Vol. **136** (2019) *ACTA PHYSICA POLONICA A* No. 2

Proceedings of the 12th International Conference "Ion Implantation and Other Applications of Ions and Electrons", ION 2018

Effects of Nitrogen Selective Sputtering and Flaking of Nanostructured Coating TiN, TiAlN, TiAlYN, TiCrN, (TiHfZrVNb)N under Helium Ion Irradiation

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Modelling of parameters of high-fluence ion irradiation of nanostructured TiN, TiAlN, TiAlYN, TiCrN, (TiHfZrVNb)N coatings by the Monte-Carlo method was carried out. The obtained results and proposed mechanisms of processes were experimentally proved in studying the structure of the coatings after irradiation with 500 keV He^+ ions in the fluence range from 5×10^{16} ion/cm^2 to 3×10^{17} ion/cm^2. The effect of depth localization of the maximum concentration of implanted helium on nitrogen selective sputtering from coatings as well as on partial flaking (exfoliation) of coatings was observed for the ion fluences higher than 2×10^{17} ion/cm^2. No phase segregation of the solid solution as the main phase in the nanostructured coatings was found after the ion irradiation. No smarco- or microblistering of the coatings after ion irradiation was found, at least to a blister size approximating the nanoscale.

DOI: 10.12693/APhysPolA.136.303

PACS/topics: nanostructured coatings TiN, TiAlN, TiAlYN, TiCrN, (TiHfZrVNb)N, radiation resistance, selective sputtering, flaking

1. Introduction

New radiation tolerant materials are in a great demand nowadays. The real need is the creation of new radiation-resistant materials for nuclear power engineering and space technology [1]. In recent years, in the literature, widely discussed information about the possible effect of nanostructuring on the radiation resistance of materials has appeared [2]. It is assumed that the nanostructured material has greater resistance to ionizing irradiation than the microstructured one due to the large integral extent of crystallite boundaries that can act as drains for the radiation-induced defects [3]. Nanostructured coatings with the large integral length of crystalline or phase interfaces are perspective candidates for fuel claddings. Their possibility to annihilate the radiation induced point defects at the interfaces acts like a "self-healing" effect [1, 2].

2. Experimental details

The investigations were carried out on the TiN, TiAlN, TiAlYN, TiCrN, (TiHfZrVNb)N coating samples formed by the method of separable vacuum arc deposition on the AISI 304 steel substrates. For the deposition of TiCrN coatings, the cathodes with different chromium contents were used: (1) 20 wt% Cr; (2) 32 wt% Cr; (3) 42 wt% Cr. The thickness of the coatings was 2 –4 μm. In order to study the radiation resistance, the coatings were irradiated at an accelerator of light ions AN 2500 by He^+ ions with an energy of 500 keV, fluences from 5×10^{16} ion/cm^2 to 3×10^{17} ion/cm^2. Next, the coatings were annealed in an argon atmosphere at 773 K for 15 min.. Calculations of the ion irradiation parameters, concentration profiles of implanted helium, elastically released energy and vacancies were conducted using the Monte-Carlo method and the SRIM 2011 software [4, 5]. The elemental composition of coatings before and after irradiation with He^+ ions was monitored by the X-ray spectral microanalysis (RSMA) performed on a special attachment to the Hitachi SU3400 scanning electron microscope. The study of the morphology and topography of coatings surface was carried out by atomic force microscopy (AFM) using a scanning probe microscope Solver P47 Pro.

3. Results and discussion

The results of modelling of the TiN, TiAlN, TiAlYN, $Ti_xCr_{1-x}N$ for $0.8 \leq x \leq 0.58$, (TiHfZrVNb)N coatings irradiation by He^+ ions with an energy of 500 keV and fluences values of 5×10^{16} ion/cm^2, 1×10^{17} ion/cm^2, 2×10^{17} ion/cm^2, 3×10^{17} ion/cm^2 are presented in the form of graphs in Fig. 1. Tables I and II show the average projected range of helium ions (R_p), straggling of helium ions ($\Delta\ R_p$) and sputtering coefficients of the coating elements (k). Table III presents the calculated maximum concentrations of interstitial helium (at.%), thickness of the helium localization layers (D), maximum intensity of the vacancy generation (V) in the TiN, TiAlN, TiAlYN, $Ti_xCr_{1-x}N$ for $0.8 \leq x \leq 0.58$, (TiHfZrVNb)N coatings.

