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EFFECT OF DEUTERIUM IMPLANTATION DOSE ON PROPERTIES OF CrN COATINGS

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The methods of X-ray diffraction analysis, atomic-force microscopy, nanoindentation and thermodesorption spectroscopy have been applied to investigate the effect of a dose (from $5\cdot10^{16}$ to $1.5\cdot10^{18}$ D/cm^2) of implanted deuterium with energy of 24 keV on the structure, surface morphology and mechanical properties of vacuum-arc CrN coatings. Deuterium ion implantation in the range of doses from $5\cdot10^{16}$ to $1.5\cdot10^{17}$ D/cm^2 decreases by 10…15% the nanohardness and elastic modulus of coatings. Under exposition to doses $\geq 1\cdot10^{18}$ D/cm^2 the coating nanohardness sharply decreases because of blisters being formed and occupying about 30% of the CrN coating surface. Deuterium implantation did not lead to formation of new phases in the CrN coating.

INTRODUCTION

Wide application of transition metal nitrides as protective coatings in industry is due to their excellent mechanical properties and high corrosion stability in aggressive environment. Vacuum-arc deposition makes it possible to obtain protective nitride films on different materials [1]. However, the use of such coatings in the atomic industry requires a comprehensive understanding of their behavior in the irradiation environments. At present, the extensive studies are carried out on stability of the properties of bulk nitrides [2] and nitride coatings [3–8] under different-type ion irradiations. Irradiation with inert gases creates radiation defects in the coating and can lead to amorphization and to blistering at high doses [9]. This negative factor can influence on the coating protective properties under radiation exposure. Nitride coatings have a higher radiation resistance, as compared to pure metals, steels and alloys, due to the perfect nanostructure and strong chemical bonds of elements. Chemically active gases, e. g. hydrogen and its isotopes, can initiate phase changes in materials leading to the degradation of their properties [10]. In water-cooled reactors, there are two major mechanisms of hydrogen formation – oxidation of zirconium and radiolysis of water [11]. For different types of nuclear power plants in structural materials accumulated various levels of the hydrogen atoms (from a few to thousands appm) [12]. Therefore, to study the hydrogen implantation effect on the properties of nitride-containing coatings is an urgent problem.

Previously we have investigated the implanted deuterium dose influence on the hardness and structure of TiN [13], TiAlSiN, TiAlYN [14] coatings and high-entropy TiZrNbHfVN [15] coating. It has been established that the deuterium thermodesorption spectrum (TDS) structure is a function of the implanted deuterium dose. As the implanted deuterium dose increases the temperature range of deuterium desorption from the coatings extends towards the temperature decrease [13, 14]. When the irradiation dose exceeds $5\cdot10^{17}$ D/см^2 the hardness of nitride coatings becomes almost twice as little [15].

Among two-component nitride coatings the CrN coating demonstrates the highest corrosion resistance in the super-critical water [16]. Application of the CrN coating, as one of the protective layers in zirconium fuel element tubes, sharply increases their resistance to the high-temperature air oxidation [17–19]. However, there are no sufficient data available as to the behavior of this coating under irradiation. For example, in [20] it is shown that the CrN corrosion resistance in the super-critical water can be decreased under γ-irradiation. The authors of [21] have observed the change in the lattice parameters, microstresses and grain size of the magnetron CrN coatings deposited on the silicon substrate under irradiation with 120 keV Ar ions at a dose of $1\cdot10^{16}$ ions/cm^2. The structure stability after irradiation of nanocrystalline CrN coatings was studied in [22]. It has been established that under nitrogen ion irradiation to the damage levels of 25 dpa their electrical resistivity increases. At the same time, the radiation exposure does not exert significant influence on the coating grain size and does not lead to their amorphization.

In the literature data on the changes of the mechanical properties of CrN coating after irradiation are absent. For use protective coating in the hydrogen (deuterium) containing environment it will need to determine what concentration of accumulated deuterium can cause structural changes and degradation of the mechanical properties of the coatings.

