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Microstructural stability and mechanical properties of the system V-Nb-Ta-Ti under helium ion irradiation

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Abstract. The purpose of this work is to study the effect of irradiation with low-energy helium ions with an energy of 40 keV on the structural-phase state of multicomponent solid solutions based on V-Nb-Ta-Ti. This type of irradiation at fluence of 2×10^{17} He/cm^2 make it possible to simulate the effects occurring in a nuclear reactor. since during its operation, a large number of neutrons are released, causing nuclear reactions in the material with the formation of inert gases. As a result of the work carried out, it was found that all alloys of the system belong to equiatomic single-phase solid solutions and show a greater value of deformation of the crystal lattice and stress after irradiation compared with pure vanadium. It has been proposed that increasing the number of elements in the V-Nb-Ta-Ti system reduces the mobility of defects created by irradiation, which leads to the formation of smaller clusters and an increase in microdeformation.

Keywords: high-entropy alloys, SP-CSA, irradiation, residual stress, deformation

. Introduction

The development of nuclear power in the world places increased demands on the structural materials used, their production technology and performance control. Under the influence of irradiation, these materials undergo structural-phase transformations, which have a negative effect primarily on mechanical properties. High-entropy alloys (HEA) based on a single-phase solid solution and a large number (more than five) of basic elements in equimolar or almost equimolar ratios are promising for obtaining radiation-resistant materials for nuclear power. It is believed that the maximization 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 or second phases, as a result of which the alloy has a simple microstructure with improved properties compared to traditional alloys [1]. Numerous studies have shown that wind turbines have high elastic limit, fatigue strength, thermal and corrosion resistance, creep resistance, radiation resistance. The properties of these alloys are associated with four main effects: high entropy, large lattice deformation, multi-element composition and delayed diffusion [2].

2. Experimental setup

Multi-component solid solutions based on V-Nb-Ta-Ti, specifically V, VNb, VNbTa, VNbTaTi were manufactured at the Beijing Technological University. Samples were synthesized using high purity metals (>99.9%) by arc melting and casting in a copper cell, followed by homogenization. Then vacuum annealing was carried out for 24 hours and 72 hours at a temperature of 1150°C with cold rolling up to 85% reduction in thickness.

The samples were irradiated at a DC-60 heavy ion accelerator located in Nur-Sultan, Kazakhstan. Low-energy He^{2+} ions with an energy of 40 keV were chosen as the type of irradiation. The irradiation fluence was $2\cdot10^{17}$ cm^{-2}.

Changes in the structural and phase composition after irradiation were evaluated using the X-ray diffraction method, implemented using Rugaku Ultima IV diffractometer. To study the effect of implanted helium on the near-surface layer, samples were taken at a small fixed angle of incidence of X-rays (1°). The irradiation effect on the microstructural stability was determined by the changes in macrostrain and microstress, calculated by the Holder-Wagner method.

The mechanical properties of the samples were evaluated on a model 750 Ubi nanoindenter (Hysitron, USA) with a conical indenter with a radius of 226 nm and a taper angle of 60° by introducing an indenter with continuous registration of deformation curves. The determination of the average values of hardness H was carried out with constant load values at value 500 microns for 9 measurements for each sample. To determine the dependences of H on the depth of penetration into the sample, indentation of up to 1000 microns with partial unloading in 30 cycles was performed.

The effect of irradiation on the morphology of the surface and the spatial distribution of elements were carried out by scanning electron microscopy (SEM) and energy-dispersive X-ray analysis (EDX).

The calculation of energy losses was carried out in the SRIM 2013 program using the Kinchin-Pease model. Threshold displacement energies for elements V, Nb, Ta, Ti are 40, 78, 91 and 30 eV respectively [3]. Fig.1 shows the distribution profiles of implanted He^{2+} ions and the results of modeling radiation damage (measured in displacement per atom, dpa). The maximum range of helium ions was 275–325 nm with maximum damage for vanadium at a depth of 160–170 nm, and for VNbTaTi alloy – 120–140 nm. The highest value of the damaging dose is 3.5–6.5 dpa, depending on the samples. The concentration of implanted He^{2+} ions does not exceed 23%.

