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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

**INVESTIGATION OF THE EFFECT OF PROTON
IRRADIATION ON THE SURFACE MORPHOLOGY AND
STRUCTURAL PROPERTIES OF CARBONITRIDE-BASED
COMPOSITES**

Speciality: 2225.01 – “Radiation materials science”

Field of science: Physics

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The work was performed at the "Radiation Detectors and Energy Conversions" Laboratory of the Institute of Physics Public Legal Entity of the Ministry of Science and Education of the Republic of Azerbaijan.

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GENERAL DESCRIPTION OF THE WORK

Relevance and degree of development of the topic: The rapid growth of technological innovations over the past decade has created broad opportunities for the development and study of new types of materials in industry. Among the material classes currently used, metal carbides and nitrides, nanocarbon composites, and carbonitride-based composites have attracted considerable scientific interest. Extending the service life of materials used in space and nuclear technologies under various external factors is regarded as one of the most important challenges of recent years. A number of nitride-based coating materials, including TiN, ZrN, TiSiN, ZrSiN, ZrTiN, ZrNbN, and ZrTiSiN, have attracted significant research interest due to their high mechanical and elastic properties and resistance to radiation and corrosion. Owing to their thermal stability at elevated temperatures and the formation of only a minimal oxide layer on the surface, nitrides have become important research materials across many industrial fields.

Carbonitride-based coating materials have demonstrated high oxidation resistance, while distinctive changes in their electrical properties have been identified depending on temperature. Producing materials in layered structures provides substantial opportunities for the targeted control of their properties. The use of nuclear-technology equipment and the development of the modern space industry have created strong demand for advanced coating materials. Ion-implantation studies on coating materials indicate that carbonitrides are among the most promising representatives of this class because of their high radiation resistance. By considering the electron-concentration characteristics of the constituent elements and controlling the C ratio, these materials can be classified and tailored for different applications.

The radiation resistance of carbonitrides under proton (H^+) and alpha-particle (He^+) irradiation at different energies, as well as under ionizing gamma radiation, has not yet been studied extensively. The use of different irradiation sources can provide detailed information on changes occurring in the surface morphology and structural characteristics of carbonitride coating materials, as well as on their

behavior and service life under irradiation conditions. Considering the high mechanical and elastic properties of carbonitride materials, the investigation of microstructural and structural changes induced by high-energy proton irradiation was defined as one of the principal scientific objectives of the dissertation and as an important aspect of the relevance of the research topic.

In addition, in order to ensure the reliability of the experimental results, the corrosion resistance and electrochemical behavior of the materials in a NaCl medium were investigated. In a corrosive environment, the diffusion of Cl^- anions from the surface layer into the bulk of the material may considerably reduce the protective properties of the coating. Taking these effects into account, the penetration of the electrolyte into carbonitride coatings makes it possible to investigate the mechanism responsible for blocking Cl^- anions at the coating–electrolyte interfaces. To improve the corrosion resistance of carbonitride coatings, the test solution, target-to-substrate distance, applied target power, substrate bias voltage, and selected chemical composition are considered important process parameters.

The solutions proposed for the problems addressed within the scope of the dissertation, together with the published scientific works and results presented at international conferences, demonstrate that the research topic has been comprehensively investigated.

Object and subject of the research: High-purity TiNbCN (Ti–58.92%, Nb–28.18%, C–4.62%, and N–8.28%), TiSiCN (Ti–63.02%, Si–22.76%, C–3.99%, and N–10.23%), TiZrCN (Ti–61.37%, Zr–26.35%, C–3.85%, and N–9.49%), TiZrSiCN (Ti–38.92%, Zr–27.11%, Si–22.07%, C–3.62%, and N–8.28%), and TiZrNbCN (Ti–35.58%, Zr–26.24%, Nb–23.00%, C–3.78%, and N–10.04%) composites were selected as the objects of the research. The subject of the research was the investigation of changes occurring in the surface morphology and structural characteristics of carbonitride composites as a result of proton irradiation and chemical corrosion.

Aim and objectives of the research: The aim of this research was to determine the regularities governing the effects of proton irradiation at different energies on the surface morphology and

structural characteristics of carbonitride materials with different compositions, as well as to investigate their electrochemical corrosion behavior. To achieve this aim, the following objectives were defined:

1. Preparation of TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN coating materials on Ti₆Al₄V substrates;
2. Investigation of neutron activation under irradiation with 1 MeV fast neutrons;
3. Investigation of the processes occurring in the structure and surface morphology of coating materials irradiated with protons of different energies;
4. Investigation of the processes occurring in the structure and surface morphology of TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN coating materials in a corrosive environment.

Investigation methods: Carbonitride coating materials with different compositions and a high degree of purity were prepared by the magnetron sputtering method. Their crystal structure, lattice parameters, symmetry type, free phases formed on the surface, and interatomic distances were determined at room temperature using a Rigaku SmartLab X-ray diffractometer with Cu K α radiation at 40 kV and 40 mA, a wavelength of 1.5405 Å, a diffraction-angle range of $30^\circ < 2\theta < 80^\circ$, and an incidence angle of 3° .

Neutron activation analysis was performed by irradiating the materials with fast neutrons, and the formation of short- and long-lived radioactive isotopes emitting gamma radiation as a result of neutron interactions with the nuclei of the constituent atoms was investigated. The elemental composition of the samples was determined by energy-dispersive X-ray spectroscopy (EDS). Scanning electron microscopy (SEM) was used to investigate the surface morphology of the materials at high magnifications, determine the surface topography, and evaluate the size and distribution of surface particles, as well as the chemical composition of the samples.

Irradiation of the materials with positively charged hydrogen ions (H⁺) at energies of 15 and 200 keV and 1.2 and 2.0 MeV was carried out at the Laboratory of Neutron Physics of the Joint Institute for Nuclear Research, an international intergovernmental

organization. Irradiation with H^+ ions is widely used in materials science, nuclear physics, semiconductor research, space technology, hydrogen energy, biological studies, and various other fields. Complex radiation-induced processes, including modification of surface properties, formation of surface defects, and gas blistering, are among the characteristic effects of this type of irradiation.

Molecular dynamics (MD) simulation was employed as a computational method for studying the physical motion and interactions of atoms and molecules under different conditions and during various processes. By solving the equations of motion for atoms within an appropriate atomistic framework, MD simulations provide information on the dynamic behavior of systems at atomic and molecular scales. Molecular dynamics simulation techniques are widely applied in radiation materials science, biophysics, chemistry, and nanotechnology to investigate phenomena that are difficult or impossible to examine experimentally.

The corrosion behavior of the materials was investigated using open-circuit potential and potentiodynamic polarization methods. Over an extended potential range, the active and passive behavior of the material surface, breakdown potential, localized corrosion processes, and surface roughness characteristics were evaluated.

