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СТРУКТУРНО-ФАЗОВЫЕ ИЗМЕНЕНИЯ В КОНЦЕНТРИРОВАННЫХ ТВЕРДЫХ РАСТВОРАХ СИСТЕМЫ V – Nb – Ta – Ti, ОБЛУЧЕННЫХ ИОНАМИ КРИПТОНА

В. В. УГЛОВ[1)]***, С. В. ЗЛОЦКИЙ***[1)]***, М. М. БЕЛОВ***[1)]***, А. Е. РЫСКУЛОВ***[2)]***,***
И. А. ИВАНОВ[2)]***, А. Е. КУРАХМЕДОВ***[2)]***, Д. А. МУСТАФИН***[2)]***, Э. Д. САПАР***[2)]***, КЕ ДЖИН***[3)]

[1)]*Белорусский государственный университет, пр. Независимости, 4, 220030, г. Минск, Беларусь*
[2)]*Институт ядерной физики Министерства энергетики Республики Казахстан, ул. Ибрагимова, 1, 050032, г. Астана, Казахстан*
[3)]*Пекинский технологический институт, ул. Южная, 5, Чжунгуаньцунь, 100811, г. Пекин, Китай*

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Авторы:
Владимир Васильевич Углов – доктор физико-математических наук, профессор; заведующий кафедрой физики твердого тела физического факультета.
Сергей Владимирович Злоцкий – кандидат физико-математических наук; старший научный сотрудник кафедры физики твердого тела физического факультета.
Михаил Михайлович Белов – студент физического факультета. Научный руководитель – В. В. Углов.
Азамат Еркнулы Рыскулов – кандидат физико-математических наук; инженер службы управления циклотрона ДЦ-60.
Игорь Александрович Иванов – кандидат физико-математических наук; начальник циклотрона ДЦ-60.
Алишер Ерметович Курахмедов – начальник службы управления циклотрона ДЦ-60.
Даулет Айтмагомбетович Мустафин – начальник службы управления циклотрона ДЦ-60.
Эсет Деулетулы Сапар – инженер службы высокочастотных систем и источника электронного циклотронного резонанса циклотрона ДЦ-60.
Ке Джин – доктор наук (материаловедение и инженерия); профессор.

Authors:
Vladimir V. Uglov, doctor of science (physics and mathematics), full professor; head of the department of solid state physics, faculty of physics.
uglov@bsu.by
Sergey V. Zlotsky, PhD (physics and mathematics); senior researcher at the department of solid state physics, faculty of physics.
zlotski@bsu.by
Mikhail M. Belov, student at the faculty of physics.
mb17023@gmail.com
Azamat E. Ryskulov, PhD (physics and mathematics); engineer at the management service of the DC-60 cyclotron.
ryskulov_nbd@mail.ru
Igor A. Ivanov, PhD (physics and mathematics); head at the DC-60 cyclotron.
igor.ivanov.inp@gmail.com
Alisher E. Kurakhmedov, head of the management service of the DC-60 cyclotron.
Daulet A. Mustafin, head of the management service of the DC-60 cyclotron.
Aset D. Sapar, engineer of the high frequency system and electron cyclotron resonance source service of the DC-60 cyclotron.
Ke Jin, PhD (materials science and engineering); professor.
jinke@bit.edu.cn

Приведены результаты влияния облучения при комнатной температуре низкоэнергетическими ионами криптона (энергия 280 кэВ и флюенс $5 \cdot 10^{15}$ см$^{-2}$) на структурно-фазовое состояние многокомпонентных твердых растворов на основе системы V – Nb – Ta – Ti. Методами сканирующей электронной микроскопии и рентгеноструктурного анализа было установлено, что сформированные бинарные, тройные и четверные сплавы системы V – Nb – Ta – Ti являются равнокомпозиционными однофазными твердыми растворами, имеют однородное распределение элементов в приповерхностном слое и обладают сжимающими микро- и макронапряжениями, рассчитанными методами Холдера – Вагнера и $\sin^2\psi$. При облучении ионами криптона сплавов системы V – Nb – Ta – Ti существенных изменений в структурно-фазовом состоянии не выявлено. Распада твердого раствора и нарушения равнокомпозиционности и однородности распределения элементов в приповерхностном слое не происходит. Облучение ионами криптона приводит к изменению уровня микро- и макронапряжений для всех сплавов системы V – Nb – Ta – Ti.

Ключевые слова: высокоэнтропийные сплавы; ВЭС; многокомпонентные твердые растворы; облучение; радиационные дефекты; ионы криптона; остаточные напряжения.