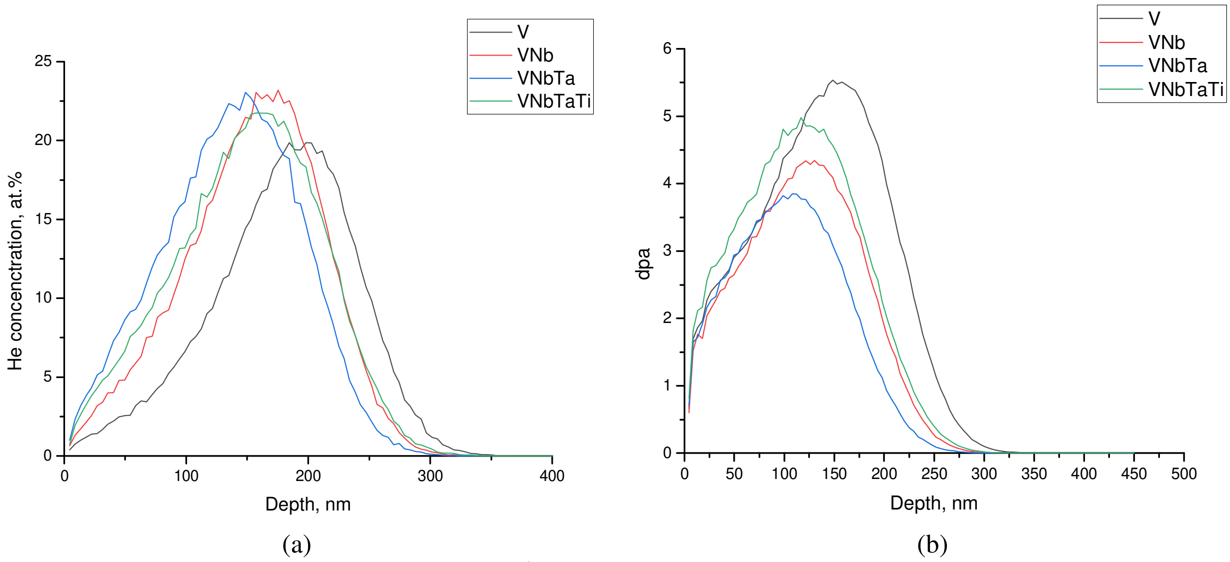


Fig.1. Profiles of the distribution of implanted He^{2+} ion (a) and the damaging dose (b) in samples V, VNb, VNbTa, VNbTaTi by depth.

3. Results and discussion

Fig.2 shows the X-ray diffractograms for non-irradiated samples. The general appearance of the diffractograms presented (narrow and intense diffraction peaks) indicates a high degree of crystallinity of the alloys; however, a slight asymmetry of the diffraction lines in more complex systems is associated with possible heterogeneity of the structure and with different atomic radii of the elements included in the alloy, which leads to deformation of the crystal lattices and is characteristic of high-entropy alloys. It can be seen that in all multicomponent solid solutions based on V-Nb-Ta-Ti, a single-phase solid solution with a BCC lattice is formed. The lattice parameter for samples V, VNb, VNbTa, VNbTaTi consistent with an increase in the average atomic radius of solid solutions and was 0.3027 nm, 0.3177 nm, 0.3227 nm, 0.3234 nm, respectively.