Main provisions submitted for defense:

1. Preparation of TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN composites as a function of the C ratio, and investigation of their structural characteristics and surface morphology;
2. Performance of neutron activation analysis (NAA) for the identification of long-lived radioactive isotopes in composites irradiated with fast neutrons of 1.0 MeV energy and a flux density of $1.65 \times 10^7 \text{ n/(s}\cdot\text{cm}^2)$;
3. Investigation of the processes occurring in the structure and surface morphology of the materials after proton (H^+) irradiation at different energies and flux densities;
4. Investigation of blistering, amorphization, pinhole formation, and the absorption mechanism associated with blisters formed on the surfaces of the composites under proton irradiation;

5. Determination of the formation time of pinholes and blisters by the molecular dynamics method, as well as determination of the radius of radial temperature distribution;
6. Investigation of the processes occurring in the structural characteristics of the composites in a corrosive environment, the formation of new phases in the surface morphology, and the distribution of crystalline particles;

Scientific novelty of the research:

1. It was established that irradiation with 1.0 MeV fast neutrons does not result in the formation of long-lived gamma-emitting radioisotopes in the investigated samples;
2. After proton irradiation at different energies, no significant blister formation and absorption processes in the surface morphology of the carbonitride composites, and no pronounced amorphization of their crystalline structure, were observed;
3. Molecular dynamics simulations were used to determine the formation time of pinholes generated on the surfaces of the composites after proton irradiation, as well as the magnitude and duration of the radial temperature distribution on the material surface;
4. The sample exhibiting the highest resistance to electrochemical corrosion was identified as a function of the C ratio in the composition of the composites;
5. It was established that the new phases formed in the crystal structure of the composites under corrosive conditions also exhibit corrosion-resistant properties;

Theoretical and practical significance of the research:

Based on the results obtained in the dissertation, carbonitride coating materials synthesized with different C ratios exhibit high resistance to irradiation with high-energy proton ions. Proton irradiation produces characteristic surface patterning along different directions of the material surface. From an applied perspective, the newly formed surface structures demonstrate high radiation resistance, while the dimensions of pinholes and blisters remain minimal compared with those reported for other coating materials, where such

features may begin at approximately 120 nm. This behavior provides favorable conditions for the long-term use of these materials in radiation environments. In addition, no blister coalescence process was observed; in other words, small blisters did not merge to form larger blisters. This behavior may be regarded as a distinctive feature of the investigated materials. The principal prospective applications of these coatings include space and satellite technologies, nuclear facilities, and various industrial sectors where radiation-resistant coating materials are required.

Approbation and implementation of the research:

The scientific results obtained in the dissertation were presented at several international and national scientific events in accordance with the different research directions and were subjected to scientific discussion. The results of the conducted studies were reported at various conferences and evaluated by specialists in the relevant fields.

1. Hasanov K., Samadov O., Garibov R., Allahverdiyev Q., Mehdiyeva R., Mirzayev M. Investigating surface morphology and crystal structure of carbon nitride coatings // “Radiasiya təhlükəsizliyi problemləri: regional aspektlər” Akademik Mahmud Kərimovun anadan olmasının 75 illiyinə həsr olunmuş elmi-texniki konfrans. Naxçıvan, Azərbaycan: – 18–19 oktyabr, – 2023, – s. 70–71.
2. Hasanov K.M. Thermal oxidation resistance of carbon nitride coatings // X Всероссийский молодежный научный форум с международным участием «Open Science 2023». Петербургский институт ядерной физики им. Б.П. Константинова Национального исследовательского центра «Курчатовский институт». Гатчина, Россия: – 15–17 ноября, – 2023, – с. 58.
3. Hasanov K.M., Mirzayev M.N. Molecular dynamics simulation of the distribution mechanism of proton in the carbonitride structure // Молодежная школа по физике конденсированного состояния (Школа ФКС-2025). Петербургский институт ядерной физики им. Б.П. Константинова Национального исследовательского центра

«Курчатовский институт». Рощино, Ленинградская область, Россия: – 24–28 марта, – 2025, – с. 133.

Publications: The main content and scientific results of the dissertation have been presented in 5 articles published in international journals and 2 abstracts published in the proceedings of international scientific conferences, as well as in 1 article and 1 abstract published within the country. In total, the principal results of the dissertation have been published in 9 scientific works, including 6 articles and 3 conference abstracts.

Name of the organization where the dissertation research was carried out: The experimental studies presented in the dissertation were conducted at the Institute of Physics, Public Legal Entity, under the Ministry of Science and Education of the Republic of Azerbaijan.

Personal contribution of the applicant to the research: Based on a comprehensive review of the relevant literature, the applicant substantiated the relevance of the research, participated in the selection of appropriate research methods and their application to the scientific problems addressed, carried out the planned experiments at all stages of the study, and systematized the obtained results. In addition, the applicant actively participated in the analysis of the experimental results, the presentation and discussion of the research at scientific conferences, and the preparation of scientific articles based on the findings obtained.

Structure and volume of the dissertation: The dissertation comprises 165 pages and includes an introduction, four chapters, conclusions, a list of references, and a list of abbreviations. The main text, excluding figures, tables, and the reference list, contains a total of 180385 characters. The character count of the individual sections is as follows: Introduction – 21,461; Chapter I – 50,399; Chapter II – 25294; Chapter III – 48,983; Chapter IV – 32,005; Conclusions – 2,243. The dissertation contains 50 figures and 9 tables, and the list of references includes 185 sources.

THE CONTENT OF THE DISSERTATION

In the Introduction, the relevance of the dissertation topic is substantiated, and the aim of the study, the main research tasks, scientific novelty, and practical significance are presented. The principal provisions submitted for defense, the research objects, and the applied methods are also specified. In addition, the Introduction provides a brief overview of the main content of the dissertation by chapters.

The first chapter summarizes, on the basis of the scientific literature, the preparation methods, crystal structure, corrosion properties, and structural and morphological changes of carbonitride-based materials under irradiation. Solution-based synthesis, carbothermal reduction, mechanochemical processing, physical and chemical vapor deposition, and cathodic arc evaporation methods are reviewed. The corrosion behavior of carbonitride coatings in aggressive environments, the formation of passive layers, and surface modifications are analyzed. Radiation-induced vacancies, dislocation loops, lattice distortions, and local amorphous regions produced by neutron, proton, heavy-ion, helium, and electron irradiation are discussed. The effects of radiation dose, ion energy, temperature, and particle size on radiation resistance are comparatively evaluated.

Thus, the first chapter summarizes the relationships among the synthesis, structure, corrosion behavior, and radiation resistance of carbonitride materials.

The second chapter provides a detailed description of the preparation conditions of carbonitride-based coatings, the experimental methods used to investigate their structural and surface properties, the principles of corrosion testing, and the technical characteristics of proton irradiation. First, the methodology for depositing TiSiCN, TiNbCN, TiZrCN, TiZrSiCN, and TiZrNbCN coatings on Ti6Al4V alloy substrates by cathodic arc deposition is described

X-ray diffraction was employed to investigate the crystal structure, phase composition, and lattice characteristics of the synthesized samples. The measurements were performed using a Rigaku SmartLab X-ray diffractometer, and the obtained diffraction

data were analyzed with FullProf Suite software to determine the crystalline phases, lattice parameters, and structural characteristics.

The surface morphology of the coatings was examined using a Hitachi 3030PLUS scanning electron microscope. SEM analysis was used to evaluate the surface topography, particle-size distribution, morphological homogeneity, and elemental composition. Following proton irradiation, the fine microstructural features were investigated using a Talos F200i transmission electron microscope in HRTEM and STEM modes, while the chemical composition was determined by EDS.

The chapter also presents the fundamentals of neutron activation analysis used to evaluate the neutron flux and its energy distribution. For this purpose, the operating principle of the IBR-2 pulsed reactor and the characteristics of its irradiation channels at the Frank Laboratory of Neutron Physics of the Joint Institute for Nuclear Research are described.^{1,2}

The corrosion behavior of the carbonitride coatings was investigated using open-circuit potential, linear and potentiodynamic polarization, cyclic polarization, and Tafel methods. Surface roughness was evaluated using a Dektak 150 profilometer in terms of the Ra, Rq, and Sk parameters

Proton irradiation was carried out using the EG-5 electrostatic accelerator located at the Frank Laboratory of Neutron Physics. The facility provides high energy stability and a controllable ion flux, enabling investigation of material behavior under irradiation conditions.

The third chapter presents a comparative investigation of the changes in the crystal structure, phase composition, lattice parameters, texture characteristics, and surface morphology of carbonitride-based coatings under corrosive conditions. At the initial stage, the X-ray

¹ Ananiev, V.D. Power startup of the IBR-2 reactor and the first physical studies of its beams / V.D. Ananiev, V.A. Arkhipov, A.I. Babaev // *At. Énerg.* – 1984. – 57. – p. 227–234.