STRUCTURAL AND PHASE STATES ON CONCENTRATED SOLID SOLUTION OF THE V – Nb – Ta – Ti SYSTEM IRRADIATED BY KRYPTON IONS

V. V. UGLOV [a]***, S. V. ZLOTSKY*** [a]***, M. M. BELOV*** [a]***, A. E. RYSKULOV*** [b]***,***
I. A. IVANOV [b]***, A. E. KURAKHMEDOV*** [b]***, D. A. MUSTAFIN*** [b]***, A. D. SAPAR*** [b]***, KE JIN*** [c]

[a] *Belarusian State University, 4 Niezaliežnasci Avenue, Minsk 220030, Belarus*
[b] *Institute of Nuclear Physics, Ministry of Energy of the Republic of Kazakhstan,*
1 Ibragimova Street, Astana 050032, Kazakhstan
[c] *Beijing Institute of Technology, 5 South Street, Zhongguancun, Beijing 100811, China*
Corresponding author: V. V. Uglov (uglov@bsu.by)

The results of the effect of irradiation at room temperature by low-energy krypton ions with an energy of 280 keV and a fluence of $5 \cdot 10^{15}$ cm^{-2} on the structural-phase state of multicomponent solid solutions based on the V – Nb – Ta – Ti system are presented. By scanning electron microscopy and X-ray diffraction analysis, it was found that the formed binary, triple and quadruple alloys of the V – Nb – Ta – Ti system are equiatomic single-phase solid solutions, have a homogeneous distribution of elements in the near-surface layer and have compressive micro- and macrostresses calculated by Halder – Wagner and $\sin^2\psi$ methods. The conducted studies have shown that no significant changes in the structural-phase state were detected when the alloys of the V – Nb – Ta – Ti system were irradiated by krypton ions. There is no decay of the solid solution and disturbance of the equiatomic and uniformity of the distribution of elements in the near-surface layer. Irradiation by krypton ions leads to a change in the level of micro- and macrostresses for all alloys of the V – Nb – Ta – Ti system.

Keywords: high-entropy alloys; HEA; multicomponent solid solutions; irradiation; radiation defects; krypton ions; residual stress.

Introduction

Recently, nuclear power has been one of the most efficient sources of electricity [1]. With the development of technological progress, the need to increase the efficiency of nuclear reactors is also growing. Thus, the development of new generation IV nuclear reactors requires the use of materials with high mechanical properties at elevated temperatures and resistance to radiation exposure (when interacting with nuclear reaction products) [2].

Currently known austenitic steels are not suitable as new reactor materials due to their strong radiation swelling, and in the case of ferritic-martensitic steels, creep resistance and embrittlement at irradiation temperatures above 550 °C remain unresolved problems [3–5]. Therefore, the issue of developing new radiation-resistant materials is currently relevant for the world's research laboratories.

High-entropy alloys (HEA) based on a single-phase solid solution and a large number of basic elements in equimolar or almost equimolar ratios are promising for obtaining radiation-resistant materials for nuclear power [6]. The HEA includes alloys consisting of five or more elements with a concentration from 5 to 30 at. %.

It is believed that the maximisation of the configuration entropy of the HEA contributes to the formation of a single-phase disordered solid solution instead of the precipitation of complex intermetallic phases, as a result of which the alloy has a simple structure with improved properties compared to traditional alloys [7–9]. Numerous studies have shown that HEA have a high elastic limit, fatigue strength, thermal and corrosion resistance, creep resistance, radiation resistance [7; 10]. The properties of these alloys are associated with four main features: high entropy, greater deformation of the crystal lattice compared to traditional metals and alloys, multi-element composition and delayed diffusion [10]. A high degree of chemical disorder and lattice distortion in HEA increase the scattering of electrons and phonons, which leads to a decrease in thermal and electrical conductivity. The consequence of this is a slowing down of energy dissipation during the collision cascade and an increase in the duration of the thermal burst, which increases recombination between vacancies and interstitials [11–13]. In addition, the energies of formation and migration of vacancies and interstitial atoms have a wider energy distribution, which also increases the recombination of defects [14–16]. Due to the complexity of the composition, impurity-defect clusters formed by the interaction of point defects move along a chaotic trajectory, unlike directional movement in simple metals. This leads to an increase in the number of internode clusters in the area enriched with vacancies, which increases the recombination of defects [17].

However, at the moment, most of the researches of radiation damage of HEA were aimed at studies of face centred cubic structure HEA based on transition metals (Co – Cr – Fe – Mn – Ni). High-entropy alloys based on refractory elements of the 4, 5, 6 alloy group show considerable potential for structural applications [18]. Thus, according to the literature, the main elements included in the composition of refractory materials are molybdenum and tantalum due to their excellent high-temperature strength and titanium due to its high ductility [19]. Research of alloys based on the NbTaV, where X is Ti, W, Mo, shown that these alloys have high strength, ductility and oxidation resistance [20; 21]. The alloy TiVNbTa shows excellent compressive mechanical properties at room temperature (σ_y = 1273 MPa) and elevated temperature (σ_y decreases to 688 MPa when the temperature reaches 900 °C) [22]. Alloys based on the Ti – V – Cr – Zr – Nb system have also considered as high temperature, structural materials which exhibit low densities and high hardness [23]. Therefore, refractory HEAs (which contain Ti, Zr, Hf, Ta, V, Nb, W, and Cr) are one type of contender because they display exceptional high melting points, ductility, and strength at elevated temperatures [24–26].

The purpose of this work is to study the structural and phase state of binary, triple and quadruple systems of concentrated solid solutions based on the V – Nb – Ta – Ti system irradiated by low-energy krypton ions.