In this paper we investigate the implanted deuterium dose influence on the mechanical properties, structure, morphology and temperature ranges of deuterium desorption from CrN coatings prepared by a vacuum-arc deposition.

1. EXPERIMENTAL

Coatings with 5 μm thickness were deposited from the vacuum-arc plasma stream [23] separated from macroparticles under 0.36 Pa nitrogen pressure, 100 A arc current and -100 V bias potential on the polished ($R_a \sim 20$ nm) substrates made from Ch18Ni10T steel (20×10×1.5 mm). Temperature of specimens during

deposition did not exceed 700 K. Cathodes (∅ 60 mm) were of chromium (99.9%).

The coatings were irradiated with a 24 keV D_2^+ ion beam at doses from $5 \cdot 10^{16}$ to $1.5 \cdot 10^{18}$ D/cm^2 at temperature of 293 K. The temperature range of ion-implanted deuterium desorption from the specimens was investigated by the thermodesorption spectroscopy method using the device "Skif" [24]. The specimens were placed on the heaters made of Cr18Ni10T steel ribbon. The temperature was measured with a tungsten-rhenium thermocouple VR5/20 attached to the specimen. After implantation of a given deuterium dose the beam was stopped and then the heating was switched on. During heating the specimen temperature was increasing to ~ 1600 K by the linear law versus time with an average heating rate of ~ 3.5 K/s. Deuterium release in the measuring chamber was recorded with a mass-spectrometer by m = 4 a.m.u. (D_2^+). The implanted deuterium distribution profile was calculated by the program SRIM 2008 [25]. Calculation results are shown in Fig. 1. Maximum of deuterium profile is at a depth of ~ 104 nm, and the maximum depth of the implanted layer is about 250 nm. We have previously shown that the calculated depth of deuterium is in good agreement with the measured in CrN coatings [26].

The phase composition and substructure of coatings were investigated by the method of X-ray diffraction analysis with a diffractometer DRON-4-07 in the filtered copper anode radiation (Cu-K**α** radiation). Diffraction patterns were taken by the θ-2θ scanning circuit with a Brag-Brentano focusing within the angle intervals from 25 to 100 degrees. The diffraction pattern processing was performed using the computer program New_Profile. By the position of diffraction lines identified as lines of nitride with a cubic structure of NaCl type the crystalline lattice period in the normal-to-film surface direction (a) was determined. The crystallite size in the nitride coating was evaluated by the broadening of line (200) (L) from the Scherrer relation.

The surface morphology of the coatings before and after irradiation was inspected with a scanning atomic-force microscope (AFM) NanoScope IIIa in the periodic contact mode. Silicon probes having a nominal point radius of 10 nm were used.

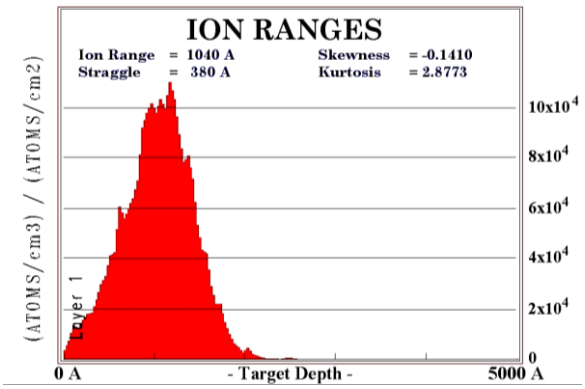


Fig. 1. The depth distribution profile of the ion-implanted deuterium with an energy of 24 keV in the CrN coating (calculated by SRIM 2008)

Nanoindentation was performed by means of the instrument Nanoindenter G200 ("Agilent Technologies", USA) using a Berkovich diamond indenter having a 230 nm radius. The instrument is provided with an attachment designed for continuous control of the contact stiffness (CSM) that makes it possible to determine the dependence of the hardness and elastic modulus on the indenter penetration depth from the results of a single test. The hardness was measured to the indenter penetration depth of 300 nm. The tests were conducted with a constant deformation rate in an imprint equals to 0.05 s^{-1}. The hardness and the elastic modulus were determined by the method of Oliver and Pharr [27].

