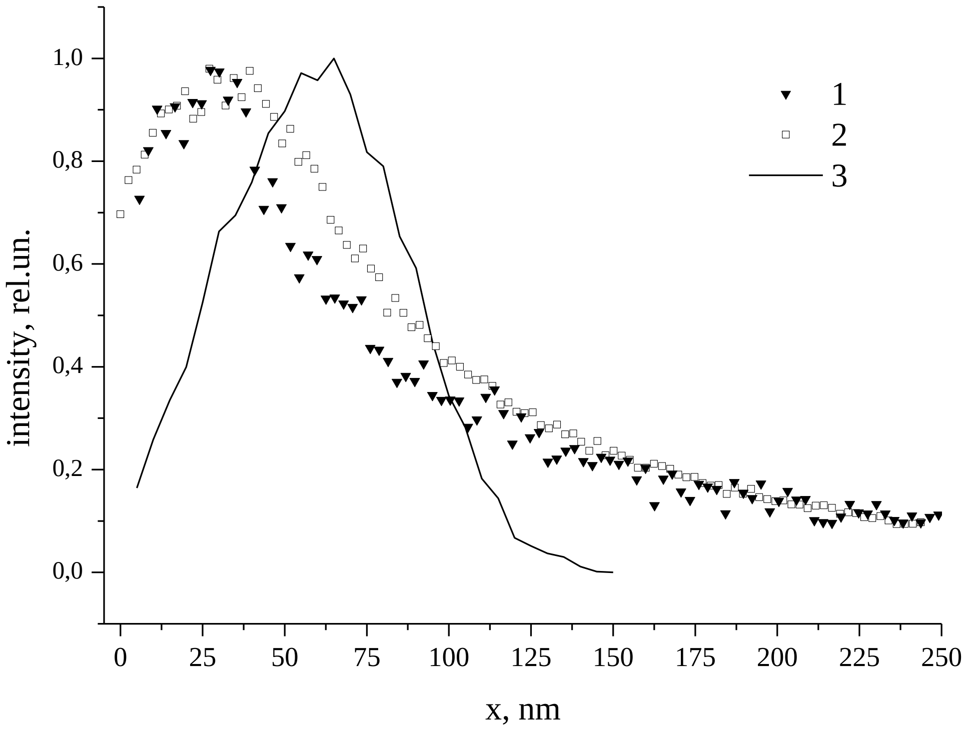

Figure1. Depth profiles of aluminum ions implanted in zirconium ceramics (Curves 1, 2 is experiment, Curve 3 is calculation. 1 – f=10^{16} ion / cm^2, 2- 10^{17}ion / cm^2).

It is seen that maximum of the experimental depth profiles (curves 1 and 2) is shifted towards the surface relative to the curve 3 calculated by TRIM code. Such displacement may be caused by sputtering of ceramic surface under ion bombardment. This virtually shifts implantation profile towards the surface. The research allowed us to determine the thickness of the ceramics implanted layer, which was τ 250 nm.

Effect of ion treatment on the phase composition of the implanted layer ceramics was investigated by X-ray diffraction. Taking x-ray patterns using the geometry of the Bragg-Bretano is incorrect, as according to numerical calculations probing depth is about 6.5 microns. This value is significantly greater than the thickness of the implanted layer ceramics, which, as seen in Figure 1, is about 250 nm. That is why the samples were analyzed using technique of GIXRD. For the case when technique of GIXRD is used, thickness of ceramic layer, which contributes in x-ray diffraction using technique of GIXRD was calculated by the method described in [9]. Calculated values of thickness of layer for different τ are the next: 820 nm for β=1° 2330 nm for β= 3°

It was found that the X-ray diffraction patterns obtained by technique of GIXRD for the unirradiated sample and the samples irradiated by the ion beam with f=10^{16} ion / cm^2 and 10^{17} ion/cm^2 were identical. We can observe diffraction reflections which belong to the tetragonal phase of zirconia dioxide only. Therefore, these modes of ion implantation do not cause any noticeable change in the phase composition of the treated surface.

Figure 2 a, b shows a typical P–h dependences obtained during indentation of ceramic specimen before (Figure. 2 a) and after ion irradiation with f =10^{17}ion/cm^2 (figure. 2 b).

