

ELEMENTAL AND PHASE ANALYSIS OF NANOCOMPOSITE COATINGS ON BASIS Ti-Hf-Si-N SYSTEM RECEIVED BY THE VACUUM-ARC DEPOSITION METHOD

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Coatings on the basis Ti-Hf-Si-N system were synthesized by vacuum-arc deposition method from the uniflow and separated ion-plasma flow. The morphology, elemental and phase composition of coatings were investigated. The dependence of the characteristics of the coating from the physical and technological parameters of deposition was installed. The two-phase structure of the coating: a substitutional solution (Ti, Hf)N and quasicrystalline silicium nitride α -Si₃N₄ was determined. The factors that determine the compressive stresses in the coatings were considered.

Keywords: vacuum-arc deposition method, nanocomposite coatings, nitrides, alloys, superhard, refractory metals.

Методом вакуумно-дугового осадження із прямого і сепарованого іонно-плазменного потоку синтезовані покриття на основі системи Ti-Hf-Si-N. Досліджено морфологію, визначено елементний і фазовий склад покриттів. Встановлено залежність характеристик покриттів від фізико-технологічних параметрів осадження. Визначено двофазна структура покриття: твердий розчин заміщення (Ti, Hf)N і квазіаморфний силіконітрид α -Si₃N₄. Розглянуто фактори, що визначають стискаючі напруження в покриттях.

Ключевые слова: вакуумно-дуговой метод осадження, нанокompозитные покрытия, нитриды, сплавы, сверхтвердость, тугоплавкие металлы.

Методом вакуумно-дугового осадження з прямого і сепарованого іонно-плазмового потоку синтезовані покриття на основі системи Ti-Hf-Si-N. Досліджено морфологію, визначено елементний і фазовий склад покриттів. Встановлено залежність характеристик покриттів від фізико-технологічних параметрів осадження. Визначено двофазна структура покриття: твердий розчин заміщення (Ti, Hf)N і квазіаморфний силіконітрид α -Si₃N₄. Розглянуто чинники, що визначають стискаючі напруження у покриттях.

Ключові слова: вакуумно-дуговий метод осадження, надтвердість, нанокompозитні покриття, нитриди, сплави, тугоплавкі метали.

INTRODUCTION

The development of the new nanostructural coating with high hardness (> 40 GPa) and thermal stability (> 1200 °C) is the actual problem of the modern material science. According to the previous experimental results it can be considered that not only grains size has strong influence on properties of the solid but also structural states of interfaces (grains boundary). As the quantity of atoms at grains bound-

ary reaches about 30 – 50% properties of the material are strongly depend on condition of the grains boundary: gap of the border band (in this band lattice parameter deviate from standard value), disorientation of the grains and interfaces, concentration of the defects at boundary and value of the free volume.

Nanocrystalline materials that contain nanosized crystallite along with rather extensive and partially

disordered boundaries structure have new properties by comparison with the large-grained materials.

Obtaining of nanocomposite coatings by ion-plasma deposition technology requires deposition on the substrate of multi-component flows. In most cases the synthesis of coatings is realized by layering or simultaneous deposition on the substrate in an atmosphere of reaction gases of flows from two sources: ion, magnetron, vacuum-arc and a combination thereof. Such methods of deposition require the of non-standard equipment. Therefore interest are the investigations in which nanocomposites receive on the traditional plants with one cathode (evaporated target) containing the necessary components in the desired ratio. Materials for evaporation receive powder technologies [1, 2] or electron beam remelting [3]. The paper presents the results of the development of the new type of Ti-Hf-Si-N-based superhard nanostructured coatings and analysis of their elemental composition, the surface morphology and structural-phase state depending on the physical and technological conditions of their production.

ELEMENTAL COMPOSITION AND SURFACE MORPHOLOGY OF NANOCOMPOSITE COATINGS BASED Ti, Hf, Si AND N RECEIVED BY VACUUM-ARC DEPOSITION METHOD

Coatings on the basis Ti-Hf-Si-N system thickness of 1.5 μm were formed on the polished surface of the samples (steel 45) with a diameter of 10 mm and a height of 3 mm by vacuum-arc deposition using a pulsed HF voltage by sputtering cathode Ti+Hf+Si in the molecular nitrogen medium. The coatings were synthesized from the uniflow ion-plasma flow and with the use of flow separation. A bias potential was varied in the range $-100 \text{ V} \div -200 \text{ V}$ and was applied to the substrate from a HF generator with impulses of damped oscillations of $\leq 1 \text{ MHz}$ frequency.

The elemental composition of coatings obtained by vacuum-arc deposition was analyzed using EDS-energy dispersion method and Rutherford back scattering of $^4\text{He}^+$ ions. These methods are complementary. Research results of the elemental composition of coatings are shown in fig. 1 and fig. 2.