2. RESULTS AND DISCUSSION

Fig. 2 presents the diffraction pattern of the as-deposited CrN coating. Here besides the substrate lines (denoted by S) one can see the lines of CrN nitride having a cubic structure (NaCl-type structure). The dashed lines show the CrN peak position (PDF card number No 11-0065, JCPDS cards numbers: 03-065-6914; 03-065-9001; 03-065-2829). The observed nitride line shift on the diffraction patterns towards the smaller angles, relatively to the position of corresponding nitride lines, can be caused by formation of compression residual stresses in coatings.

On the diffraction pattern of CrN coatings only lines (200) and (311) are visible. The crystallite size of the coating is 11 nm, the lattice parameter is **a** = 0.424 nm. A low intensity of lines, a high level and characteristic shape of the background suggests that the CrN coating contains, besides crystalline nitride, a significant amount of X-ray amorphous phase.

The deuterium ion implantation at a dose of $1.5 \cdot 10^{18}$ D/cm^2 does not lead to significant changes in the diffraction patterns of coatings: the pattern looks unchanged, new diffraction reflections do not appear, the width of lines and their intensity relation show no changes that evidence on the coating structure stability. The absence of changes on the CrN coating diffraction pattern can be explained by the fact that deuterium interacts mainly with the amorphous component of this condensate, and its implantation depth is insufficient for the X-ray diffraction analysis.

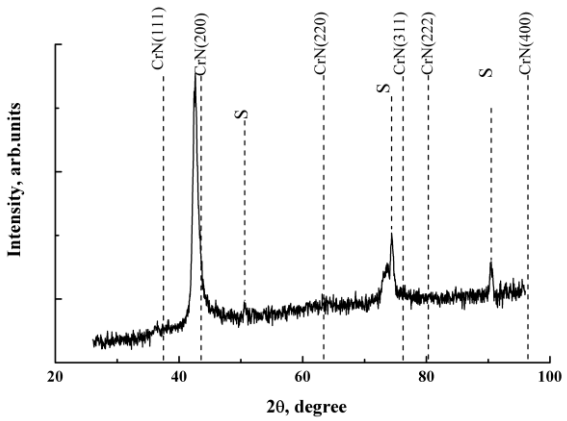
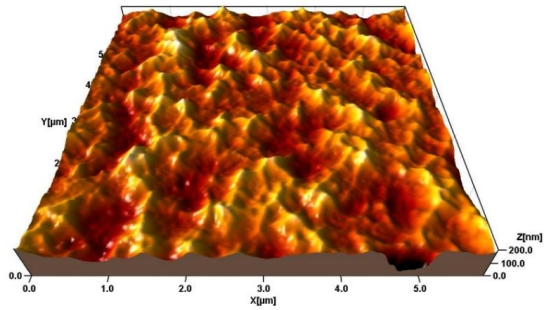
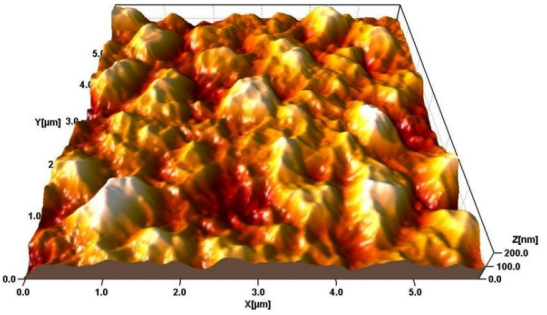


Fig. 2. Diffraction pattern of the CrN coating

The AFM images of the surface fragments of unirradiated and irradiated coatings are shown in Fig. 3,a,b. The relief of as-deposited films has a cellular structure which is characteristic for the method applied. The roughness is ~ 60 nm. Deuterium implantation at a dose of $1.5 \cdot 10^{18}$ D/cm^2 significantly changes the coating surface that can be interpreted as a blister formation.



a



b

Fig. 3. Morphology of the CrN coating surface before (a) and after (b) deuterium implantation at a dose of $1.5 \cdot 10^{18}$ D/cm^2

One can see (see Fig. 3,b) that on the CrN coating an intensive increase of cellular sizes takes place. Blisters cover ~ 30% of the surface, their height is ~ 50…200 nm and the characteristic volume of a single blister is ~ $1 \cdot 10^8$ nm^3. As a result of blister formation the coating surface roughness increases to $R_a \approx 200$ nm.