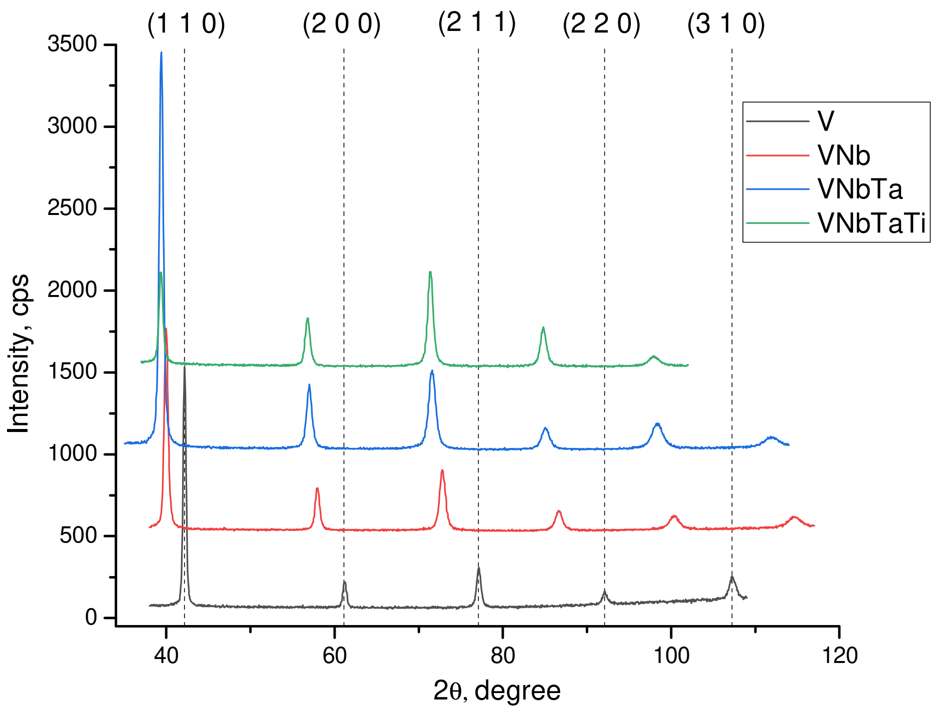


Fig.2. X-ray diffraction pattern of the initial samples of the V-Nb-Ta-Ti system.

Fig.3 shows the results of scanning electron microscopy and EDX analysis. It can be seen that in all the initial samples of the V-Nb-Ta-Ti system, they have a homogeneous distribution of elements over the surface. The grain size for 3- and 4-component systems is 100–200 nm.

Fig.4 shows diffractograms of samples irradiated with helium ions. The phase composition of the system has been preserved, but there is a more pronounced asymmetry of the peaks and their displacement towards smaller angles, which indicates deformation of the crystal lattice caused by irradiation.

After irradiation, there is no segregation of elements on the surface and violation of the equiatomic distribution of elements, as can be seen in the results of SEM and EDX (Fig.5). As with the initial samples, a homogeneous distribution of elements over the surface is observed. Small fluctuations of elements are associated with the dendritic structure of alloys due to insufficient homogenization during preparation.

To quantify the effect of radiation damage on the structure of the samples, the relative change in the lattice parameter (lattice deformation) and its volume relative to non-irradiated samples was calculated, as shown in Fig.6a. As can be seen from the figure, with an increase in the number of elements in the alloy, the deformation and relative volume of the crystal lattice increase. At the same time, with an increase in the complexity of the composition of alloys, the slope of this dependence becomes less gentle. In addition, Fig.6b shows the values of microdeformation for the initial and irradiated samples based on the V-Nb-Ta-Ti system, where it should be noted an increase in microstresses in more complex systems, both in the initial samples and in those irradiated with helium ions.

It can be assumed that in multicomponent solid solutions based on V-Nb-Ta-Ti, with increasing complexity of the composition, the mobility of point radiation defects decreases and their recombination increases. This leads to suppression of the growth of helium-vacancy clusters [4, 5] and, as a consequence, helium bubbles, reducing their size and increasing their density. This leads to an increase in the stress level in multicomponent alloys [6].