² Aksenov, V.L. Pulsed fast reactors / V.L. Aksenov, A.E. Verkhoglyadov, M.M. Podlesnyy, E.P. Shabalin // *Natural Science Review*, – 2026. – 3(7). – p. 200709–200709.

diffraction pattern of the $\text{Ti}_6\text{Al}_4\text{V}$ substrate was analyzed separately, and Rietveld refinement was performed using the FullProf program.

The substrate was mainly indexed along the (100), (002), (101), (102), (110), (103), (112), and (201) crystallographic directions, while the (200) reflection of the titanium phase was observed with low intensity. The dominant phase was identified as hexagonal α -Ti crystallizing in the $P6_3/mmc$ space group, with calculated lattice parameters of $a = b = 2.93706 \text{ \AA}$ and $c = 4.69061 \text{ \AA}$.³ In addition, the presence of β -Ti belonging to the $Im\text{-}3m$ space group and a minor amount of an Fe phase belonging to the $Fm\text{-}3m$ space group was also established. A pronounced texture was observed, with the preferred orientation determined to be along the $\{10\text{-}10\}$ direction. The texture was modeled using the function $P_h = G_2 + (1 - G_2)\exp(G_2\alpha_2)$, and a G_2 parameter greater than 7 confirmed the high degree of preferred orientation.

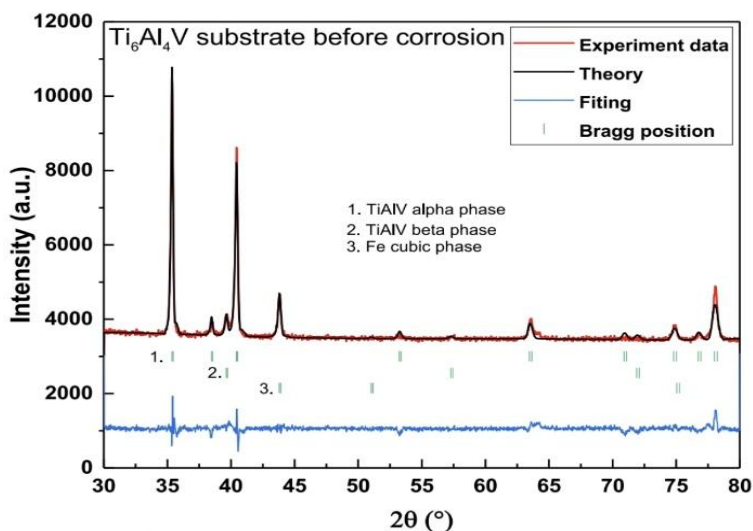


Figure 1. Room-temperature X-ray diffraction pattern and Rietveld refinement of the $\text{Ti}_6\text{Al}_4\text{V}$ substrate

³ Maimaitiyili, T. Residual lattice strain and phase distribution in Ti-6Al-4V produced by electron beam melting / T. Maimaitiyili, R. Woracek, M. Neikter [et al.] // Materials, – 2019. – 12(4). – p. 667.

The XRD results of the TiSiCN coating revealed overlapping diffraction lines from several phases within the structure. Intense peaks associated with the $\text{Ti}_6\text{Al}_4\text{V}$ substrate were observed at 42.00° , 60.4° , and 72° , while a broadened reflection was recorded near 36° . A TiN-type cubic structure was formed in the coating, with a lattice parameter of $4.31263 \text{ \AA}$. The fact that this value exceeds $4.28 \text{ \AA}$ was attributed to lattice distortion and local variations in the C ratio. Because TiC and TiN possess the same type of crystal symmetry, the possible contribution of the TiC phase was also taken into account.⁴ By applying the Scherrer equation to the broadening of the (111) reflection, the average size of the coherent diffraction domains along the {111} direction was estimated to be approximately 10 nm .⁵ At the same time, the distribution of diffraction intensities indicated a strong preferred orientation of these nanocrystalline domains along the {111} direction. The exponential texture parameter calculated using FullProf was 9.0. In the TiSiCN sample with a C ratio of 1.6, the contribution of the TiC phase increased, and the diffraction peaks shifted toward lower angles. After corrosion, a general decrease in diffraction intensity was observed, with a particularly pronounced reduction at 39.6° . Although this reflection overlaps with $\beta\text{-Ti}$ (110), it was also associated with the (200) reflection of the rutile TiO_2 phase.

Structural analysis of the TiNbCN coatings demonstrated high phase stability. In the TiNbCN sample with a C ratio of 0.6, TiC, NbC, and TiN phases were identified, with NbC acting as the dominant phase. The lattice parameter and unit-cell volume of NbC were $4.33289 \text{ \AA}$ and $81.3455 \text{ \AA}^3$, respectively. After corrosion, increases of 0.04% in the lattice parameter and 0.12% in the unit-cell volume were observed, while no phase transformation was detected. In the TiNbCN sample with a C ratio of 1.6, NbC also remained the dominant phase, indexed along the (111) plane, with a lattice parameter of $4.40012 \text{ \AA}$

⁴ Xu, H. Tribological behavior of a TiSiCN coating tested in air and coolant / H. Xu, X. Nie, R. Wei // Surface and Coatings Technology, – 2006. – 201(7). – p. 4236–4241.

⁵ Scherrer, P. Nachrichten von der Gesellschaft der Wissenschaften zu Göttingen, Mathematisch-Physikalische Klasse / P. Scherrer // Nachrichten von der Gesellschaft der Wissenschaften zu Göttingen, Mathematisch-Physikalische Klasse, – 1918. – 2. – p. 98–100.

and a unit-cell volume of 85.19109 Å³. Rietveld analysis showed that the lattice parameters and phase fraction of the NbC nanocrystals remained unchanged after corrosion.

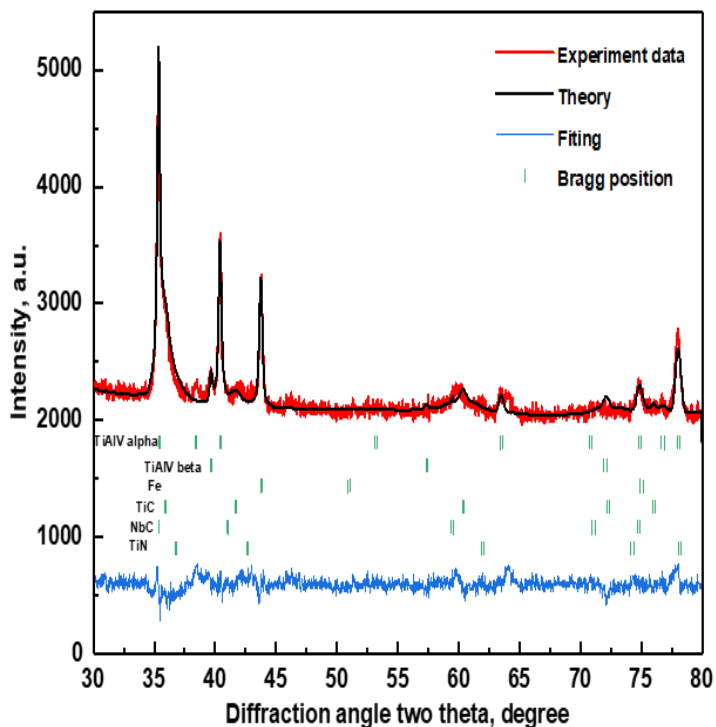


Figure 2. Room-temperature X-ray diffraction pattern and Rietveld refinement of the TiNbCN composite

In the TiZrCN sample with a C ratio of 0.6, four major cubic phases TiC, TiN, ZrC, and ZrN were identified. Their lattice parameters were 4.318 Å, 4.244 Å, 4.6828 Å, and 4.5627 Å, respectively.⁶ The peaks corresponding to the (111) direction of ZrC and ZrN were recorded at $2\theta = 34.54^\circ/34.63^\circ$ and $34.81^\circ/34.9^\circ$, respectively, while the broadening observed in the 36° – 37° range was attributed to the overlap of the (111) reflections of the TiC and TiN

⁶ Yang, M. Direct solid-state synthesis at high pressures of new mixed-metal oxynitrides: RZrO₂N (R = Pr, Nd, and Sm) / M. Yang, J.A. Rodgers, L.C. Middel [et al.] // *Inorganic Chemistry*, – 2009. – 48(24). – p. 11498–11500.

phases. The (200), (220), and (311) reflections of TiC and TiN were observed at 43°, 62°, and 74°, respectively. Rietveld analysis showed phase fractions of 47% for ZrN, 28% for TiN, 2.29%(0.17) for TiC, and 3.51%(0.16) for ZrC. After corrosion, the fractions of the main phases decreased, with ZrN and ZrC remaining at approximately 11% and 4%, respectively. Although an approximately 0.5% increase in density was recorded for the ZrC phase, this change was interpreted cautiously in view of the instrument's standard error of 3–5%.