Spectrum analysis of Ti-Hf-Si-N coating indicates the absence of oxygen, nitrogen is 48.64 at.%,

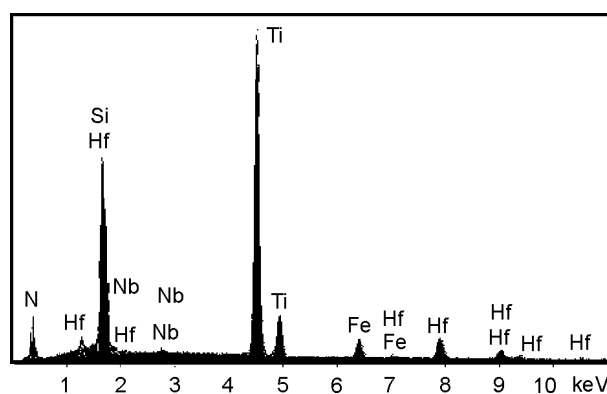


Fig. 1. EDS spectrum of coatings on the basis Ti-Hf-Si-N obtained by vacuum-arc deposition: $U_{HF} = -100 \text{ V}$, $P = 0.3 \text{ Pa}$.

34.35 at.% titanium, 4.49 at.% silicon, 7.83 at.% hafnium (fig. 1). The presence of niobium 0.24 at.% was found that is connected with purity hafnium and the presence of 4.45 at.% iron. The analysis carried out by Rutherford back scattering of $^4\text{He}^+$ ions (fig. 2) shows that the coating composition is somewhat different from the composition obtained previous microanalysis. It is well known that RBS method is an etalon for the determination of concentration of the elements with high atomic number and films thickness.

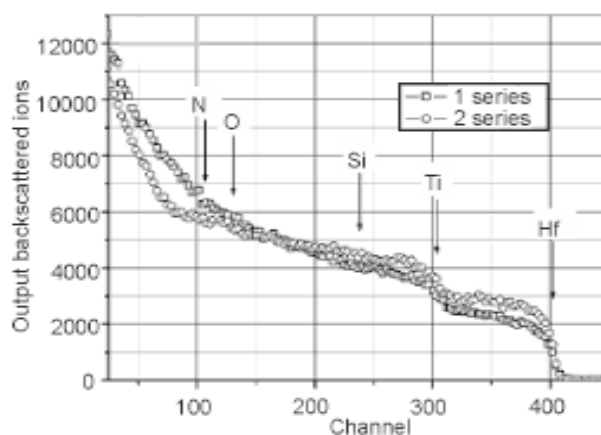


Fig. 2. RBS spectra of He^+ with 1.3 MeV energy obtained from steel sample with Ti-Hf-Si-N coating: curve 1 – potential -100 V , $P = 0.3 \text{ Pa}$, curve 2 – potential -200 V , $P = 0.7 \text{ Pa}$.

It is well known that RBS method is nondestructive and this is its advantage. At the same time SIMS is more sensitive method (threshold of sensitivity is about 10^{-6} at.%). Comparison of results obtained by the RBS, SIMS and SIMS with a glow discharge methods allows obtaining of more reliable data of the elemental composition on the depth layer synthesized coatings. The application of these methods has made it possible to analyze composition along

the film cross-section from the surface to the film-substrate interface including uncontrolled impurities O and C appearing from the residual atmosphere in the chamber in which the deposition of films carried out. In fig. 3a, b the profiles of the elements included in the coating composition Ti-Hf-Si-N, obtained by using the RBS (including uncontrolled impurity) are shown.