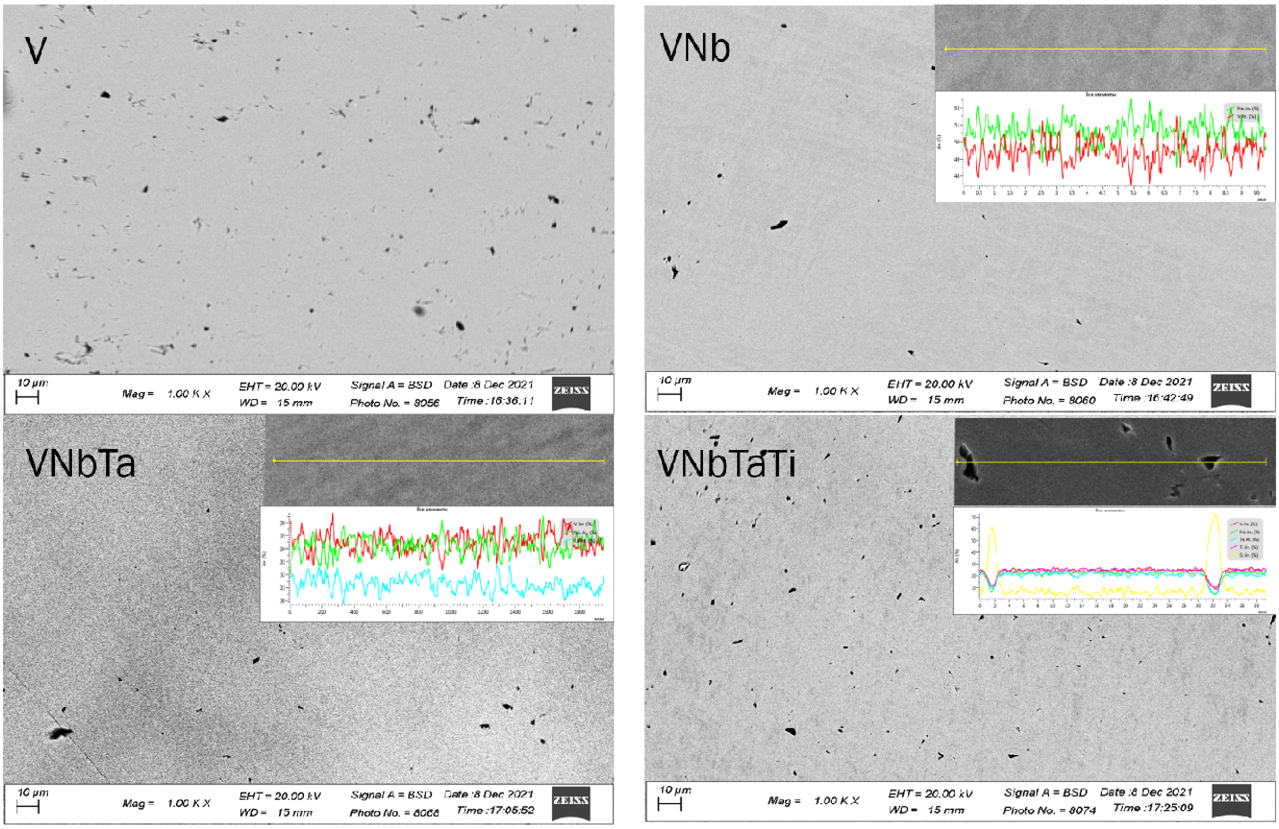


Fig.3. Surface morphology and distribution profiles of elements in the initial samples of the system.

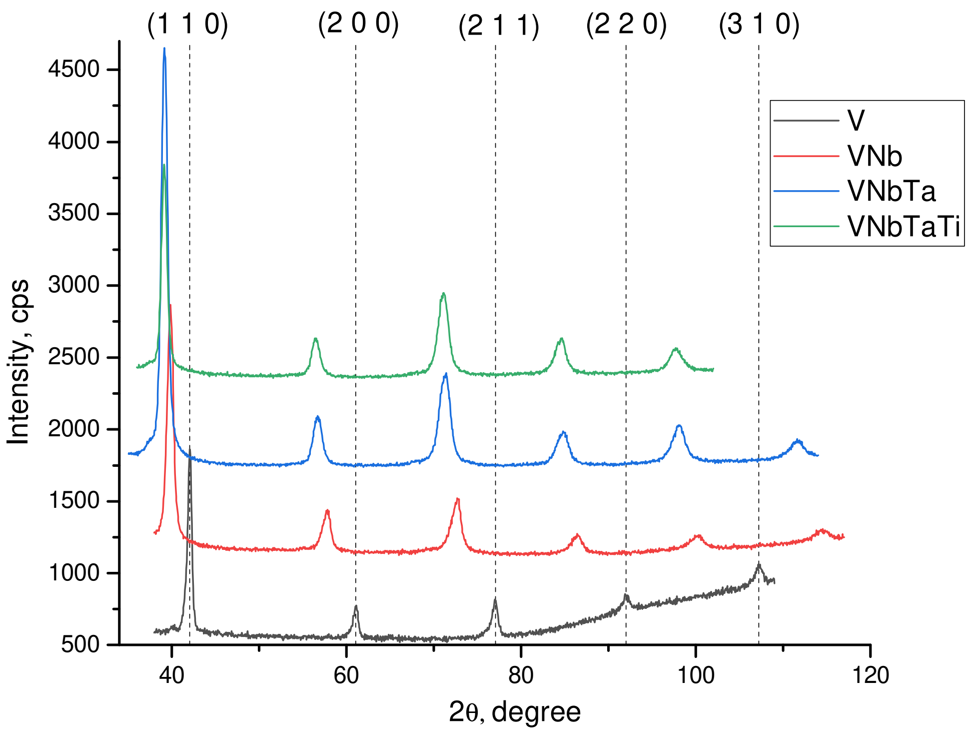


Fig.4. X-ray diffraction pattern of the samples of the V-Nb-Ta-Ti system, irradiated with helium ions.