For the TiZrCN coating with a C ratio of 1.6, Rietveld analysis revealed seven phases: $\text{Ti}_6\text{Al}_4\text{V } \alpha$, $\text{Ti}_6\text{Al}_4\text{V } \beta$, free Fe, TiN, ZrN, ZrC, and a minor TiC phase. The lattice parameters of TiN, ZrN, ZrC, and TiC were determined to be 4.45108 Å, 4.48536 Å, 4.23194 Å, and 4.19357 Å, respectively. Overlapping reflections were observed at 34.54°/34.63° and 34.81°/34.9° for ZrC and ZrN, and within the 36°–37° range for TiC and TiN. After corrosion, the TiN, ZrC, and ZrN phases were no longer detected, whereas TiC exhibited a certain degree of structural stability. The 7.7% increase in the unit-cell volume of the TiC phase was attributed to surface stresses and coating–substrate interactions.

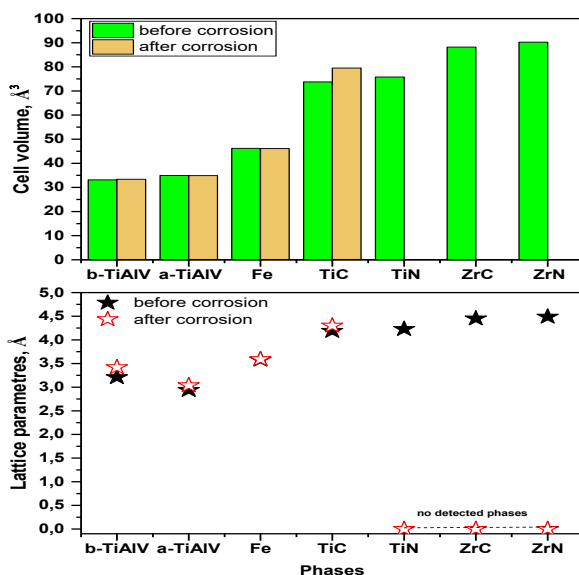
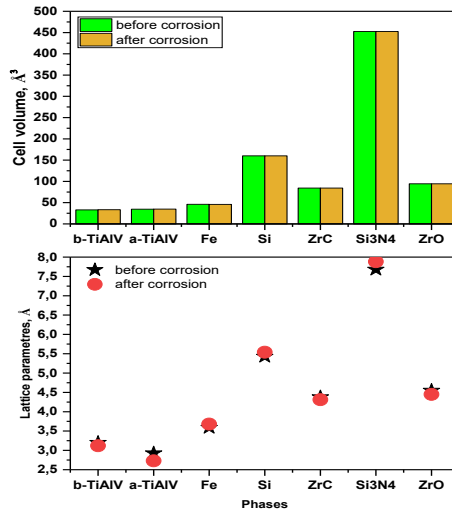


Figure 3. Comparison of the structural parameters of the TiZrCN coating with a C ratio of 1.6 before and after corrosion

In the TiZrSiCN system with a C ratio of 0.6, the addition of Si resulted in structural rearrangement and the formation of new phases, including ZrO, Si, and Si₃N₄. The phase fractions of ZrO and Si were approximately 12.5% and 15%, respectively, while ZrC, TiC, and Si₃N₄ accounted for about 19%, 2%, and 2.5%, respectively. The α phase of the Ti6Al4V substrate constituted approximately 38%, the β phase 9%, and free Fe about 2%. The broad peak observed near 58° was attributed to the overlap of the Ti6Al4V β (200), ZrC (220), ZrO (220), Si (311), Si (222), and Si₃N₄ (422) reflections. After corrosion, the phase fractions were approximately 10% for Si, 1% for ZrO, 3% for ZrC, and 3% for Si₃N₄.

In the TiZrSiCN sample with a C ratio of 1.6, the phase fractions were determined to be 15.42% Si, 4.5% Si₃N₄, 13.57% ZrO, and 17.20% ZrC. The α and β phases of the Ti6Al4V substrate accounted for 38.21% and 10.25%, respectively, while free Fe constituted 2.15%. After corrosion, an increase in the intensity of the Ti6Al4V α phase was observed, whereas decreases were recorded for the Ti6Al4V β (200), ZrC (220), ZrO (220), Si (311), Si (222), and Si₃N₄ (422) reflections. These results demonstrate that the addition of Si significantly alters the phase composition of the TiZrCN system.



Şəkil 4. C:N nisbəti 1.6 olan TiZrSiCN örtüyünün korroziyadan əvvəl və sonra struktur parametrlərinin müqayisəsi

In the TiZrNbCN system, the NbC phase was found to be dominant. According to the Rietveld analysis, NbC accounted for approximately 97.5% of the total diffraction pattern, while the remaining approximately 2.5% was attributed to the Ti₆Al₄V substrate and the TiC, TiN, and ZrC phases. The slight decrease in the NbC lattice parameter was associated with texture relaxation. Compared with the previously analyzed multiphase samples, a lower intensity was observed at approximately 58°, with the main contribution arising from NbC (220) and the Ti₆Al₄V β phase. After corrosion, the amount of the NbC phase decreased by only about 0.5%, while its main structural characteristics were preserved. The observation of peaks corresponding to NaCl indicated that electrolyte ions were able to penetrate into surface defects.

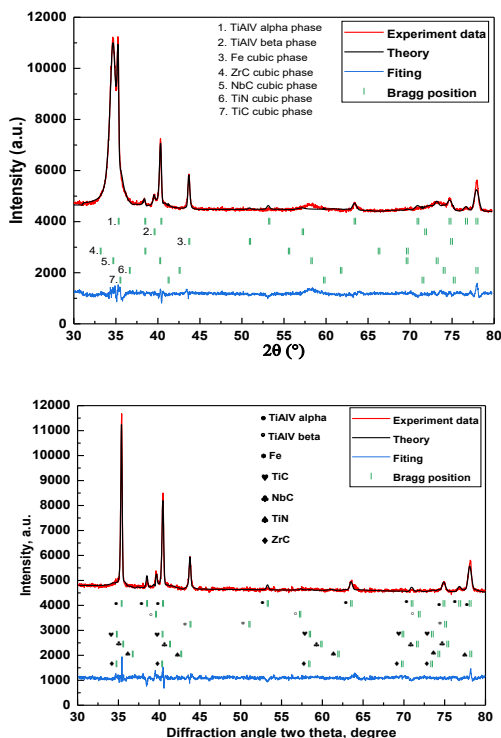


Figure 5. X-ray diffraction pattern of the TiZrNbCN coating with a C ratio of 1.6 before and after corrosion

After heat treatment at 1270 K, a significant narrowing of the diffraction peaks was observed in the X-ray diffraction patterns of the carbonitride coatings. This change was attributed to variations in crystalline ordering and relaxation of residual stresses. SEM images revealed the formation of cracks on the coating surfaces after heat treatment. The difference in thermal expansion between the carbide–nitride coatings and the $\text{Ti}_6\text{Al}_4\text{V}$ substrate resulted in the development of residual stresses during cooling. Based on the (220) reflection, the average crystallite size determined using the Scherrer equation was ≈ 5 nm. When only the Cauchy component of line broadening was considered and Type II residual stresses were assumed to follow a Gaussian distribution, the crystallite size was estimated to be ≈ 8.3 nm.

