

## Short communication

## The surface layers structure of differently oriented single titanium nickelide crystals subjected to ion implantation

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## ABSTRACT

The structure of the surface and near-surface layers of titanium nickelide (NiTi) single crystals having  $[111]_{B2}$  and  $[001]_{B2}$  orientation relative to the direction of Si-ion beam treatment was investigated. It was found that the orientation effects of selective sputtering and the channeling control the thickness of the oxide and amorphous layers, the depth of penetration of ions and impurities, the distribution of Ni with depth. Different scenarios of deformation microstructure evolution are observed in the near-surface layers of NiTi crystals having of 'soft' orientation  $[111]_{B2}$  and 'rigid' orientation  $[001]_{B2}$ .

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Titanium nickelide and NiTi base alloys are known to possess unique properties, such as shape memory and superelasticity [1,2]. These materials have found wide industrial and medical applications. A significant problem is the creation of special surface protective layers, which can improve performance characteristics and lead to minimize or completely prevent the release of toxic Ni [3,4]. The ion implantation treatment holds much promise for modifying the surface layers of NiTi [3–5]. The effect of ion beam treatment on the NiTi shape memory of the substrate is the subject of extensive studies; in-depth studies were also made of as-treated surface layers and their service properties [E.g., 2–4]. But not fully clear is the nature of the processes taking place in the surface structure during the NiTi ion-plasma treatment. The main difficulty for researchers is certainly a diversity of factors and phenomena, both occurring in the material during exposure to the ions. Recent efforts have drawn attention to the effect of crystallographic anisotropy of polycrystalline NiTi alloys [7–9]. The elastic-plastic anisotropy in NiTi can lead to strong localization of deformation followed by destruction. In particular the problem of elastic-plastic anisotropy of NiTi arises at operation of small systems, medical devices, i.e. wires, bands, thin rods; thin-wall tubes, etc. [8,9].

The effect of crystallographic orientation on the structure and properties of the surface modified layers of NiTi samples has been studied insufficiently thus far. It should also be noted the orientation effects of selective sputtering and the ion channeling which arise upon exposure of ions and can affect the crystal structure of the surface layers [5,10]. The purpose of this paper is to identify the role of the crystallographic orientation in formation of the complex structure of the ion-modified layers of the NiTi through experiments on single crystals of different orientations, subjected to the high dose Si ion implantation treatment.

The tests were performed for the single NiTi crystals with 50.6% at. Ni using samples having either 'soft' or 'rigid' orientations, which are oriented, respectively, near the poles  $\langle 111 \rangle_{B2}$  and  $\langle 001 \rangle_{B2}$  with respect to the direction of the incident ion beam, which was coincident with the normal to the sample surface. The Si ion implantation treatment was performed using an ionic implanter unit 'DIANA-3' (ISPMS SB RAS, Russia) with pulsed Si ion beams to a total fluency of  $D = 6 \times 10^{17} \text{ cm}^{-2}$  at an average accelerating voltage of 60 kV and a pulse repetition frequency of 50 Hz. The implanter chamber was pumped out to the residual pressure  $3 \times 10^{-3} \text{ Pa}$ . The sample temperature during ion implantation did not exceed 473 K.

A layer-by-layer element analysis was made for the modified sample surface with the aid of Auger electron spectroscopy, using a spectrometer unit 'Shkhuna-2' (NITPU, Russia). The target material

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was sputtered at the rate 2–3 nm/min, using argon ion beam having 1-mm diameter and ion energy 3 KeV. The structure and phase composition of the surface layer was examined before and after the treatment, using the TEM technique on a microscope JEM 2100 (JEOL, Japan). Thin foils having cross-section geometry were prepared by ion thinning on the equipment EM 09100IS (JEOL, Japan).