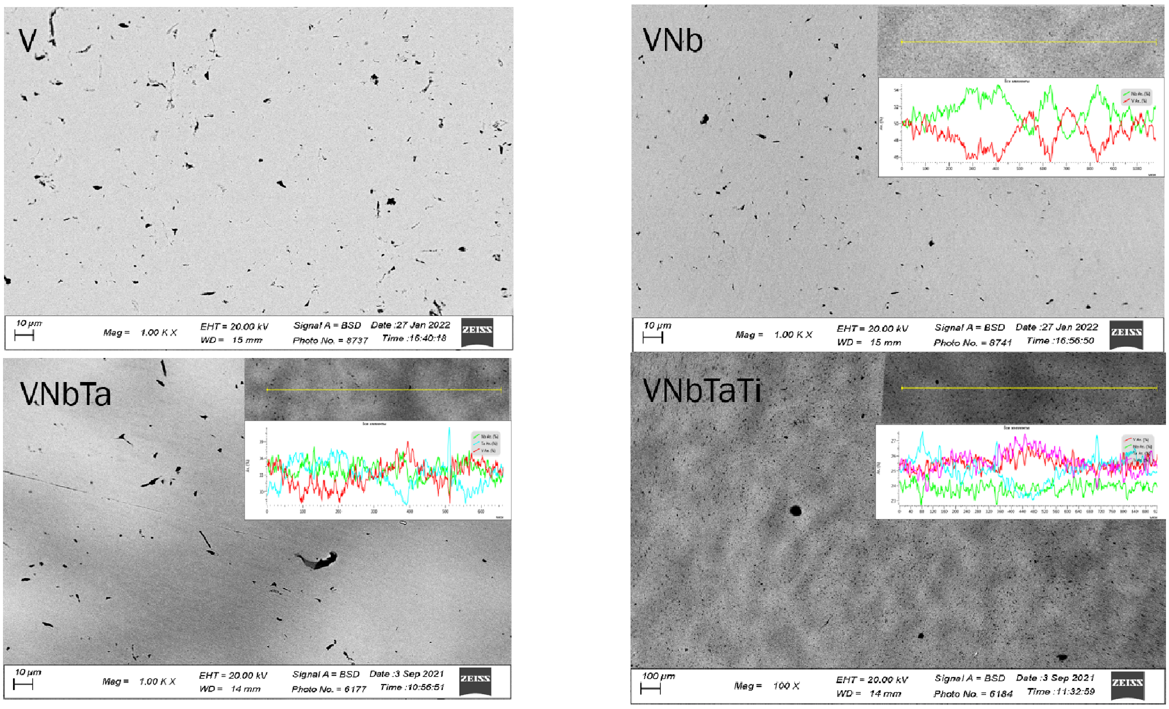
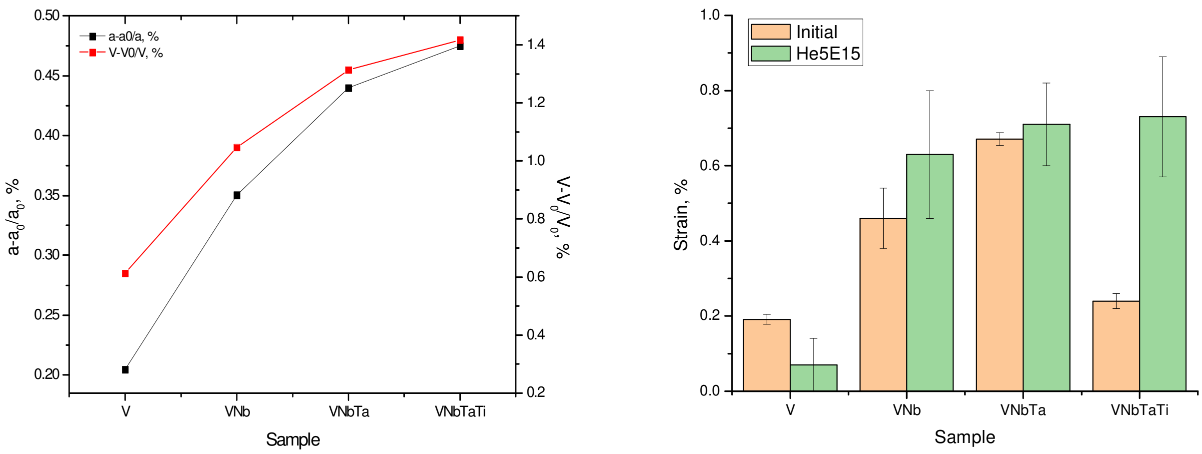


Fig.5. Surface morphology and distribution profiles of elements in the initial samples of the system.



a b

Fig.6. The relative change in the parameter and volume of the crystal lattice for the V-Nb-Ta-Ti system irradiated with He^{2+} ions (a) and the values of microstresses (stresses of the second kind) on the complexity of the composition of the V-Nb-Ta-Ti system for the initial and irradiated samples (b).