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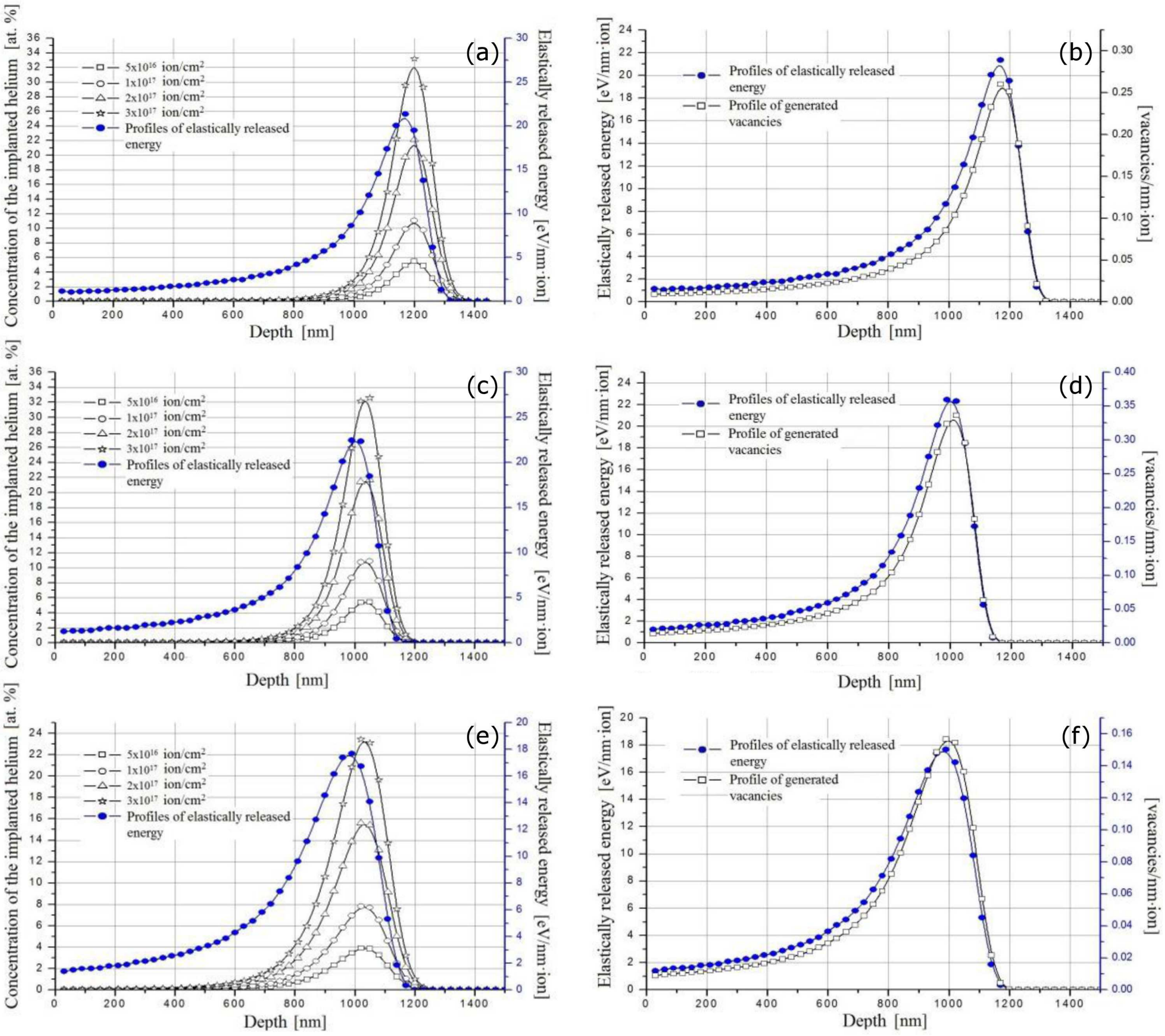

Fig. 1. Concentration profiles of implanted helium and elastically deposited energy (a, c, e), profiles of elastically deposited energy and concentration of vacancies (b, d, f) in the coatings TiAlN (a, b), $Ti_xCr_{1-x}N$ $x = 0.68$ (c, d), (TiHfZrVNb)N (e, f).