Materials and experimental details

Concentrated solid solutions based on the V – Nb – Ta – Ti system, specifically V, VNb, VNbTa, VNbTaTi were manufactured at the Beijing Institute of Technology. Samples were synthesised using high purity metals (>99.9 %) by arc melting and casting in a copper cell, followed by homogenisation. Then vacuum annealing was carried out for 24 and 72 h at the temperature of 1150 °C followed up cold rolling up to 85 % reduction in thickness.

The samples were irradiated at a DC-60 heavy ion accelerator located in Astana (Kazakhstan). The implantation was performed at room temperature with krypton ions, since krypton is one of the fission products of uranium. The energy Kr^{14+} ions were 280 keV and irradiation fluence was $5 \cdot 10^{15}$ cm^{-2}.

Changes in the structure and phase composition after irradiation were evaluated by X-ray diffraction analysis on a Ultima IV diffractometer (*Rigaku*, Japan) in the geometry of a parallel beam using copper radiation (λ = 0.154 18 nm). To study only the surface layer implanted with krypton, samples were taken at a fixed small angle of incidence of X-rays (1°) [27]. In this geometry, the penetration depths of X-rays for V, VNb, VNbTa, VNbTaTi alloys were 284; 146; 72 and 62 nm, respectively [27]. To exclude the influence of the texture of the alloys, the survey was carried out with a constant rotation of the sample at a speed of 30 rps. The effect of irradiation on the structure of the samples was studied by changes in macrostresses ($\sin^2\psi$ method) and microstresses (Halder – Wagner method) [28; 29].

The distribution of elements in the near-surface layer and its morphology were studied using Rutherford backscattering spectroscopy (RBS) and proton induced X-ray emission (PIXE) on a DC-60 accelerator, scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDX) on a scanning electron microscope LEO-1455VP (*Carl Zeiss*, Germany). Rutherford backscattering spectroscopy was conducted using an accelerated $^{14}N^{2+}$ beam with an energy of 1 MeV per nucleon.

The calculation of energy losses was carried out in the *SRIM-2013* program using the Kinchin – Pease model [30]. Figure 1 shows the distribution profiles of implanted Kr^{14+} ions and the results of modelling radiation damage (measured in displacement per atom, dpa). The maximum range of krypton ions was about 200 nm with the maximum damage for vanadium at a depth of 80 nm. The highest value of the damaging dose is 12.6 dpa for vanadium and 13.5 dpa for VNbTaTi. The concentration of implanted Kr^{14+} ions does not exceed 1 %.

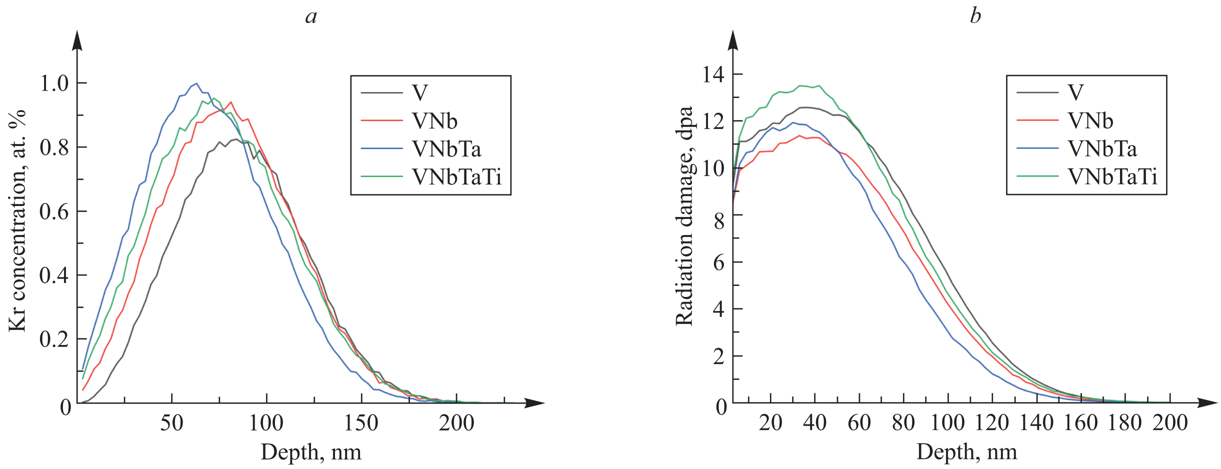


Fig. 1. Profiles of the distribution of implanted Kr^{14+} ion (*a*)
and the damaging dose (*b*) in samples V, VNb, VNbTa, VNbTaTi by depth

Results and discussions

Table 1 shows the results of the elemental composition analysis calculated by the PIXE and EDX methods. As can be seen from the table for binary, triple and quadruple systems, an equiatomic (within an error of 5–6 %) distribution of elements over the depth was confirmed by the EDX method, and the equiatomic and homogeneous distribution of elements over the surface was confirmed by the PIXE method.