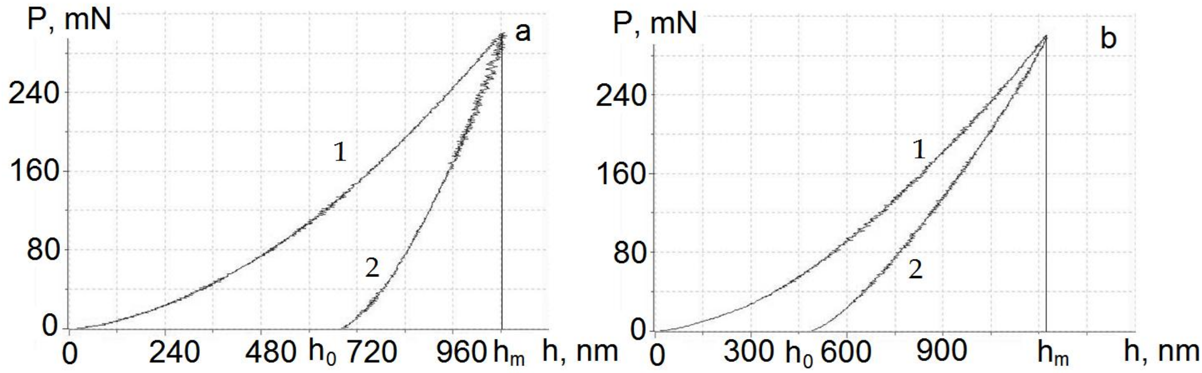

Figure 2. Dependences P(h) of zirconia ceramics: a) non- irradiated , b) irradiated with f =10^{17} ion/cm^2.

The curve consists of two sections. Section 1 corresponds to the process of indenter dipping into the specimen and characterizes material resistance to plastic deformation. Section 2 provides information on deformation behavior of the deformed region in the unloading mode. It is evident that the residual indentation depth h_0 after removal of the indenter is significantly different from its maximum indentation depth h_m. This fact is indicative of an active character of the processes of material recovery in the deformed region in the unloading mode. Their contribution into the changes in indentation size can be estimated using parameter α=h_m - h_0 / h_m.

The deformation characteristics were determined from interpretation of the resulting P–h curves within the loading and unloading sections by the technique proposed by Oliver and Pharr [10]. Deformation characteristics of the near-surface layers of zirconia ceramics before and after ion treatment are shown in Table 1.

Table 1 lists the deformation characteristics of the near-surface layers of zirconia ceramics before and after ion treatment.

Table 1. Deformation characteristics of the near-surface layers of zirconia ceramics before and after ion treatment.

	Unirradiated ceramics	Irradiated ceramics	
		f=10^{16} ion/cm^2.	f=10^{17}ion/cm^2.
H, GPa	12.2	15.5	15.2
E, GPa	214	106	97
α	0.36	0.58	0.61
H/E	0.057	0.146	0.156
H^3/E^2, GPa	0.04	0.33	0.37

Analysis of the P(h) curves on Figure 2, and results shown in Table 1, showed that ion treatment significantly stimulates proceeding of process of material recovery in the deformed region in the unloading mode. Table 1 shows that the value α for the irradiated sample almost two times higher than for unirradiated sample. In our opinion this is due to the mechanical stresses that occur in the near-surface layer during implantation of accelerated ions.

Studies have shown that implantation of aluminum ions in the near-surface layer of zirconia ceramics increases nanohardness and reduces the Young's modulus. To evaluate the resistance to elastic deformation of the material and its ability to withstand mechanical stresses without forming is often used value H / E [11].

At the same time the material's resistance to plastic deformation characterizes parameter H^3/E^2 [11]. From the data given in table1 seen that those parameters after ionic treatment of ceramics are substantially increased.

4. Conclusions

The study found:

1. Treatment of zirconia ceramics with accelerated aluminum ions does not change the phase composition of ceramic near-surface layers.
2. Implantation of aluminum ions in the zirconia ceramics significantly stimulates proceeding of process of material recovery in the deformed region in the unloading mode.
3. Implantation of aluminum ions in the near-surface layer of zirconia ceramics increases nanohardness and reduces the Young's modulus.

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