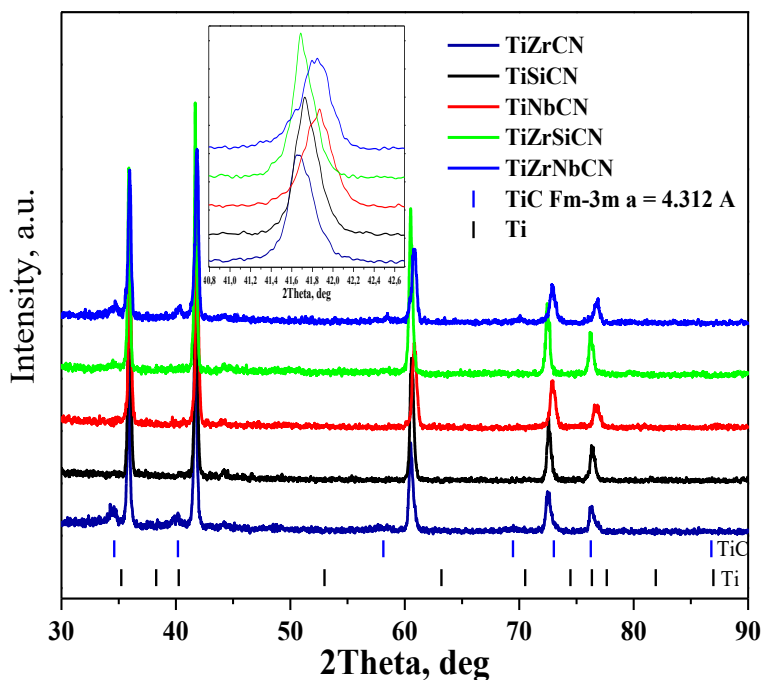


Figure 6. X-ray diffraction patterns of TiZrCN, TiSiCN, TiNbCN, TiZrSiCN, and TiZrNbCN coatings after heat treatment

SEM investigations revealed the presence of microdroplets with different diameters on the TiSiCN coatings before corrosion. The

TiSiCN coatings were found to possess a heterogeneous structure in which TiC_xNy nanocrystals were embedded in an amorphous $\text{SiC}_x\text{Ny/a-C}$ matrix.^{7,8,9} In the TiSiCN sample with a C ratio of 1.6, the lower nitrogen content suppressed the formation of the amorphous matrix and promoted the development of larger microdroplets. Nitrogen deficiency also facilitated the formation of Ti-rich regions and, consequently, the formation of a TiO_2 layer. In the TiSiCN sample with a C ratio of 0.6, the higher nitrogen content was accompanied by a smoother surface morphology. After corrosion, the principal change was limited to a reduction in the number of micro- and macroparticles.

On the surface of the TiNbCN coating with a C ratio of 0.6, an irregular distribution of crystallites of different sizes was observed, while elemental mapping demonstrated a highly homogeneous distribution of Ti, Nb, C, and N. Although a similar morphology was retained in the TiNbCN sample with a C ratio of 1.6, the number of nanoscale crystallites was lower than that observed in the sample with a C ratio of 0.6. Nevertheless, the homogeneous surface distribution of Ti, C, N, and Nb was preserved. After corrosion, no significant changes were detected in the TiNbCN system in terms of either surface morphology or elemental distribution.

⁷ Qiu, L. Microstructure, mechanical properties and cutting performances of TiSiCN super-hard nanocomposite coatings deposited using CVD method under the guidance of thermodynamic calculations / L. Qiu, Y. Du, L. Wu [et al.] // Surface and Coatings Technology, – 2019. – 378. – p. 124956.

⁸ Qin, C.P. Cavitation erosion behavior of nanocomposite Ti–Si–C–N and Ti/Ti–Si–C–N coatings deposited on 2Cr13 stainless steel using a Plasma Enhanced Magnetron Sputtering process / C.P. Qin, Y.G. Zheng, R. Wei // Surface and Coatings Technology, – 2010. – 204(21–22). – p. 3530–3538.

⁹ Wang, H. Tribocorrosion behaviors of TiSiCN nanocomposite coatings deposited by high power impulse magnetron sputtering / H. Wang, Y. Ou, X. Zhang [et al.] // Materials Research Express, – 2020. – 7(7). – p. 076407.

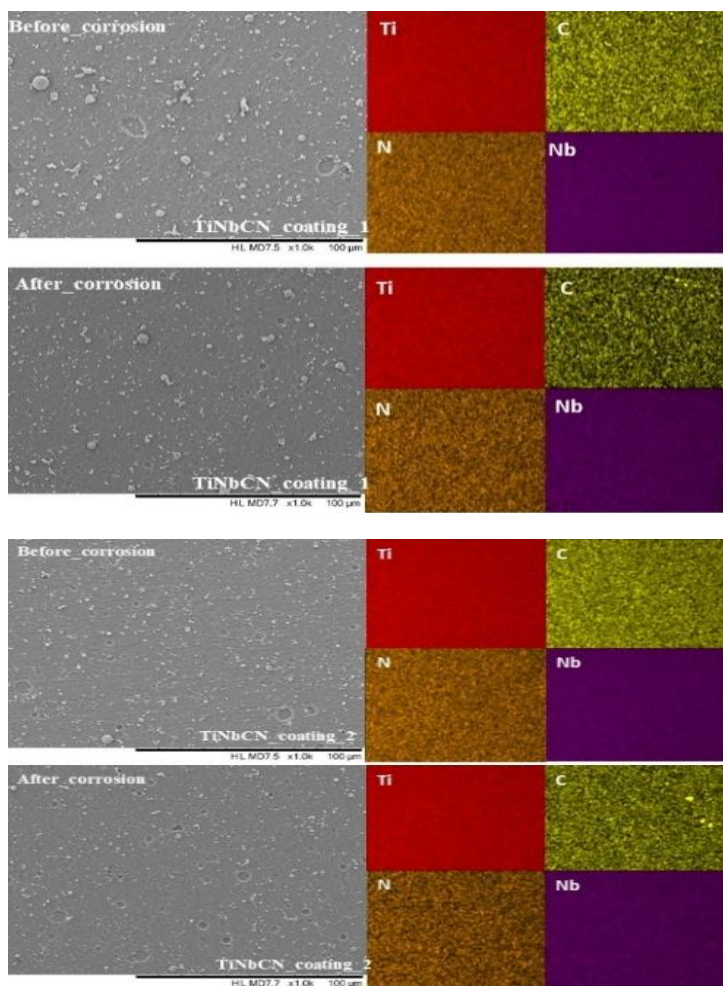


Figure 7. SEM images and elemental mapping profiles of the TiNbCN-1 and TiNbCN-2 coatings before and after corrosion

More pronounced changes in surface morphology and a reduction in the homogeneous distribution of elements were observed in the TiZrCN and TiZrSiCN coatings after corrosion. Before corrosion, the surface of the TiZrCN sample with a C ratio of 1.6 was characterized by a homogeneous distribution of crystallites of different sizes, while structural analysis identified cubic TiN, ZrN, ZrC, and TiC phases. After corrosion, the distribution of larger

crystals acquired a more amorphous character, whereas the smaller crystallites were partially preserved. Comparative structural analysis showed that mainly cubic ZrC and TiC phases remained on the surface. Elemental mapping revealed a homogeneous distribution of Ti, C, N, Al, and V, while non-uniform dark regions appeared in the Zr map.