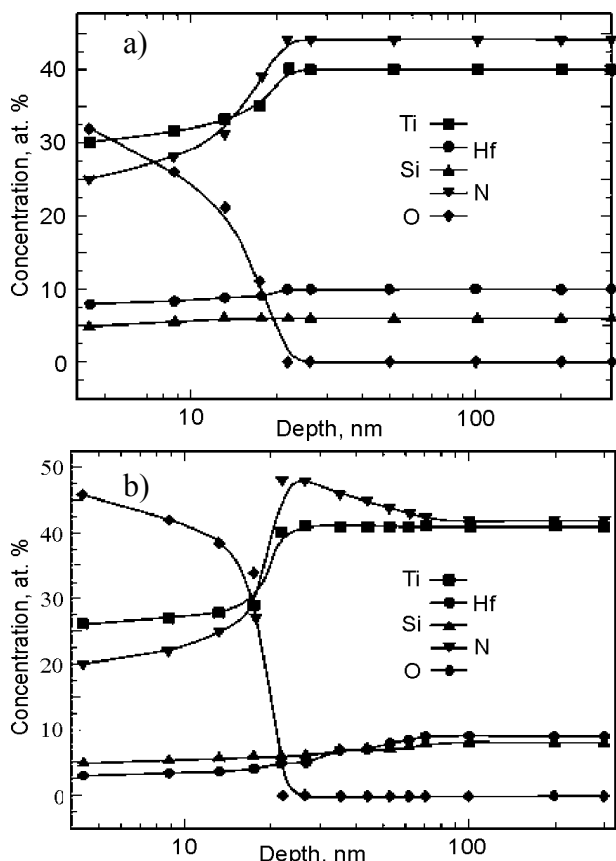


Fig. 3. The depth profiles of elements of the Ti-Hf-Si-N coating obtained from RBS spectrums considering that atomic density of layer is close to atomic density of titanium nitride.

Presented profiles are built without the results of SIMS analysis. As is seen from the presented results the oxygen in the coating is only in the surface layer of thickness up to $10 \div 12$ nm when coating thickness is above a micron. The results of SIMS analysis obtained for the same coating are presented in fig. 4.

For samples with Ti-Hf-Si-N coating ($P = 0.3$ Pa, $U_{HF} = 100$ V) the first series is received (fig. 2) with the following stoichiometry of film: Ti – 40.0 at.%; Hf – 9.0 at.%; Si – 8.0 at.%; N – 43.0 at.% when the film thickness is $(1 \pm 0.012) \mu\text{m}$ (fig. 4).

By increasing impulse bias potential up to 200 V and changing the nitrogen pressure in the chamber

to 0.7 Pa the second series of samples with films based on Ti-Hf-Si-N (Ti – 28.0 at.%; Hf – 18.0 at.%; Si – 9.0 at.%; N – 45.0 at.%) is received.

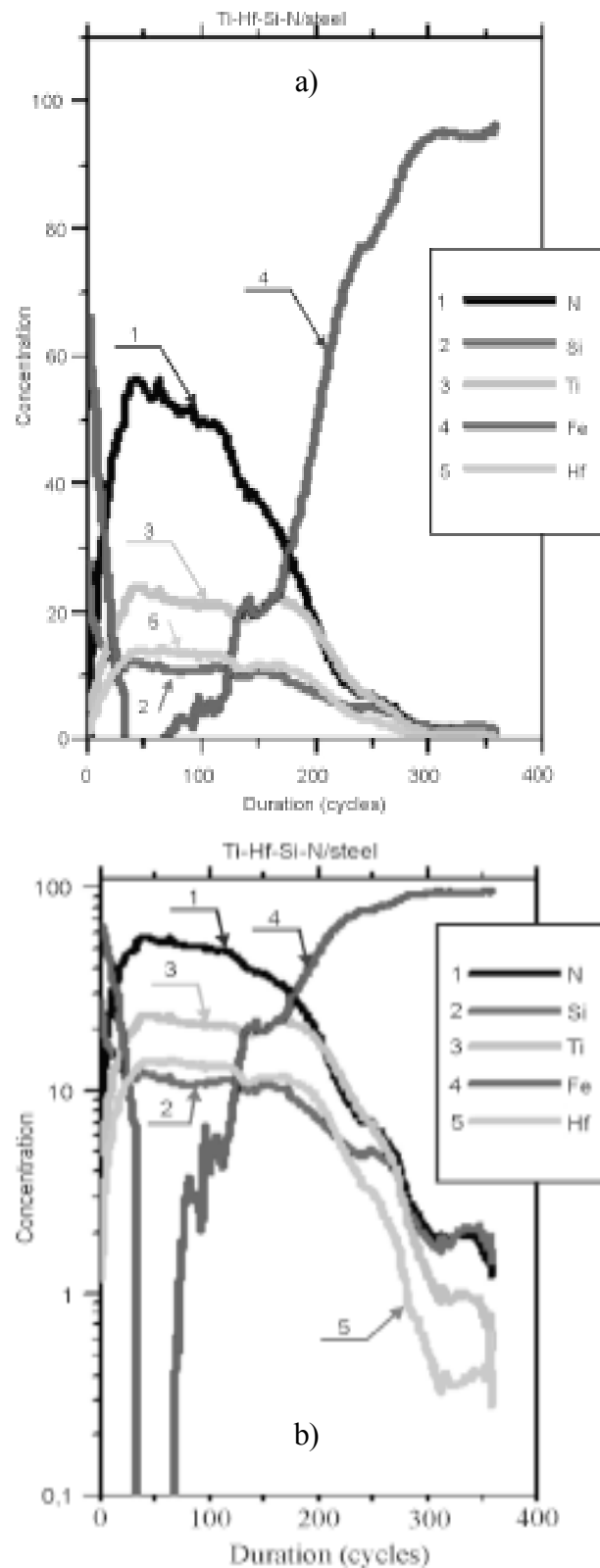


Fig. 4. SIMS analysis profiles of elements in the Ti-Hf-Si-N coating obtained under concentrations of Ti – 40.0 at.%, Hf – 9.0 at.%, Si – 8.0 at.%, N – 46.0 at.% (first series): a) – at.%, standard scale of concentration; b) – logarithmic scale of concentration.