Table 1

Results of the elemental composition in the initial samples of the V – Nb – Ta – Ti system calculated by the PIXE and EDX methods

Sample	Chemical element	Concentration, at. %	
		PIXE method	EDX method
V	V	100.0	100.0
VNb	V	50.0	49.5
	Nb	50.0	50.5
VNbTa	V	32.0	33.9
	Nb	33.0	34.2
	Ta	35.0	31.9
VNbTaTi	V	24.5	23.6
	Nb	25.5	26.1
	Ta	26.0	25.9
	Ti	24.0	24.5

Analysis of images of samples by the SEM method showed the uniformity of the surface structure of the samples V, VNb, VNbTa and VNbTaTi (fig. 2). The results of the study of the distribution of elements revealed a homogeneous distribution (within an error of 5–6 %) of elements over the surface of these samples (fig. 3). Small deviations from the uniformity of the distribution of elements are probably associated with the grain structure of materials.

According to the literature data, the equiatomic composition of multicomponent solid solutions may indicate the formation of single-phase solid solutions [8]. The study carried out by X-ray diffraction analysis confirms this assumption (fig. 4).

The general appearance of the X-ray diffraction pattern of non-irradiated binary, triple, and quadruple alloys is characterised by a slight asymmetry of the diffraction lines which is due to the possible heterogeneity of the structure due to the local heterogeneity of the alloy elements with different atomic radii of the elements and typical for multicomponent high-entropy alloys. As can be seen from fig. 4, all samples are single-phase solid solutions with a body-centered cubic lattice. The lattice parameter for samples V, VNb, VNbTa, VNbTaTi increases with increasing complexity of the composition of the systems and is 0.3027; 0.3177; 0.3227; 0.3234 nm, respectively. The growth of the lattice parameter is associated with an increase in the atomic radius of the elements in the composition.

Diffractograms of V, VNb, VNbTa and VNbTaTi samples irradiated by krypton ions show that the phase composition does not change (no decay of solid solutions has been detected). However, there is a more pronounced asymmetry of peaks and their displacement towards smaller angles compared to the original diffractograms, which indicates deformation of the crystal lattice in the near-surface region caused by irradiation (fig. 5).

Irradiation does not lead to a significant change of the equiatomic distribution of elements, as evidenced by the results of EDX and PIXE (table 2). According to the results of RBS, krypton is observed only in pure vanadium and both its concentration and depth agree with the results obtained in the program *SRIM-2013*. Krypton was not detected on the other alloys, possibly due to the low concentration and close atomic numbers of krypton and neobium.

Table 2

Results of the elemental composition in irradiated samples of the V – Nb – Ta – Ti system calculated by the PIXE and EDX methods

Sample	Chemical element	Concentration, at. %	
		PIXE method	EDX method
V	V	100.0 99.2*	100.0
VNb	V	50.0	49.7
	Nb	50.0	50.3
VNbTa	V	29.0	36.3
	Nb	35.0	31.2
	Ta	36.0	32.6
VNbTaTi	V	21.0	25.2
	Nb	29.0	25.4
	Ta	29.0	24.3
	Ti	21.0	25.1

*At depth of 90 nm the highest concentration of krypton ions (0.8 at. %) is observed.

