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WEAR RESISTANCE AND RADIATION TOLERANCE OF NANOSTRUCTURED TiAlN COATINGS

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The main goal of this research was to establish the influence of different deposition regimes on tribological properties of TiAlN coatings, as well as the effect of He^+ ion irradiation on hardness and wear resistance of these coatings. Investigations were conducted on TiAlN coatings fabricated by reactive magnetron sputtering. The irradiation of TiAlN coatings was performed at the ion accelerator «High Voltage Engineering Europa B.V.» using He^+ ions with an energy of 500 keV and fluences of $5x10^{16}$ – $3x10^{17}$ ions/sm^2. Elemental composition was examined by the Rutherford backscattering method (RBS) with 1.5 MeV helium ions and a resolution of the detector of 15 keV. Structural properties have been studied by X-ray diffraction method (XRD) and scanning electron microscopy (SEM). Tribological tests were also conducted. It was shown, that nanostructured TiAlN coatings are radiation tolerant and are perspective as fuel claddings in nuclear reactors.

Keywords: nanostructured TiAlN coatings, reactive magnetron deposition, wear resistance, radiation tolerance.

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INTRODUCTION

Hard and super-hard coatings have a lot of applications in modern physics and technique [1]. It is well known, that nitride coatings perform good tribological properties. One of the most promising applications of these coatings is tribological sphere. TiAlN coatings are widely spread in many different machine building applications as they are hard, resistant to corrosion and relatively easy in production. There are a lot of methods of vacuum coating depositions: vacuum arc deposition, chemical vapor deposition, plating and others. The reactive magnetron sputtering is specific way of vacuum coating formation. This method of reactive magnetron sputtering gives an opportunity to obtain coatings with a very dense structure and without a drop fraction [2]. It allows obtaining very hard, non-defective nanocrystalline thin solid coatings.

In some papers, it was shown that nanocrystalline coatings might perform perfect radiation tolerance [3]. Due to this interest it is very topical to investigate the influence of He^+ ion irradiation on wear properties of TiAlN coatings, as it is important for nuclear reactors applications.

EXPERIMENTAL PROCEDURE

Investigations were conducted on the TiAlN coatings prepared by reactive magnetron sputtering using a URM 327 vacuum setup equipped with a system for the automatic control of the supply of argon and nitrogen based on a S100 portable spectrometer. Gas pressure of $Ar+N_2$ was fixed at 0.7 Pa. During deposition of the TiAlN coatings, the substrate temperature was kept constant at 250 °C. Other conditions such as supply voltage, discharge current and bias on the substrate were fixed to 300-320 V, 1.4-1.75 A, -90 V, respectively. The deposition rate was ~1.5 nm/s. The thickness of the TiAlN coatings varied in the range of 0.5-2.5 μm. The deposition of the TiAlN coatings was carried out using three following regimes: with a deficiency ($TiAlN_{1–x}$), at a stoichiometric concentration (TiAlN), and with an excess ($TiAlN_{1+x}$) of the reactive gas N_2.

Elemental composition was examined by the Rutherford backscattering method (RBS) with 1.5 MeV helium ions and a resolution of the detector of 15 keV. Structural properties have been studied by X-ray diffraction method (XRD) using a DRON-3 diffractometer operating in Bragg-Brentano configuration using 1.79021 Å wavelength and scanning electron microscopy (SEM) using electron microscope Hitachi SU3400. Tribological tests were conducted using the tribometer ATVP equipment.

Irradiation of the TiAlN coatings was performed at the ion accelerator «High Voltage Engineering Europa B.V.» using He^+ ions with energy of 500 keV and fluences in the range of $5x10^{16}$ to $3x10^{17}$ ions/sm^2. The coatings were annealed at temperature of 773 K during 15 min after the He^+ ion irradiation to simulate the usage of TiAlN coatings in nuclear reactor.

RESULTS AND DISCUSSION

Figure 1 shows the RBS spectra of TiAlN coating after deposition. Curves 1, 2, and 3 in Fig. 2 correspond to the following conditions of the coating deposition: (1) deposition with a deficiency of nitrogen; (2) deposition at the stoichiometric concentration of nitrogen; and (3) deposition with an excess of nitrogen.