TABLE I

Calculated helium ion irradiation parameters of the coatings TiN, TiAlN, TiAlYN, $Ti_xCr_{1-x}N$ for $0.58 \le x \le 0.80$

Coating	R_p [nm]	ΔR_p [nm]	k [atom/ion]				
			N	Ti	Al	Y	Cr
TiN	1000	90.5	0.00537	0.00282	–	–	–
TiAlN	1153	95.3	0.00515	0.00143	0.00179	–	–
TiAlYN	1098	94	0.00473	0.00193	0.00170	0.000016	–
$Ti_xCr_{1-x}N$ $x = 0.80$	992	93.3	0.00589	0.00251	–	–	0.00074
$Ti_xCr_{1-x}N$ $x = 0.68$	987	93.7	0.00540	0.00207	–	–	0.00118
$Ti_xCr_{1-x}N$ $x = 0.58$	984	94	0.00652	0.00187	–	–	0.00171

TABLE II

Calculated helium ion irradiation parameters of the (TiHfZrVNb)N coating

Coating	R_p [nm]	ΔR_p [nm]	k [atom/ion]					
			N	Ti	Hf	Zr	V	Nb
(TiHfZrVNb)N	962	131	0.00567	0.00106	0.000502	0.00042	0.00039	0.000354

TABLE III

Maximum concentrations of implanted helium ($N_{\max}$), thickness of helium localization layers (D), maximum intensity of vacancies generation (V) in the TiN, TiAlN, TiAlYN, $Ti_xCr_{1-x}N$ for $0.58 \leq x \leq 0.80$, (TiHfZrVNb)N coatings

Coating	$N_{\max}$, at. % at the irradiation fluence, ion/cm^2				D [nm]	V [vacancy/(nm ion)]
	5×10^{16}	1×10^{17}	2×10^{17}	3×10^{17}		
TiN	6	11	22	33	300	0.4
TiAlN	5.5	10.5	21.5	32	325	0.26
TiAlYN	5.5	10.5	21.5	32	350	0.27
$Ti_xCr_{1-x}N$ $x = 0.80$	5.5	11	21.5	32.5	325	0.36
$Ti_xCr_{1-x}N$ $x = 0.68$	5.5	10.5	21.5	32	350	0.33
$Ti_xCr_{1-x}N$ $x = 0.58$	5.5	10.5	21	32	375	0.31
(TiHfZrVNb)N	3.9	7.8	15.5	23	500	0.15

In all calculations, the density of the TiN, TiAlN, TiAlYN, TiCrN, (TiHfZrVNb)N coatings was determined using the tabular data for simple nitrides [6, 7], taking into account the partial concentrations of the elements in the coatings. The threshold energy of the displacement of atoms from the lattice site E_d was taken from the literature [8–12].

The average projected range of helium ions with an energy of 500 keV amounted to 1 μm in the TiN coating, 1.153 μm in the TiAlN coating, 1.098 μm in the TiAlYN coating, 0.984–0.992 μm in the TiCrN coating and 0.962 μm in the (TiHfZrVNb)N coating. The largest average projected range of helium ions (R_p) corresponds to the TiAlN coating–1153 nm, the smallest to the (TiHfZrVNb)N coating–962 nm. It should be noted that with the increasing atomic number and mass of the element in the material composition, its elastic scattering cross section and the stopping cross sections (nuclear and electronic) increase, and thus the penetration depth of ions into the material decreases. Thus, the average projected range of helium ions (R_p) in the TiAlYN coating at an yttrium concentration of 0.3 at.% is less than in TiAlN and equals 1098 nm. The R_p value also decreases in the TiCrN coating as the chromium concentration increases from 10 at.% up to 21 at.% from 992 nm to 984 nm, respectively.

The smallest straggling (ΔR_p) was recorded in the TiN coating — 90.5 nm, the largest $\Delta R_p = 131.3$ nm — in the coating (TiHfZrVNb)N. With an increase in the thickness of the implanted layer, the stresses created by the accumulation of helium would be localized to a larger extent, which may allow the material to withstand a greater irradiation fluence without blistering and flaking. With a decrease in the thickness of the implanted layer, the volume of material subjected to tensile stresses from the embedded helium would be smaller [8, 9] which can lead to flaking of the material with lower fluence. From this point of view, it can be predicted that the TiN coating will withstand the lowest irradiation fluence without degradation in comparison with the TiAlN, TiAlYN, TiCrN coatings, and the coating (TiHfZrVNb)N will be the most resistant to irradiation.