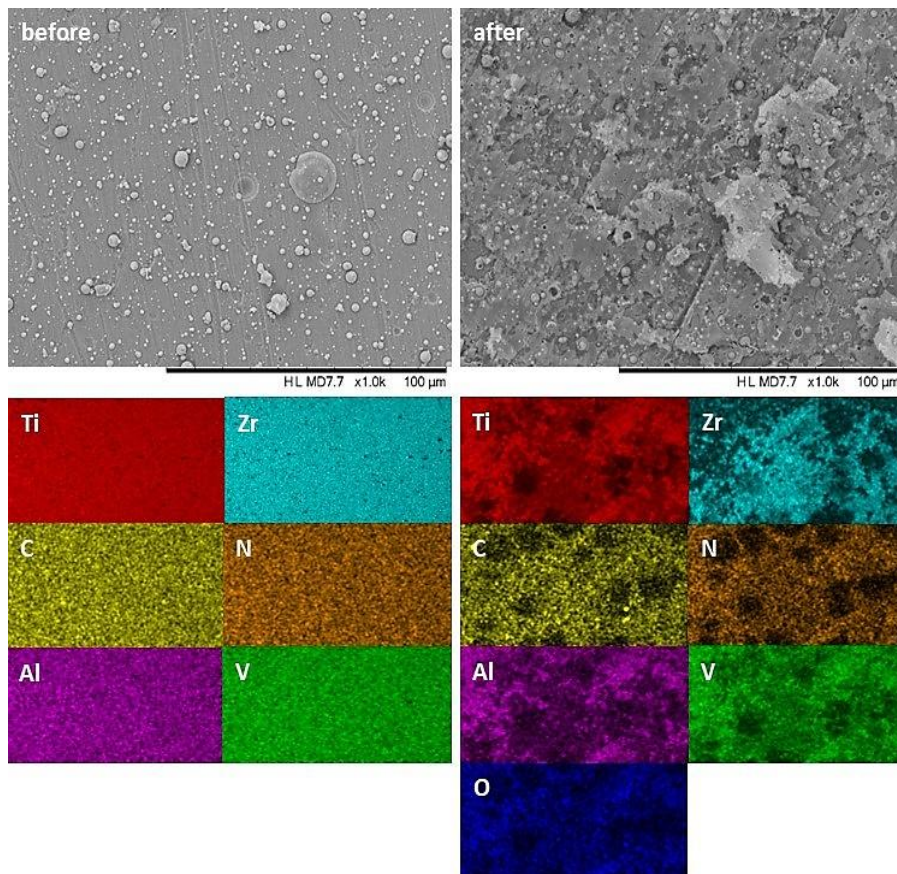


Figure 8. SEM images and elemental mapping profiles of the TiZrCN-2 coating before and after corrosion

The TiZrNbCN coatings exhibited greater stability from both structural and morphological perspectives. After corrosion, only localized surface changes were observed, while the fractions of the

main phases and the crystal structure were largely preserved. Experimental comparisons showed that NaCl exposure caused crevice corrosion in the CNTi-(Zr) and CNTi-(ZrSi) coatings, whereas localized corrosion occurred on the CNTi-(ZrNb) surface. Only the CNTi-(ZrSi) coating was characterized by the formation of an oxide phase in the NaCl environment, while blistering and degraded regions were observed in the CNTi-(Zr) coatings.^{10,11} In contrast, the CNTi-(ZrNb) coating exhibited weak oxidation reactions and a low corrosion rate.

At the end of the chapter, the obtained structural, morphological, and elemental-composition results were summarized and analyzed jointly. It was established that the stability of carbonitride coatings in corrosive environments is closely related to their phase composition, C ratio, crystal structure, and surface morphology. In particular, the crystal structure and surface characteristics of the TiNbCN coatings were found to be largely preserved after corrosion. The obtained results are scientifically significant for evaluating the structural stability of carbonitride-based coatings in aggressive environments and for assessing their potential application as protective materials.

The fourth chapter investigates amorphization, blister and pit formation, micro- and nanoscale surface changes, dislocation generation, and local lattice amorphization in TiSiCN, TiNbCN, TiZrCN, TiZrNbCN, and TiZrSiCN composites irradiated with protons of different energies. Long- and short-lived radioisotopes that may form in the structure under fast-neutron irradiation were also analyzed. The concentration of irradiation-induced defects in the crystal structure is important for assessing radiation resistance and clarifying the interaction mechanism of ionizing radiation.

¹⁰ Shu, Y. Synergistic effect of NaCl and water vapor on the corrosion of 1Cr-11Ni-2W-2Mo-V steel at 500–700 °C / Y. Shu, F. Wang, W. Wu // *Oxidation of Metals*, – 1999. – 51(1). – p. 97–110.

¹¹ Sankara Narayanan, T.S.N. Strategies to improve the corrosion resistance of microarc oxidation (MAO) coated magnesium alloys for degradable implants: Prospects and challenges / T.S.N. Sankara Narayanan, I.S. Park, M.H. Lee // *Progress in Materials Science*, – 2014. – 60(1). – p. 1–71.

During neutron irradiation, gamma radiation was measured using a high-purity germanium detector. Neutron spectra were obtained from standard activation samples using nickel (Ni) isotopes. At the end of the process, gamma-activated ^{58}Co was used to calculate the fast-neutron flux density. Nickel was selected because its neutron reaction threshold begins at about 1 MeV. An effective cross section of $\sigma_{\text{eff}} = 92 \text{ mb}$ was used to estimate the neutron flux density, while the $^{60}\text{Ni}/^{58}\text{Ni}$ ratio in the measuring wire and the integrated reaction cross sections were also considered. After irradiation, the absorption peak observed at 810.7 keV in the gamma spectrum of the neutron-activation products was assigned to the ^{58}Co radioisotope.¹² Based on the activity measured by gamma spectrometry, the fast-neutron flux density was calculated for each sample. The fast-neutron flux density was $1.65 \times 10^7 \text{ n}/(\text{cm}^2 \cdot \text{s})$, while the total neutron fluence was $1.71 \times 10^{13} \text{ n}/\text{cm}^2$.

Table 1.

Neutron flux density for neutron activation analysis of carbonitride samples prepared on Ti6Al4V substrates

Samples	Intensity, $\text{n}/(\text{cm}^2 \times \text{s})$	Fluence, n/cm^2
TiSiCN, with Ti6Al4V substrate	1.65×10^7	1.71×10^{13}
TiZrSiCN, with Ti6Al4V substrate	1.65×10^7	1.71×10^{13}
TiZrCN, with Ti6Al4V substrate	1.65×10^7	1.71×10^{13}
TiZrNbCN, with Ti6Al4V substrate	1.65×10^7	1.71×10^{13}
TiNbCN, with Ti6Al4V substrate	1.65×10^7	1.71×10^{13}

¹² Barry, J.F. The cross section of the $^{58}\text{Ni}(\text{n,p})^{58}\text{Co}$ reaction for neutrons in the energy range 1.6 to 14.7 MeV / J.F. Barry // Journal of Nuclear Energy. Parts A/B. Reactor Science and Technology, – 1962. – 16(10). – p. 467–472.

These values were identical for the TiSiCN, TiZrSiCN, TiZrCN, TiZrNbCN, and TiNbCN samples. After fast-neutron irradiation, the samples showed no significant activity. Neutron activation analysis of the Si-, Zr-, and Nb-containing samples demonstrated that irradiation of TiZrCN, TiZrNbCN, and TiZrSiCN coatings with neutrons of $E > 1$ MeV did not lead to the formation of short-lived radioactive isotopes.