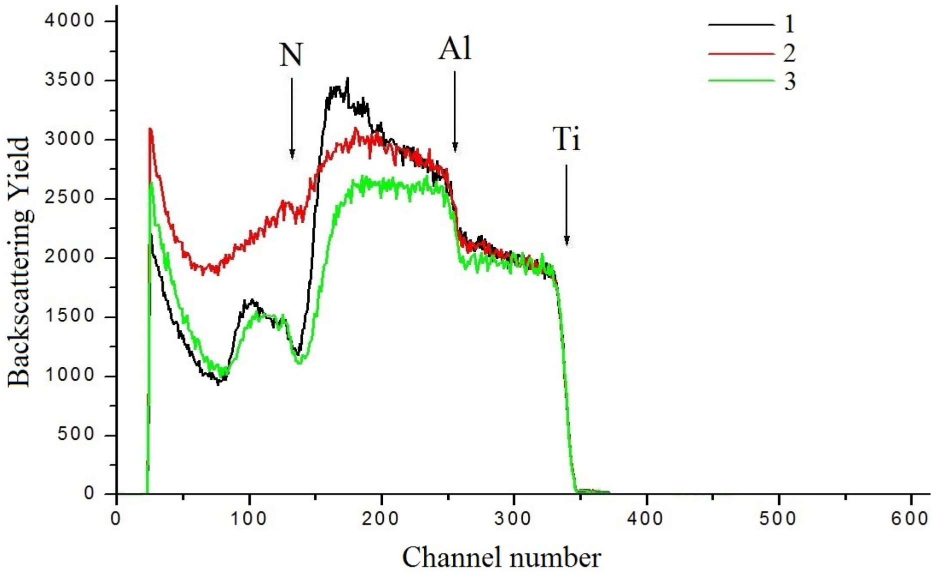

Fig. 1. – RBS spectra of TiAlN coatings 1 – deposition with a deficiency of nitrogen; 2 – deposition at the stoichiometric concentration of nitrogen; and 3 – deposition with an excess of nitrogen.

Titanium and aluminum present in the TiAlN coatings in approximately equal concentrations (0.5 ± 0.02), but their concentrations are not homogeneous in the depth of the coating in the case of deposition with a deficiency of nitrogen and at the stoichiometric concentration of nitrogen (Fig. 1, curves 1, 2). In the case of deposition with an excess of nitrogen, the concentrations of Ti and Al are homogeneous (Fig. 1, curve 3). The concentration of N is not homogeneous in all cases of coating deposition. There is a strong gradient in element concentrations in TiAlN coating, as it can be seen from RBS spectra (Fig. 1), which indicates that coatings deposition process is highly nonequilibrium.

Figure 2 shows XRD spectra of deposited and irradiated TiAlN coatings. There are observed reflexes from λ-Fe (steel substrate) and from complex nitride (Ti, Al)N, which is a substitutional solid solution. The lattice parameter of (Ti, Al)N is 4.226 Å. It was observed, that there is no changes in structure, phase composition and lattice parameter of TiAlN coating after He^+ irradiation. The estimated average crystalline size of the TiAlN coating is 10-15 nm. After the irradiation, the crystalline fragmentation takes place, the average crystalline size decreases to 9-12 nm. Also, there is no spinodal phase segregation in the TiAlN coating after ion irradiation, there is still only one phase of substitutional solid solution (Ti, Al)N. According to our investigations of irradiated TiAlN coatings by optical and electron microscopies, there are no blistering, that is very exiting result, since bulk materials, such as stainless steels (AISI 304, etc.) show great blistering, sometimes even at the irradiation fluence less than 10^{16} ions/cm^2.

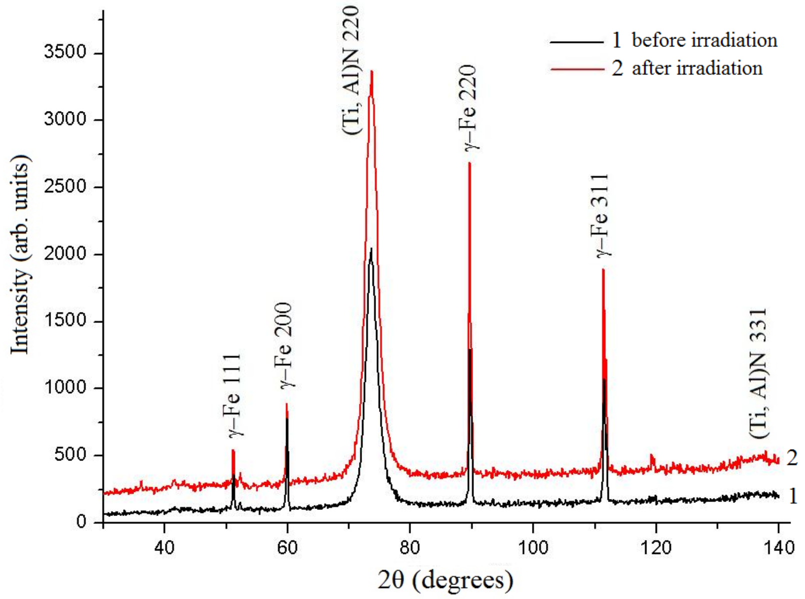

Fig. 2. – XRD spectra of as-deposited TiAlN coating (black curve 1) and of irradiated by He^+ ions with an energy 500 keV and fluence $2x10^{17}$ ion/cm^2 TiAlN coating (red curve 2).