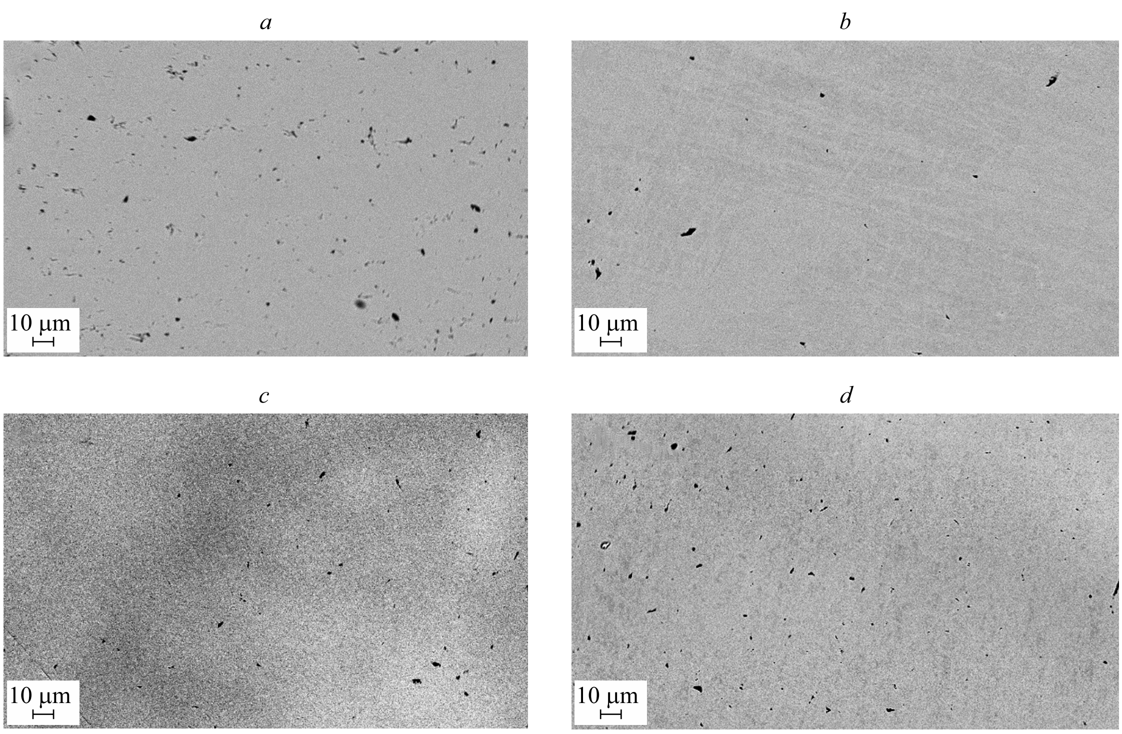


Fig. 2. Surface morphology in the initial samples surface V (*a*), VNb (*b*), VNbTa (*c*), VNbTaTi (*d*)

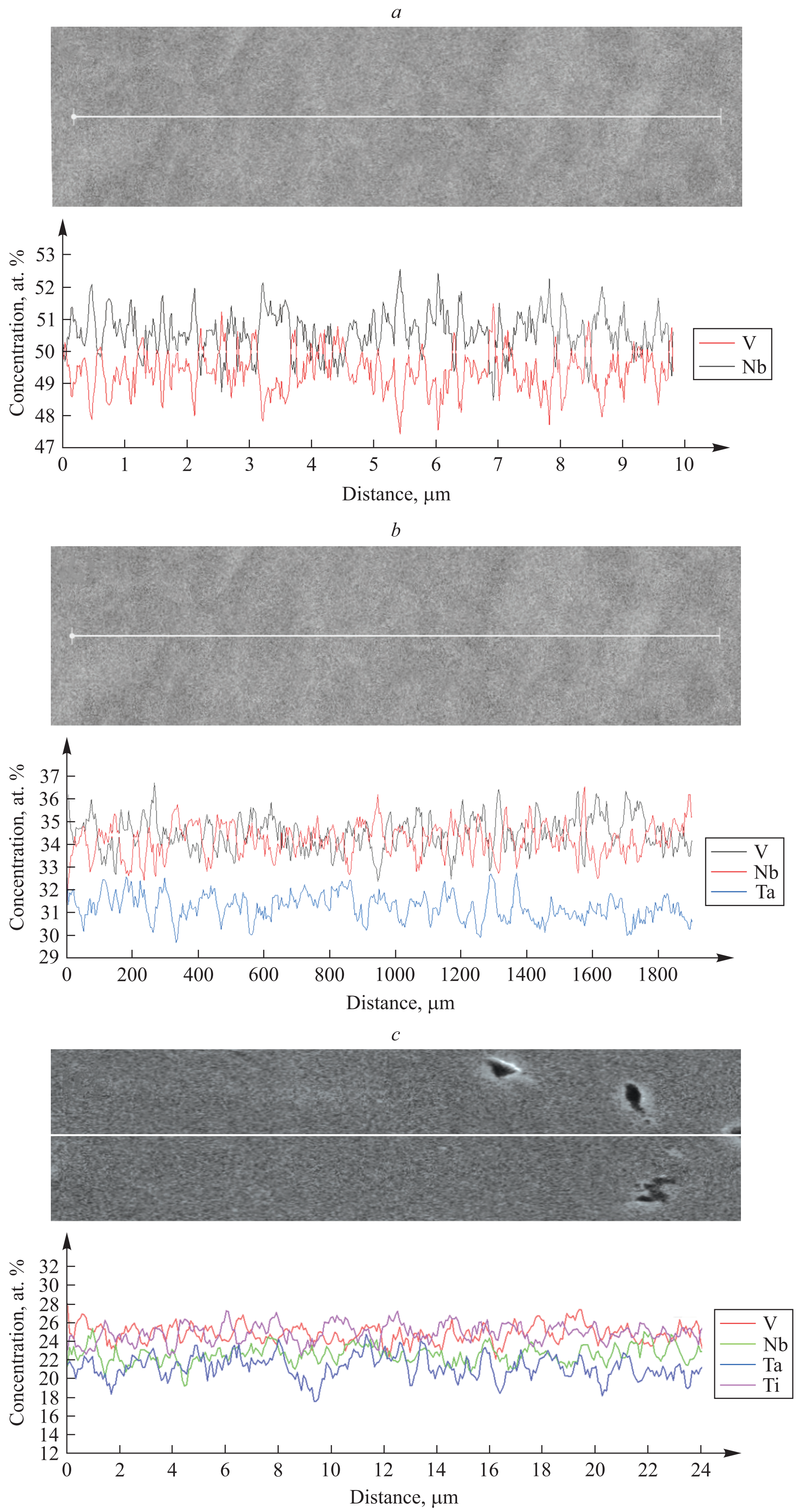


Fig. 3. Distribution profiles of elements in the initial samples VNb (*a*), VNbTa (*b*), VNbTaTi (*c*)