The authors of [28] have observed the hydrogen blistering on the specimens of TiN and HfN nitride coatings irradiated with 40 keV deuterium ions at a dose of $1.25 \cdot 10^{18}$ D/cm^2. It should be noted that in our case blisters do not break up even at a maximum irradiation dose of $1.5 \cdot 10^{18}$ D/cm^2 probably due to the presence in the CrN coatings structure of an amorphous component which promotes the deuterium dissolution in the implantation layer.

Fig. 4 presents the values of the hardness and elastic modulus for coatings before irradiation and after deuterium irradiation at doses within the range $5 \cdot 10^{16}$…$1.5 \cdot 10^{18}$ D/cm^2 at a depth of indenter penetration to 300 nm. It is seen that the CrN coatings are characterized by the high hardness of ~ 30 GPa and the elastic modulus of ~ 330 GPa. As a result of deuterium ion implantation the hardness and elastic modulus of the CrN coating are decreased. The implantation dose range can be conventionally divided into three parts by the degree of their influence on the mechanical properties of the coating. The changes are minimal at a dose of $5 \cdot 10^{16}$ D/cm^2, and at doses of $(1…5) \cdot 10^{17}$ D/cm^2 the hardness decreases insignificantly (by 10…15%). A sharp decrease of the nanohardness to ~ 12 GPa and of the elastic modulus to 200 GPa occurs under deuterium irradiation at doses of $(1…1.5) \cdot 10^{18}$ D/cm^2.

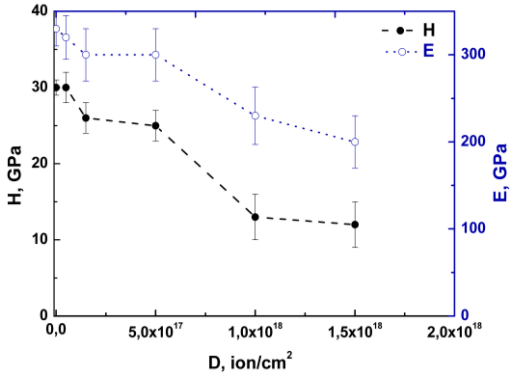


Fig. 4. Effect of the deuterium irradiation dose on the hardness (H) and elastic modulus (E) of CrN coatings

The indenter penetration diagrams obtained for the as-deposited CrN coatings and for the coatings irradiated at a dose of $1.5 \cdot 10^{18}$ D/cm^2 are presented in Fig. 5. For the as-deposited CrN coating the penetration diagram is typical, i. e. the indenter tip moves with load increasing. After coating irradiation the character of the load-displacement curves varies. For all the indentations on the load-displacement curve a sharp increase of the indenter movement by 10…25 nm (pop-in) is observed and the depth of pop-in formation for each indentation is different (200…250 nm) (see Fig. 5). This is somewhat deeper than the calculated profile maximum of deuterium depth of ~ 160 nm. The critical load value, at which a pop-in is formed, varies between 20 and 25 mN. In our opinion such a behavior of the penetration diagram for CrN coatings irradiated at a dose of $1.5 \cdot 10^{18}$ D/cm^2 can be explained by the presence in the coating of regions with very low hardness. It is quite possible that the regions being formed are hydride blisters. The authors of [6] have observed a similar effect of the pop-in formation under irradiation of Ti-Zr-N magnetron coatings with 360 keV Xe ions at a dose of $8 \cdot 10^{14}$ ions/cm^2 that explained by the formation of voids and gas bubbles in the coating. Judging from a sharp decrease of the nanohardness in Fig. 3, the blister formation begins after the implantation dose above $5 \cdot 10^{17}$ D/cm^2.