The presented experimental result explained by an increase pressure working gas (nitrogen) which causes smooth decrease of silicon concentration caused by that the average kinetic energy of emitted from the cathode of titanium ions (122 eV) is higher than that of silicon ions (97 eV) on the one hand [4]. Thus as a result of collisions with gas targets there is “impoverishment” of the plasma flow less energetic particles. On the other hand to this process also contributes the selective silicon sputtering by the incident particle flux [5]. With further increase of the nitrogen pressure is rather steep increase of the Si content in the coating. That is increase of value of the potential bias on the substrate affects the energy value of condensed particles and the concentration of silicon.

Research of the surface morphology of Ti-Hf-Si-N coatings at concentration Ti – 40.0 at.%; Hf – 9.0 at.%; Si – 8.0 at.%; N – 43.0 at.% (thickness 4.0 μm) showed that the surface coating is present drop component (fig. 5a). It is known [6] that the coatings deposited at different pressures of nitrogen have different level of internal stress: high microhardness is usually accompanied by higher internal stresses that actively relax (fig. 5b).

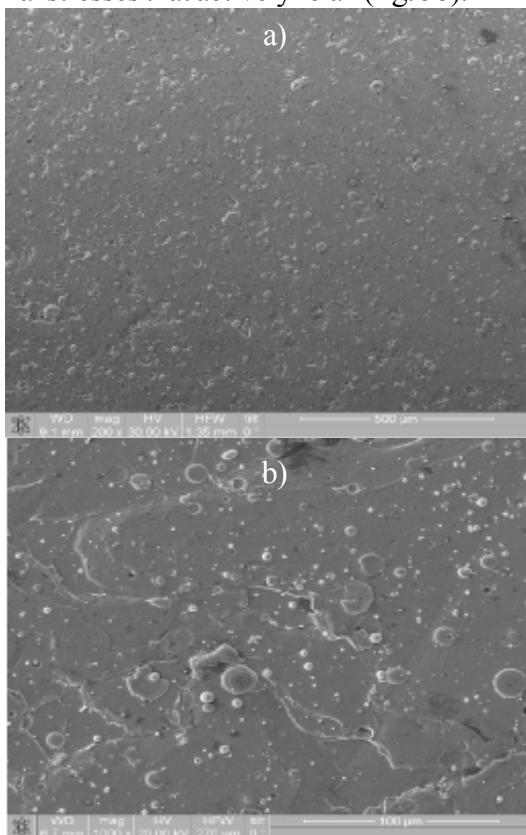


Fig. 5. The surface morphology of the coatings obtained at a partial pressure of nitrogen $P_N = 0.3$ Pa: a) – the coating surface structure; b) – areas with delamination interlayer of coating.

A comparison of the surface morphology of the samples obtained at different partial pressures of nitrogen indicates that in comparison with coatings obtained at a nitrogen pressure $P = 0.3$ Pa the drop component at $P = 0.7$ Pa decreases (fig. 6). Thus increasing the pressure of the reaction nitrogen medium leads to a decrease in the number and size of macroparticles that is to reduce the roughness of coatings.