Figure 3 shows the microphotographs of TiAlN coatings structure obtained by SEM.

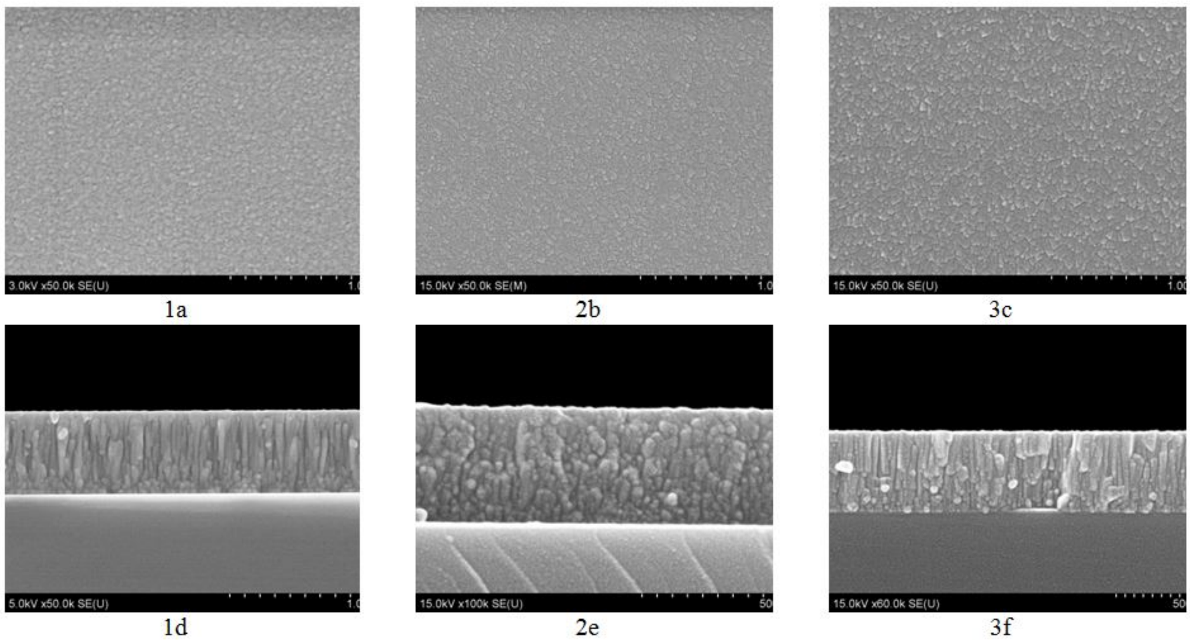

Fig. 3. – SEM microphotographs of TiAlN coatings: 1a, 2b, 3c – plan view images, 1d, 2e, 3f – cross-section images 1 – deposition with a deficiency of nitrogen; 2 – deposition at the stoichiometric concentration of nitrogen; 3 – deposition with an excess of nitrogen.

According to Fig. 3, in all the cases of coating deposition, we have a fine nanocrystalline structure with an average crystalline size of 10-20 nm. In the case of TiAlN coating deposition with a deficiency of nitrogen and with an excess of nitrogen, there is observed a columnar crystalline structure of coatings (Fig. 3. 1d, 3f). In the case of TiAlN coating deposition at the stoichiometric concentration of nitrogen, a globular crystalline structure is formed (Fig. 3. 2e). It is interesting to note, that coating's thickness depends on the deposition regime and is the highest at the deficiency of nitrogen and is the lowest at the excess of nitrogen.

The lowest friction coefficient and mass wear of TiAlN coatings were obtained in the case of the deposition with a deficiency of nitrogen (Fig. 4. Curve 1). Also, the TiAlN coating shows a good tribological performance in the case of deposition at the stoichiometric concentration of nitrogen, where a globular crystalline structure was formed (Fig. 3. 2e.).