One of the important parameters affecting the applicability of alloys as structural materials are strength characteristics, in particular, the values of hardness. This characteristic makes it possible to access with high accuracy the resistance of materials to external influences, as well as the structural deformations and disordering caused by them.

An increase in nanohardness is observed in the initial samples from V to VNbTa (Fig.7). Such an increase in hardness for two and three-component alloys is in good agreement with the theory of high-entropy alloys, according to which the addition of niobium or tantalum to the composition leads to an increase in the strength properties, as well as the density of the alloys. In turn, the addition of titanium to the alloy led to a decrease in the hardness values in the initial state. In the literature [7], a change in the elastic properties of high-entropy alloys is associated with solid-solution hardening and a change in distortion. The distortion for VNb, VNbTa, VNbTaTi alloys decreases and amounts to 4.25%, 3.95% and 3.61%, respectively. Therefore, for the V-Nb-Ta-Ti system, distortion does not affect the change in mechanical properties. It was assumed that the decrease in H in the VNbTaTi alloy is associated with the addition of Ti to the system, since it has a lower melting point and is more ductile compared to other elements.

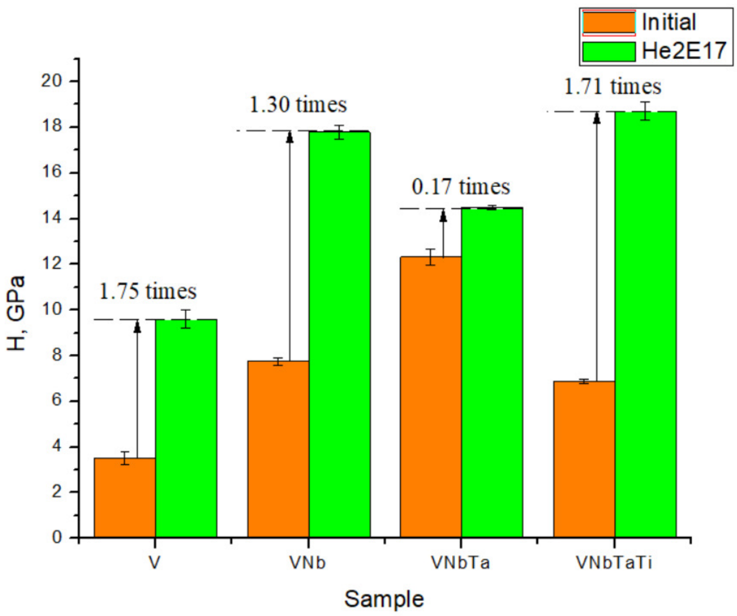


Fig.7. Diagram of changes in hardness before and after irradiation.

After irradiation with helium ions, an increase in nanohardness is observed in all samples of the system V-Nb-Ta-Ti. Alloys based on VNb and VNbTa have the highest resistance to an increase in hardness. The nanohardness in the alloy VNbTaTi has increased significantly compared tocomparison with VNb and VNbTa alloys due to the softening properties of titanium when it is added to high-entropy alloys.

4. Conclusion

Thus, it was established that equiatomic single-phase multicomponent solid solutions based on the V-Nb-Ta-Ti system were obtained by arc melting with subsequent homogenization. The phase composition of a multicomponent solid solution based on V-Nb-Ta-Ti is resistant to irradiation with helium ions with an energy of 40 keV and a fluence of $2 \cdot 10^{17}$ cm^{-2}. It has been shown that after irradiation, multicomponent solid solutions have a greater deformation compared to single-component ones. It has been proposed that increasing the number of elements in the V-Nb-Ta-Ti system reduces the mobility of defects created by irradiation, which leads to the formation of smaller clusters and an increase in microdeformation. The change in mechanical properties (hardness) does not always depend only on the number of elements that make up multicomponent solid solutions, and is also related to the properties of individual elements that make up alloys.

5. References

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