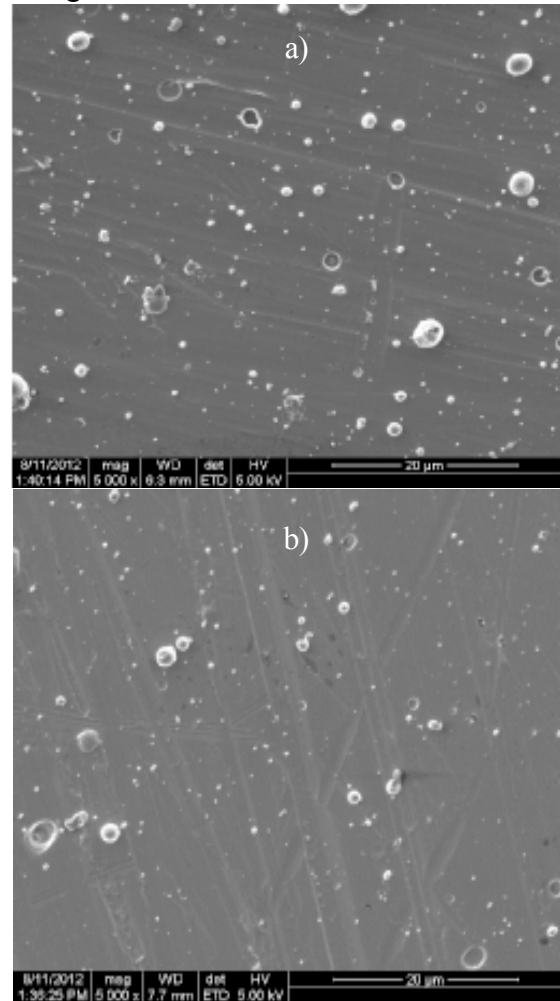


Fig. 6. The surface topography of coatings based on Ti-Hf-Si-N: a) $P = 0.3$ Pa, $U_{HF} = -200$ V; b) – $P = 0.7$ Pa, $U_{HF} = -200$ V.

Presence in the vacuum chamber of active reaction gases determines the formation with the vaporized material of refractory compounds. In our case the nitrides in the surface layer of the coating are formed that determines its thermalphysical properties.

The above results show that in the case of low thermal conductivity of the sputtered material which is characteristic as titanium and hafnium ($\lambda_{400}(\text{Ti}) = 20 \text{ W}/(\text{m}\cdot\text{K})$, $\lambda_{400}(\text{Hf}) = 22 \text{ W}/(\text{m}\cdot\text{K})$) [7] the gen-

eration of macroparticles is increased that leads to high density of drop fraction in coatings.

The use of separated ion-plasma flow in order to minimize the drop component in the coating [8, 9] offers great opportunities for deposition coatings on precision machine parts. The separation of the flow reduces the amount of macroparticles that gives the coated product high physical and mechanical properties thereby improving its functional characteristics.

Thus it can be concluded that by changing the physical and technological parameters of deposition (pulsed HF bias potential, the partial pressure of nitrogen) can affect the surface morphology during the formation of coatings and change percentage content of the coating components.

PHASE COMPOSITION OF NANOCOMPOSITE COATINGS ON BASIS Ti, Hf, Si and N

Prior to analysis of XRD data it should be noted that for better understanding of processes occurred at near-surface region during deposition it is necessary to compare formation heats of the probable nitrides.

According to [10] standard heats of formation of such nitrides are next: $\Delta H_{298}(\text{HfN}) = -369.3 \text{ kJ/mole}$, $\Delta H_{298}(\text{TiN}) = -336.6 \text{ kJ/mole}$, $\Delta H_{298}(\text{Si}_3\text{N}_4) = -738.1 \text{ kJ/mole}$.

I.e. values of the formation heats are quite large and negative. It indicates high probability of those systems formation during all stages of transport of the material from target to substrate. In addition, proximity of formation heats for TiN and HfN establish conditions for formation of the sufficiently homogenous (Ti, Hf)N solid solution.

The XRD-analysis revealed the formation of a two-phase system in coatings. This system was determined as the substitutional solid solution (Ti, Hf)N because diffractions peaks of this phase are located

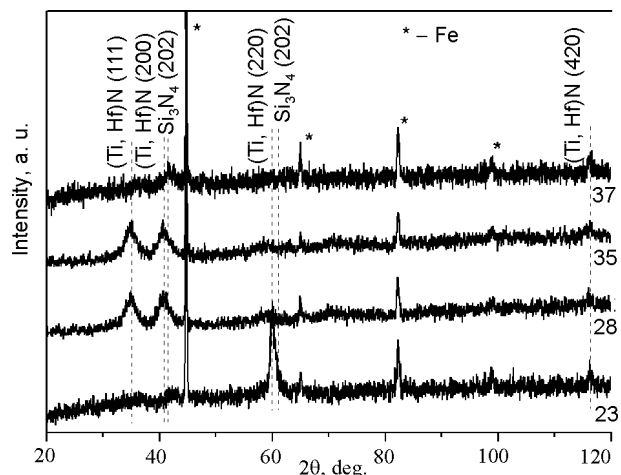


Fig. 7. XRD spectra of the coatings deposited on a steel substrate at regimes: (23) –100 V, magnetic separated; (28) –200 V, uniflow beam; (35) – 100 V, uniflow beam; (37) –200 V, magnetic separated.

between peaks related to mononitrides TiN (JCPDS 38-1420) and HfN (JCPDS 33-0592). The diffused peaks with less intensity at 2θ values from 40° to 60° are related to the $\alpha\text{-Si}_3\text{N}_4$ phase (fig. 7).

The results of the effect of technological parameters on the structural state of coatings based system Ti-Hf-Si-N are represented in tabl. 1.

The analysis of the XRD data (see tabl. 1) shows that by the characteristic structural features the coatings obtained from the target of the same composition are very different depending on the conditions of formation (the uniflow plasma flow or separated) and technological deposition parameters which is manifested in change of the average crystallite size and the amount of Hf in the coating.