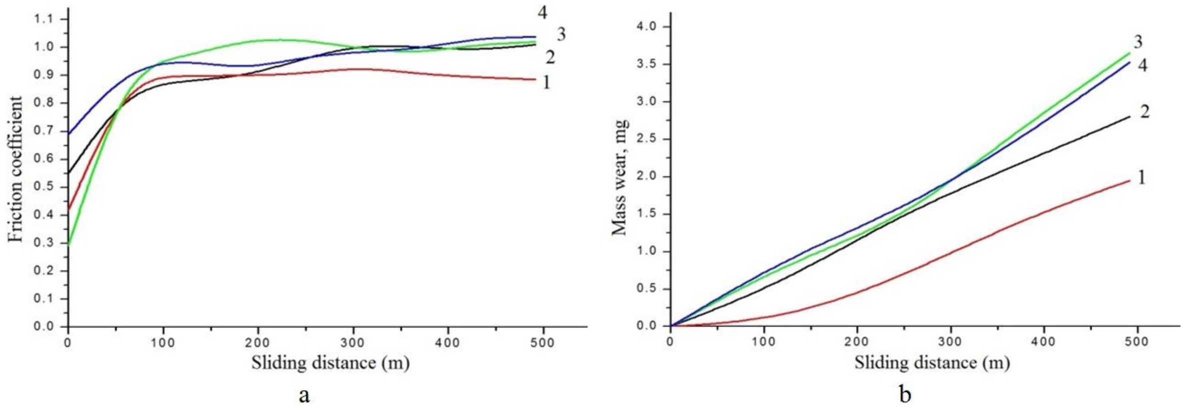

Figure 4 – Friction coefficient of TiAlN coatings and steel substrate (a), mass wear of TiAlN coatings and steel substrate (b). 1 – deposition with a deficiency of nitrogen; 2 – deposition at the stoichiometric concentration of nitrogen; and 3 – deposition with an excess of nitrogen; 4 – steel substrate.

Tribological investigations of irradiated TiAlN coatings were also performed. Presently, the obtained results are under consideration, but we can announce, that the irradiation influences a nonlinear behavior of the friction coefficient of TiAlN coatings on ion fluence. He^+ ion irradiation leads to some hardening of TiAlN coatings up to 5-10% at the fluence of $2x10^{17}$ ions/cm^2 (Fig. 5).

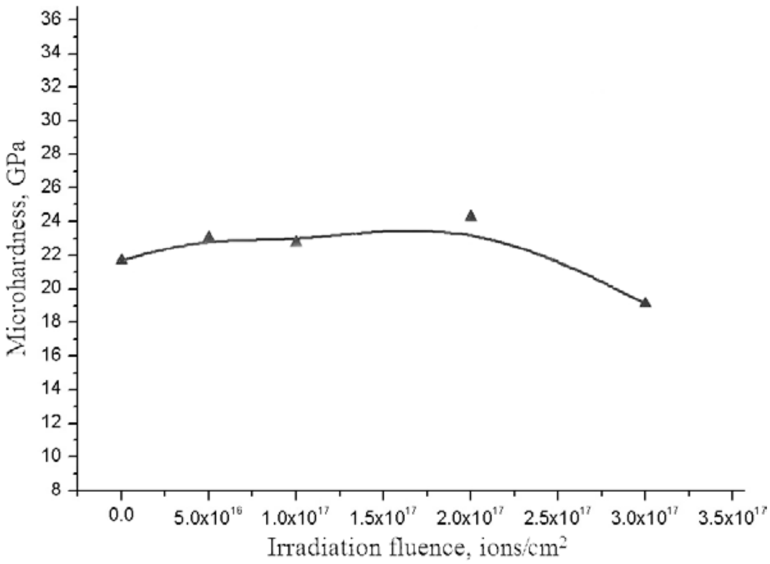

Fig. 5. – TiAlN coatings hardness dependence on the He^+ ion irradiation fluence.

Then, a softening of TiAlN coatings appears for more then 20% at the fluence of $3x10^{17}$ ions/cm^2.

CONCLUSIONS

According to the investigations results, it can be stated, that the method of reactive magnetron sputtering allows obtaining wear resistant TiAlN coatings with dense globular structure. Only one phase of substitution solid solution forms in the coating. The distribution of elements in the deposited TiAlN coating is homogeneous. The efficient regimes for obtaining the best performance properties of TiAlN coating are regimes with a stoichiometric concentration of nitrogen and with a deficiency of nitrogen. It is found, that the nanostructured coatings TiAlN are radiation resistant and not susceptible to degradation under high fluence ion irradiation. TiAlN coatings are perspective as fuel claddings in nuclear reactors.

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