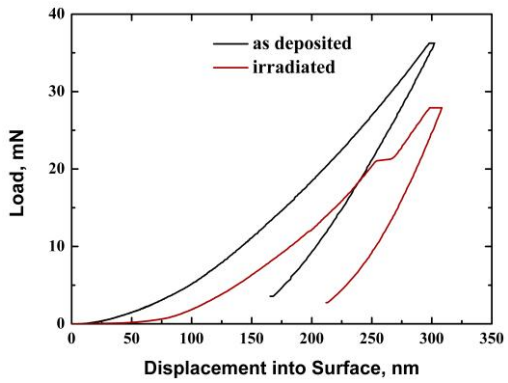


Fig. 5. Load-displacement curves of the CrN coating: as-deposited and irradiated at a dose of $1.5 \cdot 10^{18}$ D/cm^2

So, the presence of pop-ins in the loading diagram for the irradiated coating compared with the unirradiated coating confirms the assumption of blistering formation in the CrN coatings irradiated at a dose of $1.5 \cdot 10^{18}$ D/cm^2.

Fig. 6 shows the deuterium thermodesorption spectra of CrN coatings irradiated at different doses.

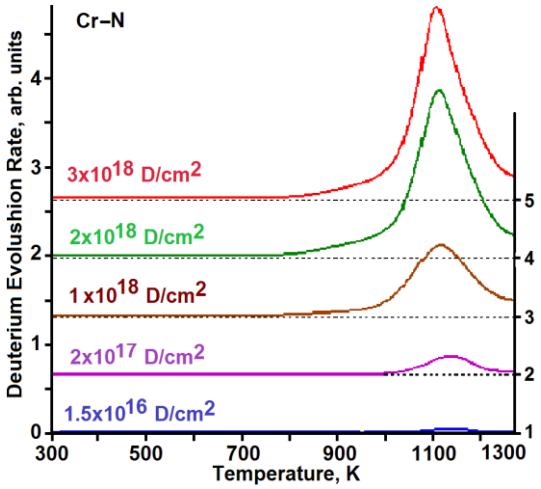


Fig. 6. Deuterium thermodesorption spectra of CrN coatings irradiated at different doses: $1.5 \cdot 10^{16}$...$3 \cdot 10^{18}$ D/cm^2

One can see that in the deuterium TDS of CrN coatings there is only a single desorption temperature range with the center of gravity at 1120 K throughout the range of implantation doses under consideration. A single-peaked behavior of the deuterium TDS points to the presence of only one coating structure state which is unchangeable throughout the range of implanted deuterium.

Fig. 7 presents the dependence of the total amount of retained deuterium on the irradiation dose. This dependence is linear only up to a dose of $2.5 \cdot 10^{18}$ D/cm^2. Then a sharp deviation from the linearity and the attainment of saturation is observed.

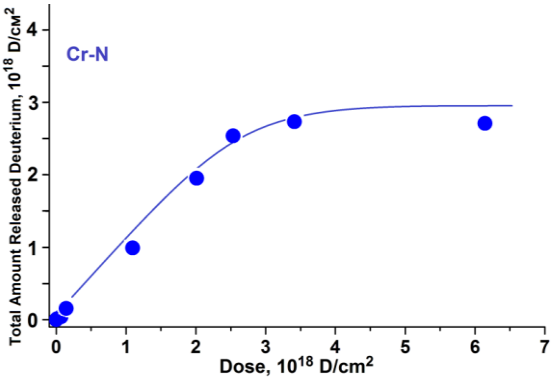


Fig. 7. Total amount of desorbed deuterium as a function of an irradiation dose in the CrN coating

Implanted deuterium in the CrN coating is practically motionless and takes place within the bounds of the implantation profile. Therefore, taking into account the average projective range of 24 keV D_2^+ ions in the chromium nitride it is easy to show that at a saturation dose of $2.5 \cdot 10^{18}$ D/cm^2 the deuterium concentration in the implantation layer is of about 2 D atoms per 1 Cr atom. A single peak in the deuterium TDS generally characterizes the formation and decomposition of the deuterium solid solution phase in metals [29] (CrN coating in our case). A lack of low-temperature peak formation in the thermodesorption spectra by increasing the implanted deuterium dose, characteristic for hydride-forming metals, indicates to the absence of deuterium-CrN coating interaction leading to the formation of hydrides with low-temperature decomposition.