From X-ray diffraction spectra analysis (fig. 7) follows that at uniflow regime of the plasma flow non-textured polycrystalline coatings with significant relative intensity of the peaks are formed. Rather high intensity of the peaks at XRD-patterns of (Ti, Hf)N solid solutions is attributed to relatively large

Table 1

Characteristics of the investigated Ti-Hf-Si-N coatings

№	Nitrogen pressure in chamber (P), Pa	Potential bias, V	Lattice parameter, nm	Average size of crystallite, nm	Hf content in solid solution (Ti, Hf)N, at. % ¹⁾	Notes
23	0.7	-200	0,4290	6.7	19	separation
37	0.6	-100	0,4337	5.0	33	separation
31	0.3	-200	0,4370	3.9	45	separation
35	0.6 – 0.7	-100	0,4337	4.3	69	uniflow beam
28	0.6 – 0.7	-200	0,4430	4.0	65	uniflow beam

¹⁾ Calculation was carried out according to Vegard rule from period values of solid solution (the influence of macrostresses on the change of diffraction lines was not taken into account).

concentration of hafnium, which has larger reflection value than titanium.

In case of beam separation the coatings have different texturation. At low substrate potential (100 V) coatings have [110] texture, and coatings consist of textured and non-textured crystallites. The volume content of textured crystallites is about 40% of total amount of the crystallites and their lattice parameter enlarged in comparison to non-textured crystallites. The most likely reason for the increase the lattice period can be the inhomogeneous distribution of the hafnium atoms in coating (mainly in the lattice sites of the textured crystallites).

At the same time the formation texture leads to increasing of the average grains size of the crystallites along the direction of incidence the film-forming particles (perpendicular to the growing surface). For example, in non-textured fraction of the crystallites the average grains size is about 6.7 nm, whereas in textured crystallites the value of the average grains size is substantially more, namely 10.6 nm.

In the case of increasing the voltage to 200 V the schemes with separation of ion-plasma flow were used. In this case the coatings with reduced average crystallite size (up to 5 nm) were formed and significantly decreased the fraction of textured crystallites (less than 20% vol.). In this case texture axis was defined as {001}.

Note that the increase of the accelerating voltage from 100 to 200 V (i. e. the energy increase of the plasma flow) is characterized by identical values the lattice period of the crystallites for both textured and non-textured fractions. However the lattice period in this case exceeds period for non-textured fraction when applying a low potential to the substrate and is 0.4337 nm.

According to Vegard law the value of the lattice period corresponds to 33.0 at.% of Hf in metallic (Hf, Ti)N solid solutions of the nitride phase (the reference data of the lattice periods of a TiN = 0.424173 nm (JCPDS 38-1420) and a HfN = 0.452534 nm (JCPDS 33-0592) were used).

However, as a rule, the compressive stresses in coatings caused the decrease of the angles of corresponding diffraction peaks during $\psi \div 2\psi$ scan hence calculated values of lattice period can be overestimated. As a result inaccuracy of the calculation of Hf concentration in solid solutions can achieve about 5 \div 10 at.%. Therefore presented results can be considered as estimation of upper limit of the Hf concentration in solid solution.

If we compare the phase-structural state of the coatings produced at high pressure nitrogen ($P_N = 0.3$ Pa, the bias voltage $U_{HF} = -100$ V) with the largest lattice period in a relaxed state $a = 0.4437$ nm (the maximum value of the lattice period studied in this paper coatings apparently determined by the highest content of hafnium atoms and high pressure) then it is a significant change in the phase-structural state of the coating.

All above mentioned results are related to samples obtained at typical pressure 0.6 \div 0.7 Pa. In a case of coating deposition at -200 V substrate potential in mode of separation (set of samples 31) the decreasing of pressure up to 0.3 Pa caused the increase of relative content of heavy Hf atoms in coatings (tabl. 1). In addition, the average grains size of the crystallites decreased with pressure.

Observed in this case the effects can be associated with an increase in of the radiation factor with decreasing operating pressures. Indeed, the decrease in pressure should be accompanied by decrease of the probability of energy loss of atoms during collision between targets and substrate. Thus atoms at substrates have relatively high energy which can promote secondary sputtering and radiation defect formation. So, secondary sputtering leads to decrease of relative content of heavy Hf atoms, while radiation defect formation provide the decrease of grain size with the increase of nucleus amount.

The coatings obtained under the typical pressure 0.6 \div 0.7 Pa in case of non-separated beam (direct-flow mode) have considerably larger lattice parameter; it can be explained by the high concentration of heavy Hf atoms (tabl. 1). Apparently, the more intensive direct-flow mode leads to the increase of the nucleus density and hence to the decrease of average grain size. In addition, more pronounced decrease of the grains size is caused by the higher substrate potential 200 V. It is obviously because increasing of radiation factor leads to the dispersion of structure [12].