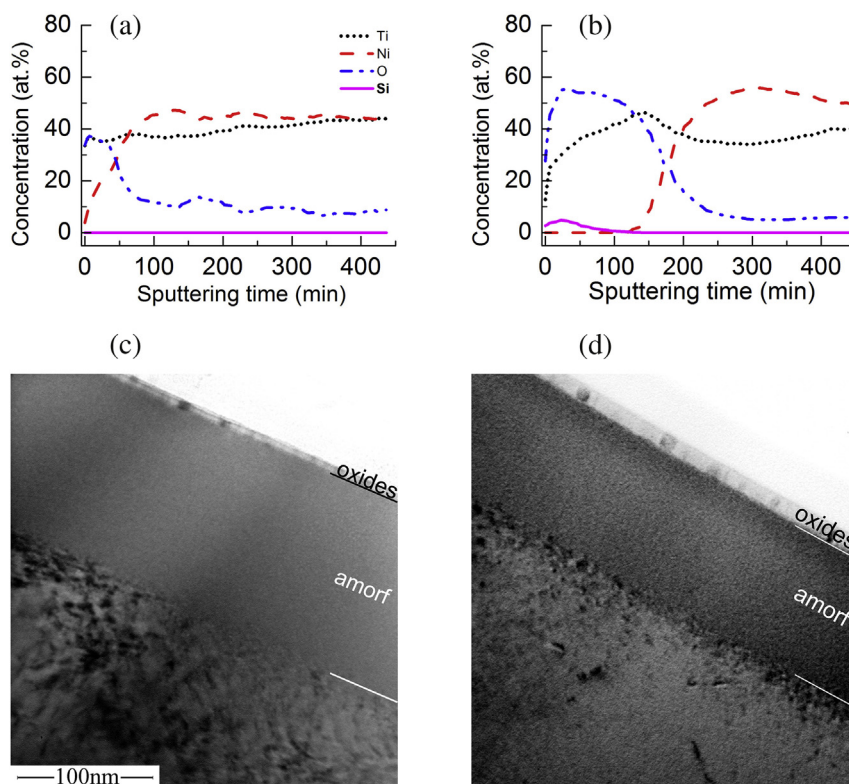
The ion-modified layer was found to have a top oxide layer 10–20 nm thick and a lower-lying amorphous layer (Fig. 1). The upper oxide sublayer of the modified surface is nanocomposite ceramic containing TiO<sub>2</sub> and SiO<sub>2</sub> oxides having size of about 10 nm, which stand in different quantitative proportions to one another, depending on the Si ion distribution with depth. In the case of NiTi sample [111]<sub>B2</sub> the maximal Si content to amount to 15 at.% was found to occur in the same layer enriched in oxygen (Fig. 1b). Thus the presence of Si in the surface layer of the sample [001]<sub>B2</sub> was indicated by the occurrence of bends on the Auger spectra only (Fig. 1a). We suggest that the Si atoms are uniformly distributed in the surface layer in a depth range up to 180 nm or even deeper. Apparently the formation of the SiO<sub>2</sub> nanocrystals in the oxide layer of [111]<sub>B2</sub> is more intense than on the surface of a single crystal oriented in the direction [001]<sub>B2</sub>. However, no statistically reliable quantitative data on the volume fractions of oxides has been obtained thus far.

The diffraction patterns taken from selected area of diameter 190 nm (SAED) suggest that TiO<sub>2</sub> occurs among modifications of brookite and rutile and SiO<sub>2</sub>, among modifications of  $\alpha$ -quartz and low-tridymite (Fig. 2). Due to their low solubility, Ti and Si oxides occur as individual phases. The nanobeam diffraction (NBD) pattern was obtained for oxides (Fig. 2b) using the probe diameter of about 20 nm. Fig. 2b shows the sections of the reciprocal lattice of two neighboring particles of SiO<sub>2</sub> (low-tridymite) having lattice parameters  $a = 18.524 \text{ \AA}$ ;  $b = 5.003 \text{ \AA}$ ;  $c = 23.810 \text{ \AA}$  and  $\beta = 105.82^\circ$

disoriented relative to one another.

The important parameter characterizing the state of the NiTi surface layers is the content of Ni atoms. In the case of NiTi crystals [111]<sub>B2</sub>, the Ni concentration of the oxide layer increases sharply from zero up to  $\approx 56\%$  at (Fig. 1c). At the same time [001]<sub>B2</sub> the Ni content on the upper/lower-lying oxide layer interface will change gradually from  $\approx 5\%$  at. to  $\approx 40\%$  at. (Fig. 1a), respectively, which might be due to an insignificant loss of Ni. As a whole the depth of depletion of the Ni in the implantation of the sample [001]<sub>B2</sub> is almost two times smaller than for the [111]<sub>B2</sub>. It is known that a significant loss of Ni atoms might be due to the preferential sputtering of Ni atoms: the sputtering yield of Ni is 3–4 times higher than that of Ti for a wide range of bombardment energies [6]. Another reason might be the fact that the Ti–O (Si–O) bond is stronger than the Ni–O bond, which leads to diffusion of Ni atoms from the sample surface towards the lower-lying layers [3]. Taking into account the identical conditions of ion bombardment of NiTi samples, the observed effects can be explained as being a result of the additional influence of the crystal anisotropy on the sputtering yield. The minimal sputter yield is observed for the ion incidence that is in line with the maximal lattice transparency, which enables ion penetration into along the directions parallel to the axes or planes of the target having low crystallographic indices ([001]<sub>B2</sub>). A lower sputtering yield of Ni in this case results in a lower oxide layer thickness and higher concentration of Ni retained at the sample surface.

The ion-modified surface layer was characterized by a diffuse halo (Figs. 2a and 3a), which corresponds to the reflection of a B2 amorphous structure in the plane {110}, which exhibits a short-range order. It is believed that distortion-type amorphization takes place, causing thereby distortion of the original NiTi crystal on a scale level of the unit cell [11]. The crystallographic anisotropy



**Fig. 1.** The microstructure of the surface layers of NiTi single crystals: (a) and (b) – auger depth profiles of chemical components for [001]<sub>B2</sub> and of [111]<sub>B2</sub> samples implanted layers respectively; (c) and (d) – cross-sectional TEM bright-field images of the surface layers of [001]<sub>B2</sub> and [111]<sub>B2</sub> samples respectively.

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