DPA and SRIM calculations were performed to characterize the distribution and energy loss of H^+ ions in TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN composites irradiated at room temperature with protons of 15 keV, 200 keV, 1.2 MeV, and 2.0 MeV.¹³ Proton irradiation was carried out using the EG-5 electrostatic accelerator under high-vacuum conditions. For structural studies, the samples were irradiated at 15 keV, 200 keV, 1.2 MeV, and 2.0 MeV with a fluence of 1.0×10^{17} ions/cm². For morphological studies, the effects of 2.0 MeV protons at fluences of 1.0×10^{15} , 1.0×10^{16} , and 1.0×10^{17} ions/cm² were compared.

Figure 9 presents the vacancy and SRIM analysis results for TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN. The mean kinetic energy of H^+ ions and electronic stopping along the depth profile were evaluated using SRIM. Vacancy and free-volume formation in coatings irradiated with low- and high-energy protons produced different radiation effects and determined the ion distribution within the crystal structure. Depending on coating density and proton energy, displaced atoms and lattice-coordinate shifts generated additional free volume in the microstructure. The maximum penetration depth, dependent on ion energy, was considered the dose peak. The density of TiCN has been reported in the range of 5.08–6.0 g/cm³ and, depending on composition and chemical form, up to 6.0–6.8 g/cm³. SRIM analysis of 15 keV, 200 keV, 1.2 MeV, and 2.0 MeV H^+ ions in the studied composites showed penetration depths ranging from 100 nm to 28 μ m. Modeling indicated that proton ions could

¹³ Ziegler, J.F. SRIM—The stopping and range of ions in matter (2010) / J.F. Ziegler, M.D. Ziegler, J.P. Biersack // Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, – 2010. – 268(11–12). – p. 1818–1823.

penetrate carbonitride samples to a maximum depth of about 20 μm . For 15 and 200 keV ions, the penetration depth was approximately 150 nm, whereas increasing the proton energy to 1.2–2.0 MeV increased the penetration depth to the micrometer scale.

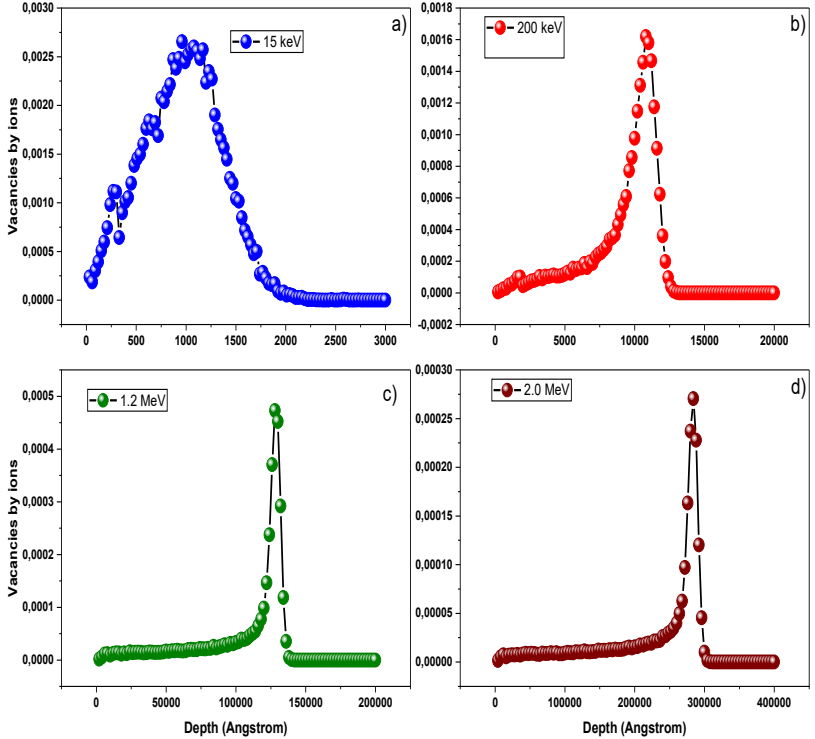


Figure 9. Vacancy formation and penetration depth induced by 15 keV, 200 keV, 1.2 MeV, and 2.0 MeV H^+ ions in carbonitride composites

Figure 10 presents the surface morphologies of unirradiated TiZrCN, TiZrNbCN, and TiZrSiCN composites and of the same materials after irradiation with 2.0 MeV protons at fluences of 1.0×10^{15} , 1.0×10^{16} , and 1.0×10^{17} ions/cm². No large-scale crystal-to-amorphous transition or formation of voluminous blisters was observed on the surface as a function of proton energy and fluence. After proton irradiation, the carbonitride coatings generally retained stable surface morphology at both the micro- and nanoscale levels. Nevertheless, local amorphization was detected in some micro- and macroscale

crystalline particles as a result of irradiation. The surface distribution was heterogeneous, and the carbon structure was assessed as an actively interacting but weakly coordinated structure. It was established that the micro- and macroscale particles present on all investigated surfaces were formed during the deposition process and acted as defect centers.

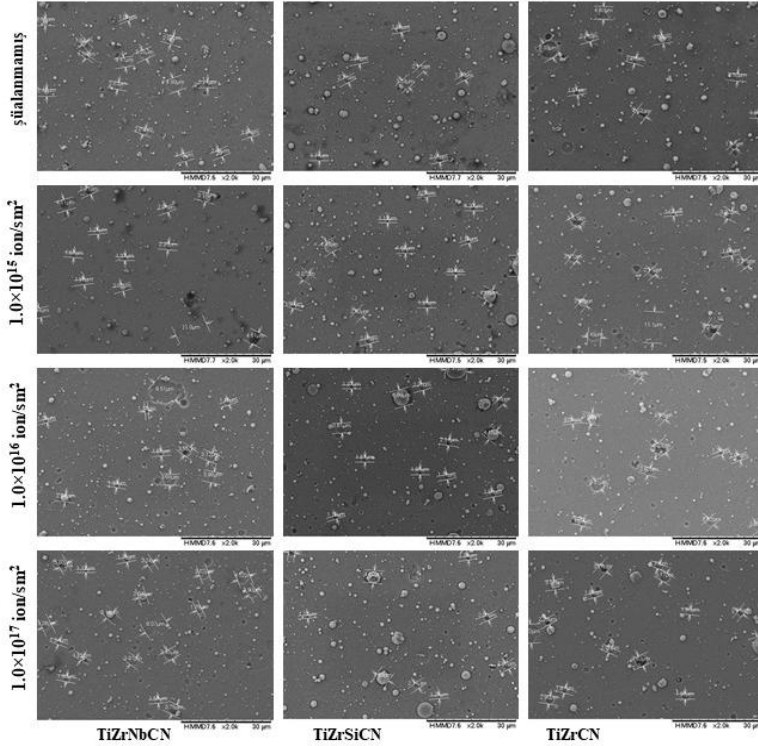


Figure 10. Surface morphology of TiZrNbCN, TiZrSiCN, and TiZrCN composites irradiated with 2.0 MeV protons at fluences of 1.0×10^{15} , 1.0×10^{16} , and 1.0×10^{17} ions/cm²

The processes and changes occurring on the surfaces of TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN composites under the effect of 2.0 MeV H⁺ ions were investigated using high-resolution transmission electron microscopy (TEM). The study of blistering and pit formation induced on coating surfaces by proton ions of different energies is of considerable importance for assessing

radiation resistance.¹⁴ Before proton irradiation, the sizes of the microdroplets were similar for all coatings, and it was established that Si and Nb additions did not affect these dimensions. After irradiation, a slight increase in size was observed; however, this change was not considered significant, as it depended on the selected observation area.

Figure 11 shows the morphologies, at a scale of 400 nm, of unirradiated TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN composites and of the same composites after irradiation with 2.0 MeV proton H^+ ions at different fluences, namely 1.0×10^{15} , 1.0×10^{16} , and 1.0×10^{17} ions/cm². Comparative images demonstrated the overall morphological stability of the coatings under high-energy proton exposure. After proton irradiation, neither blister formation nor a blister “adsorption” mechanism was observed on the surfaces of the carbonitrides, and no “blister interaction transition effects” occurred between the substrate and the coating under the action of proton ions. At the same time, the formation of homogeneously distributed pinholes with sizes of 3–25 nm was clearly identified on the surface.