In the case of pulsed HF stimulation mikrodeformation of the crystallites is less. In this case compressive macrodeformation defined according to the X-ray tensometry from “ $a \cdot \sin^2\psi$ ”-graph is connected in such condensates with manifestation of “atomic peening” effect [11]. Crystallites of (Ti, Hf) N solid solution in the coating based on Ti, Hf, Si and N are under action of compressive elastic macrostresses of system “condensate-substrate” (fig. 8).

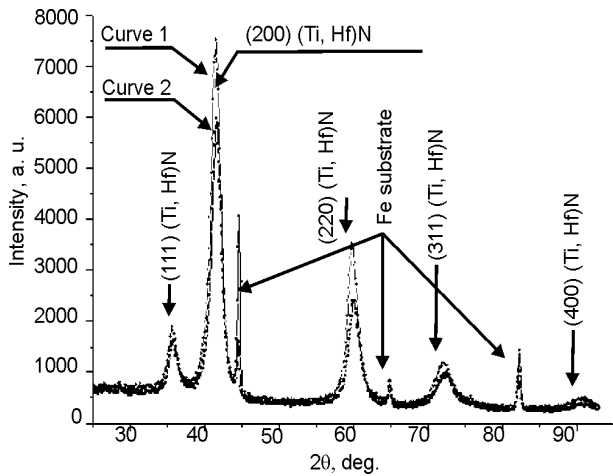


Fig. 8. X-ray picture of coating based on Ti-Hf-Si-N ($P=0.7$ Pa): 1 – $U=-100$ V, $\langle \epsilon \rangle = -1.9\%$, $a = 0.435$ nm; 2 – $U=-200$ V, $\langle \epsilon \rangle = -1.6\%$, $a = 0.431$ nm.

The research of Ti-Hf-Si-N coating in a transmission electron microscope showed that the order of the size of nanograins is corresponds to the data XRD-analysis, namely, ultradispersive structure with an average size of the crystallites within $5 \div 10$ nm is formed. Dark-field image of such a structure is shown in fig. 9.

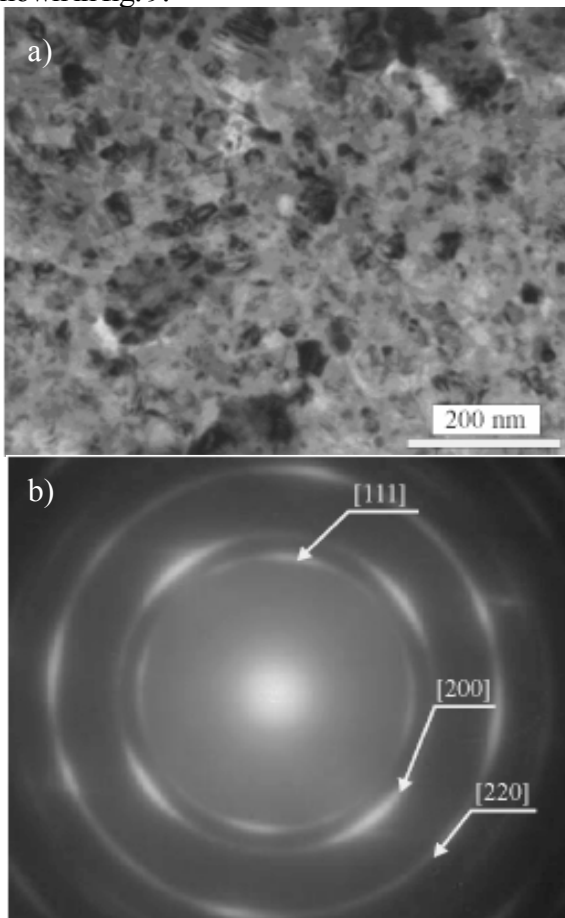


Fig. 9. Photographs of the structure sections of the nanocomposite coating based on the Ti-Hf-Si-N solid solution obtained using a transmission electron microscope: a) – surface structure, b) – dark-field image of nanostructure (transmission electron microscope JOEL 2010 F).

Differences in phase state and structure of thin coatings deposited on the steel silicon substrates are not found.

Great interest is the information about the features of deformation in relation to multicomponent coatings with nanocrystalline structure. Scanning microscope Quanta 600 is used for fractographic studies. The cracks surface passing through pricks inflicted by the indentation (at loads $F = 0.1$ N and $F = 0.25$ N) is studied. The appearance of fracture surfaces of Ti-Hf-Si-N system thickness of 1.2 microns is shown in fig. 10. Measurements were carried out device 402 MVD (Instron) at loads $F = 0.1$ N and $F = 0.25$ N.