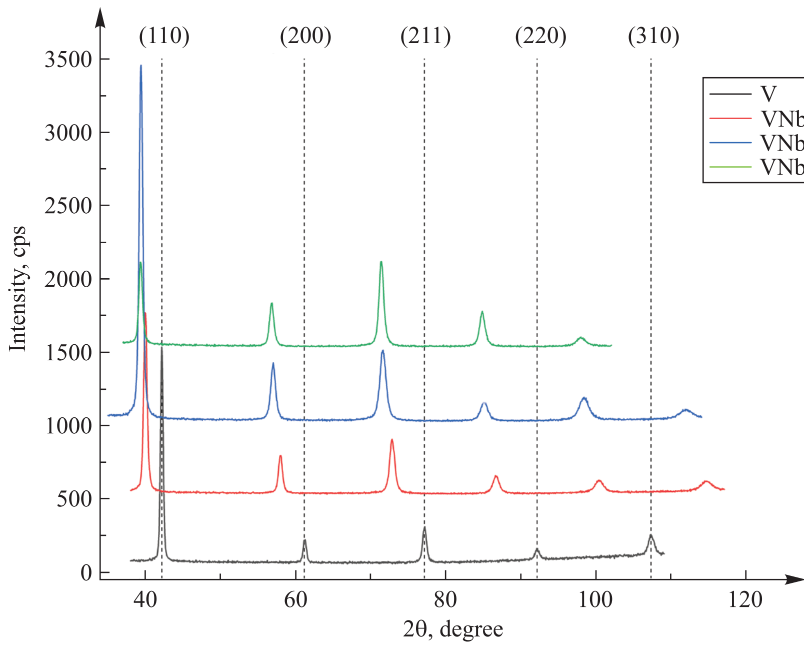


Fig. 4. X-ray diffraction pattern of the initial samples of the V – Nb – Ta – Ti system

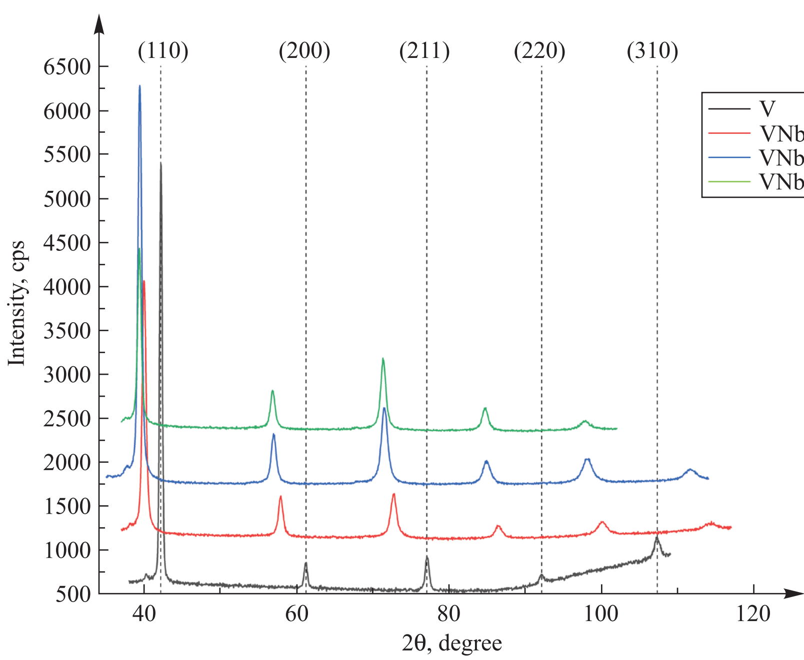


Fig. 5. X-ray diffraction pattern of samples of the V – Nb – Ta – Ti system irradiated by Kr^{14+} ions with an energy of 280 keV

In addition, after irradiation, there is no segregation of elements on the surface, as can be seen in the results of the SEM (fig. 6). As well as in the initial samples, a homogeneous distribution of elements over the surface is observed (fig. 7).

To quantify the effect of radiation damage on the structure of samples, changes in micro- and macrostresses relative to non-irradiated samples were calculated. Orientation (110) was used to determine macrostresses. The obtained dependences were approximated by a linear function to obtain stress values.

Figure 8 shows the values of the received stress. Compressive stresses prevail in all initial samples. The addition of niobium and tantalum in VNb and VNbTa samples leads to an increase in the level of compressive stresses, which is associated with a large atomic radius of these elements compared to vanadium. In the VNbTaTi alloy, due to the presence of Ti, which has a lower atomic radius, and also reduces the modulus of elasticity of the alloy, a lower level of compressive stresses is observed. Irradiation by krypton ions leads to a decrease in macrostresses, which can be explained by several reasons. Probably difference in atomic size between the elements leads to the reduction of electron and phonon mean free paths, which can affect the formation energy and migration barriers of defects in the material [31]. The atomic size difference of the elements in the solid solutions also contributes to an increase in atomic scattering and a decrease in the focused movement of interstitials along the close-packed direction, which prevents interstitials from moving quickly out of the region with a high concentration of vacancies. This promotes defect recombination in these alloys. Besides, lattice distortion can also reduce defect mobility therefore many of the interstitial clusters were stationary and remained in the region where defects were formed, resulting in a higher rate of defect recombination [32; 33]. As a result, a large number of interstitial clusters of small size are formed, which causes tensile stresses and leads to a decrease in the overall level of compressive stresses [34]. In contrast, an increase in compressive stresses is observed in microstresses, which may be due to radiation-stimulated diffusion of lighter elements to the boundaries of the coherent scattering region [35]. However, it is not possible to assess which of the elements is more stable at the moment. It is also hard to say with certainty that there is a dependence of stresses on the complexity of the composition.