The sputtering coefficient (k) of titanium in the TiN, TiAlN, TiAlYN, TiCrN, (TiHfZrVNb)N coatings was in the range of 0.00143–0.00282 atom/ion, (k) of aluminum 0.00170–0.00179 atom/ion, (k) of chromium 0.00074–0.00170 atom/ion, (k) of yttrium 0.000016 atom/ion. The sputtering coefficient (k) of nitrogen was in the range of 0.00473–0.00652 atom/ion, and (k) of coating metals of (TiHfZrVNb)N was in the range of 0.000354–0.00106 atom/ion. It is found that the sputtering coefficient of nitrogen by He^+ ions with 500 keV energy is 2–3 or more times higher than the sputtering coefficients of other elements that make up the coatings. Consequently, the surface layer of coatings after the high-fluence irradiation can be depleted in nitrogen and enriched with metal atoms.

As can be seen from the calculation results, the sputtering coefficient of the elements of coatings irradiated with He^+ ions of 500 keV does not exceed 0.007 atom/ion. On the whole it indicates that the effect of sputtering is very small.

As can be seen from Fig. 1, the maximum of the elastically deposited energy in all coatings is shifted to a smaller depth by $\sim$ 25–50 nm in comparison with the localization of the maximum concentration of the implanted helium. This fact corresponds to the theoretical base of ion implantation, within the framework of which it is considered and explained [10, 11]. The maximum of the generated vacancies is shifted in the depth by $\sim$ 5–25 nm in comparison with the maximum of the elastically released energy, and is simultaneously shifted to a smaller depth by $\sim$ 20–25 nm compared to the localization of the maximum concentration of the implanted helium.

According to these calculations, it is found that the elastically released energy from He^+ ions with an energy of 500 keV upon irradiation of all coatings increases from 1–2 eV/(nm ion) to 17.5–22.5 eV/(nm ion) during the passage of an ion from the surface to a depth of 900–1100 nm. In the 0–800 nm depths range of coatings, inelastic stopping dominates and as a consequence, in this region there is practically no implanted helium and the concentration of point radiation defects is small.

In the depth range of 800–1100 nm, the elastic energy loss dominates and therefore the generation of point defects as well as accumulation of helium occurs. In this region the concentration of helium is maximal: according to the calculations from 4 at.% up to 34 at.%, depending on the fluence of irradiation in the range of 5×10^{16}–3×10^{17} ion/cm^2. The maximum concentration of interstitial helium in the TiN coating at a fluence of 5×10^{16} ion/cm^2 is 6 at.%, at 1×10^{17} ion/cm^2–11 at.%, at 2×10^{17} ion/cm^2–22 at.%, at 3×10^{17} ion/cm^2–33 at.%. The thickness of the localized layer of implanted helium in the TiN coating is 300 nm. In the TiAlN, TiAlYN, TiCrN coatings, the maximum concentrations of the incorporated helium are slightly smaller and equal to: 5.5 at.% at a fluence of 5×10^{16} ion/cm^2, 10.5–11 at.% at a fluence of 1×10^{17} ion/cm^2, 21–21.5 at.% at a fluence of 2×10^{17} ion/cm^2, 32–32.5 at.% at a fluence of 3×10^{17} ion/cm^2. The thickness of the localized layer of implanted helium in the TiAlN, TiAlYN, TiCrN coatings increases with the increase in straggling from 325 nm to 375 nm. As mentioned above, this helps to reduce the level of tensile stresses per unit volume of material at high irradiation fluences [8, 9, 13] and consequently, increase the radiation resistance of coatings. In the coating (TiHfZrVNb)N, the calculated maximum concentration of interstitial helium is minimal among the studied coatings and amounts to: 3.9 at.% at a fluence of 5×10^{16} ion/cm^2, 7.8 at.% at a fluence of 1×10^{17} ion/cm^2, 15.5 at.% at a fluence of 2×10^{17} ion/cm^2, 23 at.% at a fluence of 3×10^{17} ion/cm^2. In these cases, the thickness of the localized layer of implanted helium in the coating (TiHfZrVNb)N is maximal — 500 nm. High mass numbers and a large number of constituent elements of the coating (TiHfZrVNb)N contribute to an increase in the straggling of helium ions and can provide an increase in the radiation resistance of this coating.