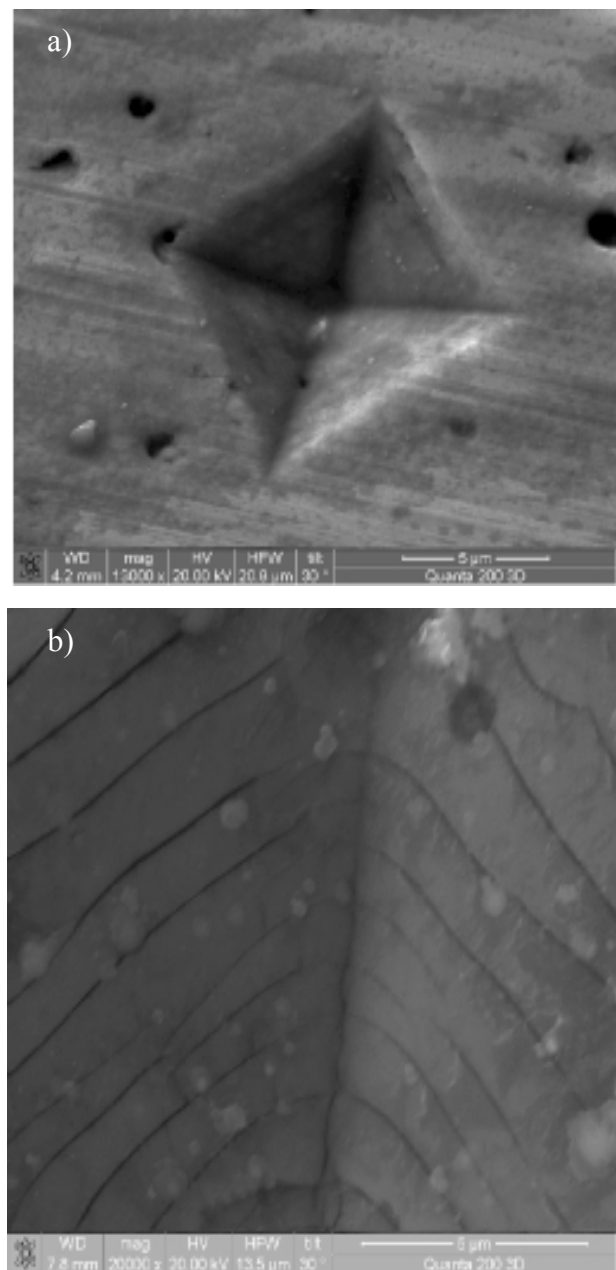


Fig. 10. Fraktograms of crack of Ti-Hf-Si-N film: a) – $F = 0.1$ N; b) – $F = 0.25$ N.

Looking at the nature of the impression and the fracture plane the deformation is sufficiently uniform. When using large loads $F > 0.1$ N at indentation on the surface of the impression ring and radial cracks are fixed (Fig. 10b) which can be observed when measuring the hardness of fragile bodies, but the formation of steps shift was not observed. Features of deformation of thin coatings based on Ti-Hf-Si-N can be related with the structure. The deformation is performed by sliding relative to each other of individual columnar structure elements. For the investigated coatings typically clearly expressed columnar structure is characteristic and it is obvious homogeneous indenter sliding along the columns. Can also note that the investigated objects by their nature are fragile and by nature the fracture are intercrystalline [13, 14].

CONCLUSIONS

1. The elemental composition of coatings obtained by vacuum-arc deposition with the use of pulsed HF voltage of Ti-Hf-Si-N system depends on the nitrogen pressure and HF potential applied to the substrate. The coatings are formed on the basis of a two-phase system: the first phase of the solid solution (Ti, Hf)N and quasiamorphous phases δ -Si₃N₄ and HfSi₂-nc.
2. The size of the nanocrystallites of the solid solution of nanostructured coating (Ti, Hf)N is changed from 3.8 to 10.6 nm. Interlayer of δ -Si₃N₄ surrounding nanograins of the solid solution has a thickness $\sim 0.8 \div 1.2$ nm.
3. Crystallites of the solid solution (Ti, Hf)N in the coating based on Ti-Hf-Si-N are under action of compressive elastic macrostresses of system "condensate-substrate". Compressive stresses in the plane of condensate growth determine the development of compression deformation of the crystal lattice reaching a value of 1.9%.
4. The obtained multicomponent films have clearly expressed columnar structure which is clearly seen at the deformation. It is shown that the deformation of films Ti-Hf-Si-N at indentation is performed by sliding columnar elements of the structure parallel to the applied load, i.e. the grain-boundary sliding is the main deformation mechanism.
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