CONCLUSIONS

1. A CrN coating, deposited from the filtered vacuum-arc plasma stream, has a cubic crystalline structure with an axial texture and crystallite size ≈ 11 nm. The methods of X-ray diffraction analysis did not reveal a new phase formation at a deuterium irradiation dose of ~ $1.5 \cdot 10^{18}$ D/cm^2.

2. The atomic-force microscopy data show that deuterium irradiation up to a dose of ≥ $1 \cdot 10^{18}$ D/cm^2 causes the formation on the coating surface of blisters having a characteristic height of ~ 150…200 nm which cover about 30% of the irradiated coating surface.

3. A CrN coating is characterized by a high hardness of ~ 30 GPa. The deuterium ion implantation results in the hardness decrease. In the range of doses from $5 \cdot 10^{16}$ to $5 \cdot 10^{17}$ D/cm^2 the hardness decreases by 10…15%. The nanohardness decrease to ~ 50% occurs at doses > $5 \cdot 10^{17}$ D/cm^2, most likely, due to the blister formation.

4. A maximum deuterium concentration in the implantation layer of the CrN coating is ~ 2 D atoms per 1 Cr atom after irradiation at a dose of ~ $2.5 \cdot 10^{18}$ D/cm^2.

5. In the deuterium desorption spectrum of CrN coatings only a single temperature peak at temperature of 1120 K is observed throughout the range of implanted doses being investigated that indicates to a lack of hydride formation.

The obtained investigation results show that significant changes in the CrN coatings deposited by the vacuum-arc method take place at implanted deuterium doses of > $5 \cdot 10^{17}$ D/cm^2.

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ВЛИЯНИЕ ДОЗЫ ИМПЛАНТИРОВАННОГО ДЕЙТЕРИЯ НА СВОЙСТВА ПОКРЫТИЙ CrN

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Методами рентгеноструктурного анализа, атомно-силовой микроскопии, наноиндентирования и термодесорбционной спектроскопии исследовано влияния дозы ($5\cdot10^{16}$…$1{,}5\cdot10^{18}$ D/см^2) имплантированного дейтерия с энергией 24 кэВ на структуру, морфологию поверхности и механические свойства вакуумно-дуговых покрытий CrN. Имплантированный дейтерий в интервале доз $5\cdot10^{16}$…$5\cdot10^{17}$ D/см^2 приводит к уменьшению на 10…15% нанотвердости и модуля упругости покрытий. Облучение дозами $\geq 1\cdot10^{18}$ D/см^2 вызывает резкое снижение нанотвердости покрытий из-за формирования блистеров, которые занимают около 30% поверхности покрытия CrN. Имплантация дейтерия не приводит к образованию новых фаз в покрытии CrN.

ВПЛИВ ДОЗИ ІМПЛАНТОВАНОГО ДЕЙТЕРІЮ НА ВЛАСТИВОСТІ ПОКРИТТІВ CrN

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Методами рентгеноструктурного аналізу, атомно-силової мікроскопії, наноіндентування і термодесорбційної спектроскопії досліджено вплив дози ($5\cdot10^{16}$…$1{,}5\cdot10^{18}$ D/см^2) імплантованого дейтерію з енергією 24 кеВ на структуру, морфологію поверхні та механічні властивості вакуумно-дугових покриттів CrN. Імплантований дейтерій в інтервалі доз $5\cdot10^{16}$…$1{,}5\cdot10^{17}$ D/см^2 призводить до зменшення на 10…15% нанотвердості і модуля пружності покриттів. Опромінення дозами $\geq 1\cdot10^{18}$ D/см^2 викликає різке зниження нанотвердості покриттів через формування блістерів, які займають близько 30% поверхні покриття CrN. Імплантація дейтерію не призводить до утворення нових фаз у покритті CrN.