¹⁴ Badrutdinov, A. In situ observations of blistering of a metal irradiated with 2-MeV protons / A. Badrutdinov, T. Bykov, S. Gromilov [et al.] // *Metals*, – 2017. – 7(12). – p. 558.

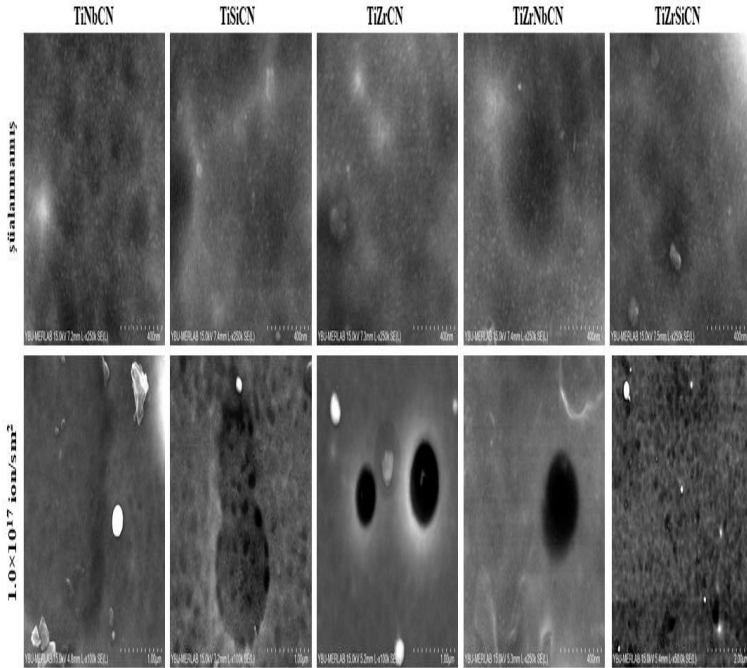


Figure 11. Surface morphology of unirradiated TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN composites and after irradiation with 2.0 MeV protons at 1.0×10^{17} ions/cm²

For ions with energies in the keV range, the penetration depth was about 100 nm, whereas increasing the energy to the MeV range increased the penetration depth to the micrometer scale. A transition from keV to MeV energies was accompanied by an increase in the number of surface pinholes. Dislocations along different directions and motion of atomic layers perpendicular to the crystal lattice were rarely observed. The formation of Frank loops in the coating morphology was associated with the generation of new atomic layers within the structure. Although the interaction mechanism of H⁺ ions with material atoms differs from that of swift heavy ions, the evolution of free volume and defect clusters was found to follow similar mechanisms.

TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN composites were formed on Ti6Al4V substrates as cubic NaCl-type

carbonitride matrices, with preferred (111), (200), and (220) orientations characteristic of the FCC structure in the initial state. Because substrate peaks partly overlapped those of the coatings, the substrate contribution had to be considered separately in the analysis of the initial diffractograms. Small deviations in peak-intensity ratios from those of a random powder indicated the presence of texture in the coatings.

Structural analyses after irradiation with 15 keV, 200 keV, 1.2 MeV, and 2.0 MeV protons at a fluence of 1.0×10^{17} ions/cm² showed that low-energy protons caused no significant changes in the structural parameters. With increasing proton energy, only slight and monotonic variations occurred, attributed to weak elastic relaxation without disrupting long-range order. No new phases or phase transitions were detected after irradiation, and all coatings retained the cubic NaCl-type carbonitride matrix.

Table 2.

Changes in the lattice parameters of TiSiCN, TiNbCN, TiZrCN, TiZrSiCN, and TiZrNbCN composites irradiated with 15 keV, 200 keV, 1.2 MeV, and 2.0 MeV protons

Samples	H ⁺ ion energy	Lattice parameters before and after irradiation, Å		Unit-cell volume before and after irradiation, Å ³	
TiSiCN	15 keV	4.33289	4.33289	81.34550	81.34550
	200 keV	4.33289	4.33294	81.34550	81.34578
	1.2 MeV	4.33289	4.33322	81.34550	81.34596
	2.0 MeV	4.33289	4.33363	81.34550	81.34614
TiNbCN	15 keV	4.33289	4.33289	81.34550	81.34550
	200 keV	4.33289	4.33294	81.34550	81.34578
	1.2 MeV	4.33289	4.33322	81.34550	81.34596
	2.0 MeV	4.33289	4.33363	81.34550	81.34614
TiZrCN	15 keV	4.45521	4.45521	88.43130	88.43130
	200 keV	4.45521	4.45528	88.43130	88.43146
	1.2 MeV	4.45521	4.45573	88.43130	88.43171
	2.0 MeV	4.45521	4.45612	88.43130	88.43223
TiZrSiCN	15 keV	4.29398	4.29398	79.17368	79.17368
	200 keV	4.29398	4.29403	79.17368	79.17379
	1.2 MeV	4.29398	4.29431	79.17368	79.17403
	2.0 MeV	4.29398	4.29489	79.17368	79.17437
TiZrNbCN	15 keV	4.67704	4.67704	102.30899	102.30899
	200 keV	4.67704	4.67712	102.30899	102.30907
	1.2 MeV	4.67704	4.67745	102.30899	102.30933
	2.0 MeV	4.67704	4.67812	102.30899	102.30975

Thus, irradiation with 15 keV, 200 keV, 1.2 MeV, and 2.0 MeV protons caused no major changes in the basic crystal structure of the carbonitride coatings. With increasing proton energy, the

penetration depth increased from the nanometer to the micrometer scale, while local vacancies, dislocations, and defect centers formed, together with 3–25 nm surface pinholes. However, no large-scale amorphization, phase transition, or blistering was observed. Neutron activation results showed no formation of short-lived radioactive isotopes, while morphological studies indicated that the TiNbCN coating retained greater structural stability after high-energy proton irradiation.

MAIN SCIENTIFIC RESULTS

1. It was established that titanium-based carbonitride composites prepared with different C ratios exhibit a corrosion-resistant surface morphology at C = 1.6. Electrochemical processes lead to the formation of passive layers on the coating surfaces. Depending on stoichiometry, all composites show favorable electrochemical properties, including low capacitance, high pore resistance, low current density, and high polarization resistance. At C = 1.6, TiNbCN shows the smallest crystal changes after corrosion.
2. Although post-corrosion amorphization occurs only on a small scale, local degradation zones of 100–350 nm form in different surface regions. Surface-morphology analysis showed that after chemical corrosion, local degradation occurs at C = 0.6, whereas at C = 1.6 these regions are replaced by pit-like structures.
3. After activation analysis with 1 MeV fast neutrons at a flux density of $1.65 \times 10^7 \text{ n}/(\text{cm}^2 \cdot \text{s})$ and a total fluence of $1.71 \times 10^{13} \text{ n}/\text{cm}^2$, no long-lived radioisotopes were detected in TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, or TiZrSiCN.
4. No crystal-to-amorphous phase transition was observed in the surface morphology of composites irradiated with protons of different energies. Low-energy proton irradiation also did not produce needle-like surface structures. After high-energy proton irradiation, dislocations in different directions, layered crystal features, and nanoscale Frank loops formed. Among the studied composites, TiNbCN showed the least structural and

morphological change, while surface homogeneity was preserved.

5. Under 2.0 MeV H^+ irradiation, the (111), (200), and (220) orientations characteristic of the FCC structure were retained in TiNbCN, TiSiCN, TiZrCN, TiZrNbCN, and TiZrSiCN. No significant changes in lattice parameters or unit-cell volume were observed before and after irradiation, indicating the potential of these coatings for use in space and nuclear technologies.

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