It is found that the most active generation of point defects upon irradiation with He$^+$ ions with an energy of 500 keV occurs upon irradiation of TiN coatings. The maximum intensity of generation of vacancies in this case is 0.4 vacancies/(nm ion). The same parameter of the TiAlN, TiAlYN coatings is 0.26 vacancies/(nm ion) and 0.27 vacancies/(nm ion), respectively. Therefore the addition of aluminum and yttrium to the TiN coating reduces the concentration of generated vacancies upon irradiation and can increase their radiation resistance. With an increase in the chromium concentration in the TiCrN coating from 10 at.% up to 21 at.%, the maximum intensity of the vacancy generation decreases from 0.36 vacancies/(nm ion) to 0.31 vacancies/(nm ion). It can be assumed that an increase in the chromium concentration in the composition of the TiCrN coating affects the reduction of the concentration of radiation-induced point defects and an increase in radiation resistance. The lowest value of the maximum intensity of the generation of vacancies, 0.15 vacancies/(nm ion), is predicted on the basis of calculations in the case of (TiHfZrVNb)N coating irradiation. In addition, it is possible that the high entropy of mixing combined with the large threshold energy of formation of vacancies and interstitial atoms makes the coating (TiHfZrVNb)N the most resistant to the accumulation of point radiation-induced defects.

Determination of the mechanism of helium ions interaction with the coating material is possible when estimating the energies of the primary knocked out atoms (PKA). The energy E_2 obtained by the target atom in each individual collision is proportional to the energy of the incident ion and is determined by the relation [14, 15]:

$$E_2 = \frac{4M_1M_2}{(M_1+M_2)^2} E \sin^2 \frac{\phi}{2}, \qquad (1)$$

where φ is the scattering angle in the center-of-mass system, M_1 is the mass of the ion, M_2 is the mass of the target atom [15].

In head-on collisions, E_2 is determined from the expression

$$E_2^{\max} = \frac{4M_1M_2}{(M_1+M_2)^2} E. \qquad (2)$$

Based on the results of calculations, the energy of the primary knocked-out atoms was plotted against the atomic mass of the elements of the TiN, TiAlN, TiAlYN, TiCrN, (TiHfZrVNb)N coatings irradiated with 500 keV He$^+$ ions shown in Fig. 2.

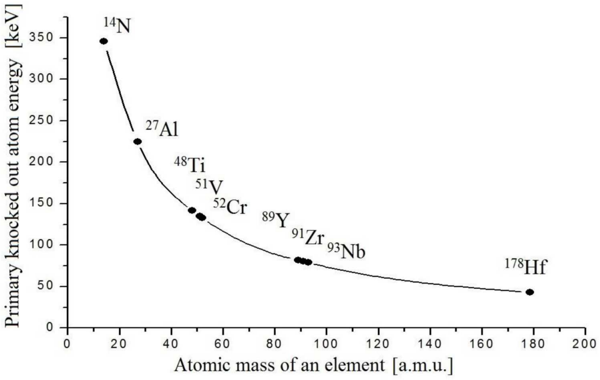

Fig. 2. The dependence of energy of primary knocked-out atoms on the atomic mass of elements.

When the coatings are irradiated with 500 keV He$^+$ ions, PKA has an energy of 345 keV for nitrogen, 225 keV for aluminum, 142 keV for titanium, 135 keV for vanadium, 133 keV for chromium, 82 keV for yttrium, 81 keV for zirconium, 79 keV for niobium, 43 keV for hafnium. The energies of PKA of all the considered elements exceed significantly the threshold energies of the atoms displacement from the crystal lattice sites [10]. The energy of nitrogen PKA allows them to scatter at large angles in collision with heavier atoms, leave the material through the surface and exit into the environment. Also, some sputtering and escape to the environment of aluminum atoms can be observed. PKAs of titanium, vanadium, chromium, yttrium, zirconium, niobium, hafnium have small ranges (~ 10–50 nm), and therefore their escape from the surface is an inefficient process. The results of these calculations indicate that the selective sputtering in the near-surface region of the material may occur preferably for nitrogen atoms.