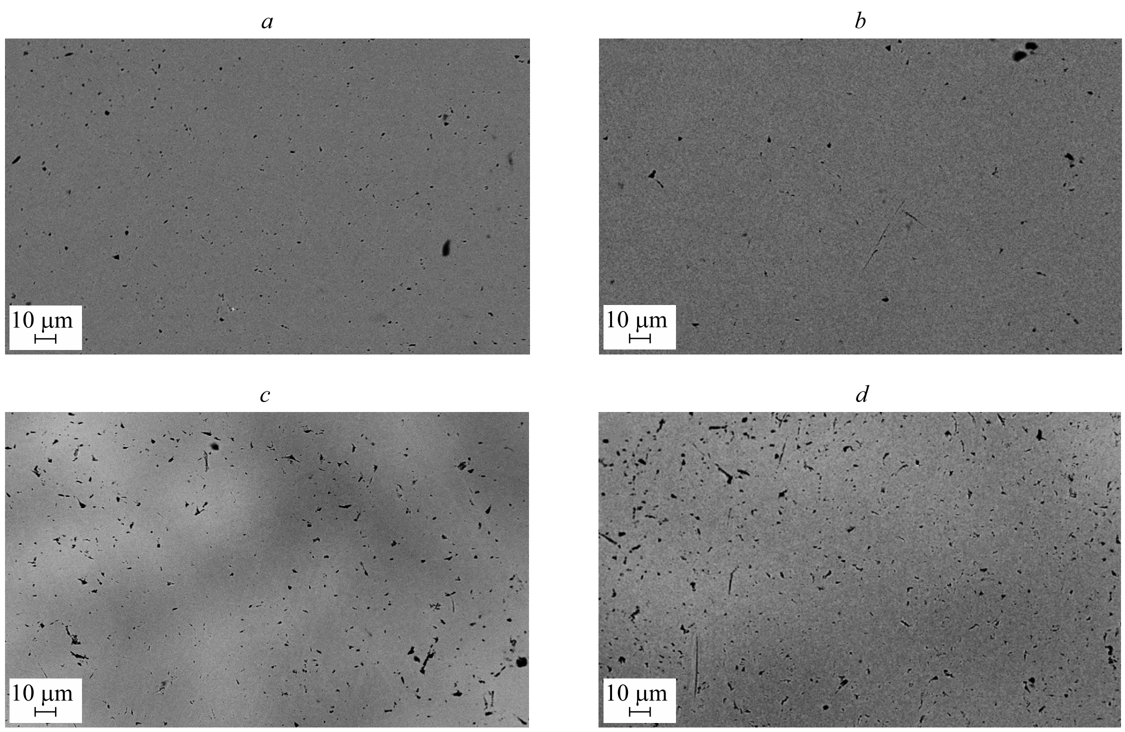


Fig. 6. Surface morphology in the samples surface
V (*a*), VNb (*b*), VNbTa (*c*), VNbTaTi (*d*) irradiated by Kr^{14+} ions with an energy of 280 keV