On the basis of calculations, it can be asserted that irradiation of coatings by He^+ ions with an energy of 500 keV leads to the nitrogen atoms sputtering by the mechanism of the primary knocked-out atoms and the cascade mechanism. Atoms of aluminum, titanium, vanadium, chromium are sputtered only by a cascade mechanism. Atoms of yttrium, zirconium, niobium, and hafnium are mainly sputtered by the mechanism of displacement peaks [15].

A widely used parameter in the nuclear and radiation materials science is the number of displacements per atom (dpa) [10, 13, 14, 16, 17]. One dpa physically means the irradiation fluence in which on the average each atom of the material undergoes a displacement from its position in the crystal lattice [18]. The number of dpa is calculated by the formula [18]:

$$\mathrm{dpa} = \frac{0.8}{E_d}\frac{D}{\rho}\left(\frac{\mathrm{d}E}{\mathrm{d}x}\right)_{\mathrm{nuclear}}, \tag{3}$$

where E_d is the threshold energy of displacement of the atom from the node of the crystal lattice, D is the irradiation fluence, ρ is the atomic density of the material, $(\frac{\mathrm{d}E}{\mathrm{d}x})_{\mathrm{nuclear}}$ is the average nuclear energy loss per unit path length.

Table IV shows the calculated dpa values for the coatings under investigations when irradiated with helium ions with an energy of 500 keV. For most materials of modern nuclear reactors (PWR, BWR), the amount of dpa is less than 1 for 40 years of operation. However, for active elements of fast neutron reactors, the defect accumulation rate can reach more than 10 dpa/year [19]. Taking into account the calculated values of dpa, it can be asserted that the study of the TiN, TiAlN, TiAlYN, $\mathrm{Ti}_x\mathrm{Cr}_{1-x}\mathrm{N}$ for $0.58 \le x \le 0.8$, (TiHfZrVNb)N coatings with the chosen fluences of He^+ ions is practically important for nuclear material science.

TABLE IV

Calculated dpa values for the TiN, TiAlN, TiAlYN, $\mathrm{Ti}_x\mathrm{Cr}_{1-x}\mathrm{N}$ for $0.58 \le x \le 0.8$, (TiHfZrVNb)N coatings when irradiated with 500 keV He^+ ions, in the fluence range 5×10^{16} ion/cm^2–3×10^{17} ion/cm^2

Coating	Irradiation fluence [ion/cm^2]			
	5×10^{16}	1×10^{17}	2×10^{17}	3×10^{17}
	dpa			
TiN	3.324	6.648	13.296	19.944
TiAlN	8.116	16.232	32.464	48.696
TiAlYN	7.615	15.23	30.46	45.69
$\mathrm{Ti}_x\mathrm{Cr}_{1-x}\mathrm{N}$, $x = 0.8$	6.628	13.256	26.512	39.768
$\mathrm{Ti}_x\mathrm{Cr}_{1-x}\mathrm{N}$, $x = 0.68$	6.385	12.77	25.54	38.31
$\mathrm{Ti}_x\mathrm{Cr}_{1-x}\mathrm{N}$, $x = 0.58$	6.45	12.9	25.8	38.7
(TiHfZrVNb)N	3.556	7.112	14.224	21.336

As follows from Table IV, the selected fluences by the number of dpa exceed the characteristic values in the known structural materials of the reactors for the period of their operation. Tables V and VI give the data on the elemental composition of the coatings before and after irradiation according to the results of RSMA (at.%). It was found that after irradiation of the coating, the homogeneity of the elemental composition is maintained. There are no transmutational nuclear reactions in the coating material during the irradiation process since 500 keV energy is not enough to trigger these processes [10]. As can be seen from the abovementioned results, after irradiation, there is no significant change in the average concentration of elements in the coating. The fluctuations in the concentrations of the elements do not exceed 1 at.%. However, it is noteworthy that in all cases the concentration of nitrogen decreases (not more than 1–2 %). This is a consequence of the predominant radiation sputtering of nitrogen as the lightest element by the incident He^+ ion flux. The possibility of such a process was indicated by the results of theoretical calculations. A decrease in the nitrogen concentration leads to an increase in the recorded relative concentration of other elements.