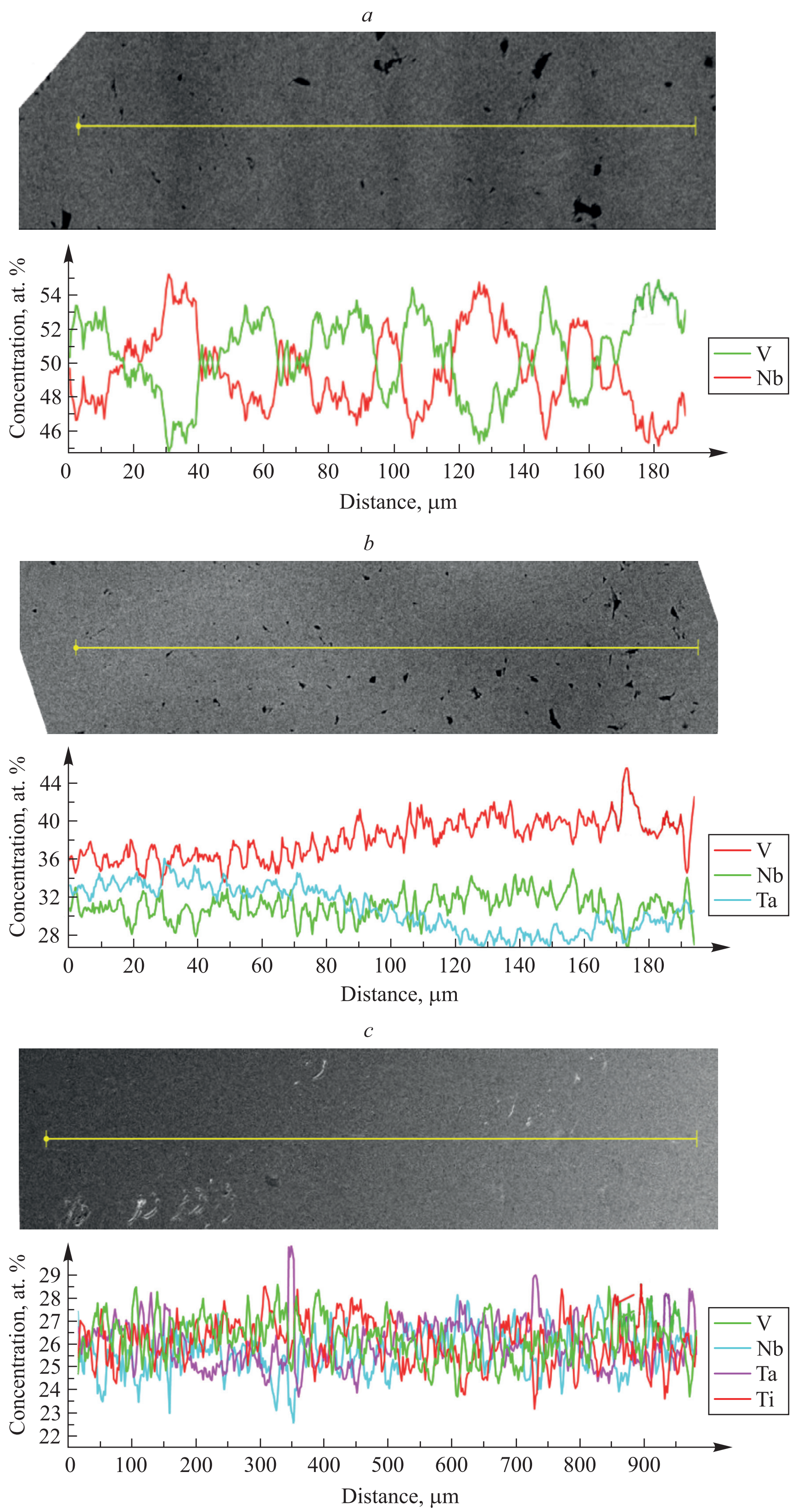
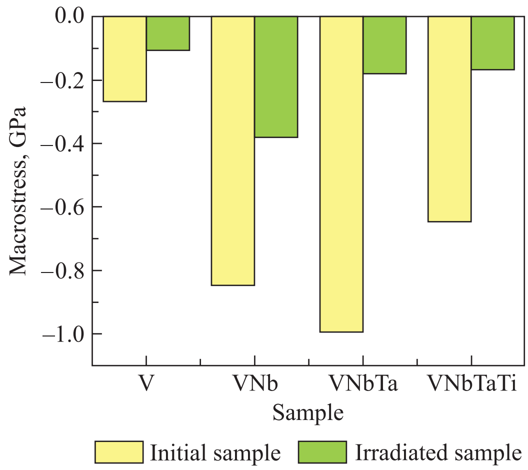


Fig. 7. Distribution profiles of elements
in the samples VNb (*a*), VNbTa (*b*), VNbTaTi (*c*)
irradiated with Kr^{14+} ions by an energy of 280 keV



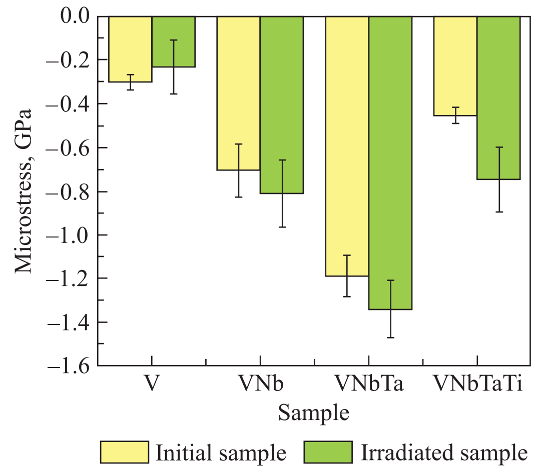


Fig. 8. Values of macrostresses (*a*) and microstresses (*b*) depending on the complexity of the composition of the V – Nb – Ta – Ti system for the initial and irradiated samples

Conclusions

Equiatomic single-phase binary, triple and quadruple solid solutions based on the V – Nb – Ta – Ti system with a body-centered cubic lattice were obtained by arc melting with subsequent homogenisation. Compressive stresses were detected in all initial materials. The addition of Nb and Ta to the alloy leads to an increase in the level of compressive stresses, while the addition of Ti leads to their decrease.

The phase composition and structure of the near-surface layer of binary, triple and quadruple solid solutions based on the V – Nb – Ta – Ti system are resistant to irradiation by krypton ions with an energy of 280 keV and a fluence of $5 \cdot 10^{15}$ cm^{-2}. Irradiation by krypton ions leads to the formation of tensile stresses of the first kind. There is an increase in microstresses in multicomponent solid solutions, which may be due to the radiation-stimulated diffusion of lighter elements to the boundaries of the coherent scattering region, which leads to an increase in microstresses due to the dimensional factor.

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