TABLE V

Elemental composition of the TiN, TiAlN, TiAlYN coatings before and after irradiation with He^+ ions with the energy of 500 keV and the fluence 2×10^{17} ion/cm^2

Coating	Elements concentration before and after helium ion irradiation, [at.%]							
	Ti		Al		Y		N	
	before	after	before	after	before	after	before	after
TiN	51.5	52.5	0	0	0	0	48.5	47.5
TiAlN	29	29.5	27	27.5	0	0	44	43
TiAlYN	31	31.3	25	25.3	0.3	0.4	43.7	43

TABLE VI

Elemental composition of the (TiHfZrVNb)N coatings before and after irradiation with He^+ ions with the energy 500 keV and the fluence 3×10^{17} ion/cm^2

Sample	Elements concentration before and after helium ion irradiation [at.%]						
	Element	Ti	Hf	Zr	V	Nb	N
1	before	17	11.5	8.5	6	9	48
	after	17.2	11.7	8.7	6.2	9.2	47
2	before	25.5	7	6	5	7.5	49
	after	25.7	7.2	6.2	5.2	7.7	48

Figure 3 shows the results of AFM studies on the surface of the TiN, TiAlN, TiCrN coatings after irradiation with 500 keV He^+ ions, 2×10^{17} ion/cm^2 fluence, and annealing at 500 °C for 15 min. Table VII shows the calculated depths of the average projected range of helium ions (R_p) and the experimentally obtained results of the AFM (Fig. 3) for the depths of TiN, TiAlN, $\mathrm{Ti}_x\mathrm{Cr}_{1-x}\mathrm{N}$, with $x = 0.8$ coatings destruction.

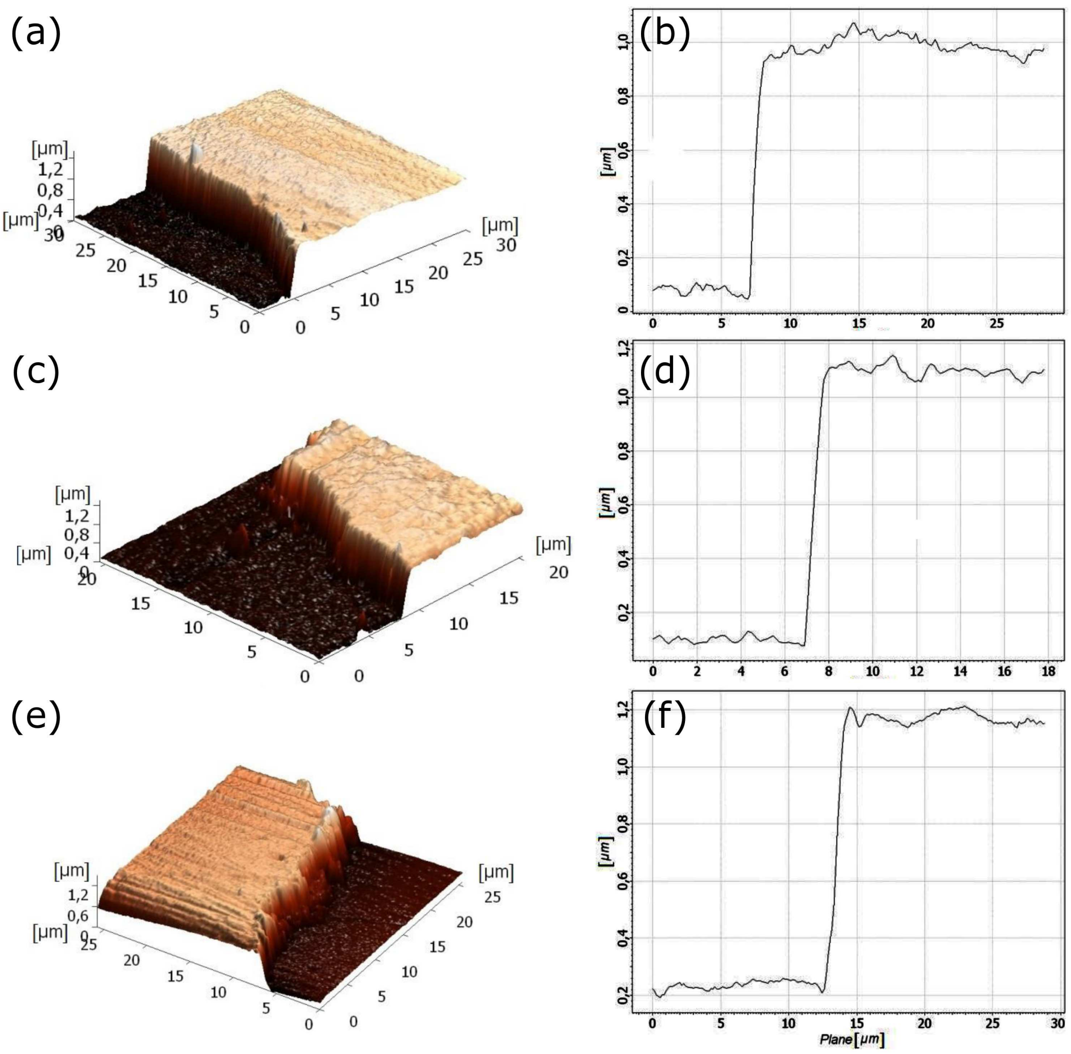

Fig. 3. The morphology of the surface (a, c, e) and the profilograms (b, d, f) obtained for the coatings of TiN (a, b), TiAlN (c, d), $Ti_xCr_{1-x}N$, with $x = 0.80$ (d, e) after irradiation with He^+ ions with an energy of 500 keV, fluence 2×10^{17} ion/cm^2 and annealing at 500 °C for 15 min.

TABLE VII

Calculated average projected ranges of helium ions with an energy of 500 keV (R_p) and the experimentally determined by the results of AFM depths of TiN, TiAlN, $Ti_xCr_{1-x}N$, with $x = 0.80$ coatings destruction

Coating	R_p [nm]	The depth of coating destruction [nm]
TiN	1000	1016
TiAlN	1153	1068
$Ti_xCr_{1-x}N$, $x = 0.80$	992	971

From the analysis of the results of AFM (Fig. 3, Table VII), it follows that the destruction of coatings (flaking) under the influence of He^+ ion irradiation with an energy of 500 keV by a fluence of 2×10^{17} ion/cm^2 and an annealing at 500 °C for 15 min occurs at about the depth of the average projected range of helium ions or, more accurately, to the depths of localization of the maximum of radiation-induced defects and implanted impurities. The absence of micro- and macroblasting of coatings at the studied irradiation fluences up to 3×10^{17} ion/cm^2 is also detected, which is caused by the effective recombination of point defects at the grain surfaces in the nanostructured coatings.

5. Conclusion

The effect of selective sputtering of nitrogen (1–2 at.%) upon irradiation of the nanostructured TiN, TiAlN, TiAlYN, (TiHfZrVNb)N coatings with helium ions with an energy of 500 keV in the fluence range from 5×10^{16} ion/cm^2 to 3×10^{17} ion/cm^2 was observed. It was found that ion irradiation causes a partial coatings flaking at the depth of localization of the maximum concentration of the incorporated helium at an irradiation fluence of more than 2×10^{17} ion/cm^2, apparently due to the previous formation of nanoblisters. The absence of micro- and macroblistering of coatings at the investigated irradiation fluences up to 3×10^{17} ion/cm^2 is also found which is caused by the high specific surface area of nanograins in the nanostructured materials in which the grain boundaries are effective drains for the point defects generated by irradiation that is their effective recombination in these areas. Thus the results of the theoretical calculations and modelling of the ion irradiation of the nanostructured TiN, TiAlN, TiAlYN, TiCrN, (TiHfZrVNb)N coatings using the Monte-Carlo method and the SRIM 2011 software correspond to the observed results.

Based on the results of the study, it can be stated that the thin-film nanostructured TiAlN, TiAlYN, $Ti_xCr_{1-x}N$ for $0.58 \leq x \leq 0.8$, (TiHfZrVNb)N coatings have high phase stability and radiation resistance up to the fluence of 2×10^{17} ion/cm^2. These coatings have great perspectives of practical application as protective radiation-resistant on fuel claddings in nuclear reactors, as well as in equipment and bodies of spacecrafts.

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