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STABILITY OF THIN QUASI-CRYSTALLINE Ti-Zr-Ni FILMS AND RELATED CRYSTALLINE PHASES UNDER LOW-ENERGY TRANSIENT PLASMA IRRADIATION

S.V. Malykhin[1], V.V. Kondratenko[1], V.A. Makhlai[1,2,3], I.E. Garkusha[2,3], I.A. Kopylets[1], Yu.S. Borisov[1], S.S. Herashchenko[2,3], S.V. Surovitskiy[1], S.S. Borisova

[1]National Technical University "Kharkiv Polytechnical Institute", Kharkiv, Ukraine;
[2] Institute of Plasma Physics, National Science Center
"Kharkov Institute of Physics and Technology", Kharkiv, Ukraine;
[3]V.N. Karazin Kharkiv National University, Kharkiv, Ukraine

E-mail: malykhin@kpi.kharkov.ua

The properties of Ti41Zr38.3Ni20.7 thin films under radiation-thermal action of hydrogen plasma with a surface heat load of 0.2 MJ/m^2 was studied at the QSPA Kh-50 quasi-stationary plasma accelerator (NSC KIPT). The phase composition, structural state, and surface morphology were studied using X-ray diffraction and scanning electron microscopy. It was found that the quasicrystalline phase and related crystalline phases, the Laves phase, the α-solid solution, and the 2/1 phase of the Ti-Zr-Ni approximant crystal were stable under irradiation with up to 20 hydrogen plasma pulses. The phase composition did not change. It is shown that the changes in the coatings mainly manifest themselves as changes in the substructure of the observed phases. With an increase in the plasma exposure dose, the structure of the quasicrystalline icosahedral phase improves, and the size of the coherence regions increases. In the films consisting of crystalline phases, a partial phase transformation is observed with a redistribution of components between the 2/1 phase of the approximant crystal and the α-solid solution phase. It was found that thin films of the Ti-Zr-Ni system containing a quasicrystalline icosahedral phase, irradiated with radiation-thermal plasma pulses, are less prone to cracking than coatings with crystalline phases of the same system.

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INTRODUCTION

A feature of the structure of icosahedral quasicrystals (QCs) is the combination of rotational symmetry of the fifth order with a strict aperiodic long-range order at the disposal of atoms in the absence of translational invariance [1]. Therefore, QCs are characterized by anomalous and unique physical properties, in particular, high strength and low thermal conductivity [2]. For icosahedral quasicrystals of the Ti-Zr-Ni system, the ability to accumulate a large (up to 2 H/at.) amount of hydrogen in the form of a solid solution is known [2-4]. The lack of periodicity implies increased stability under radiation and thermal exposure [5-7]. The widespread use of quasicrystals in bulk and ribbon forms is hindered by their high fragility. It is believed [8] that the use of thin-film coatings on metal substrates solves this problem. We assume that the film quasicrystalline Ti-Zr-Ni coating can perform the functions of thermal protection, prevention of hydrogen embrittlement, and resistance to blistering. Steel elements with quasicrystalline coatings can be used as structural-functional elements of a fusion reactor. Previously, we worked out a laboratory technique for the formation of coatings with an icosahedral quasicrystalline phase with a fairly perfect structure. The features of the formation of thin-film coatings with quasicrystalline and crystalline phases of the Ti-Zr-Ni system are described in detail in [9, 10]. According to the first experiments on the modification of crystalline Ti-Zr-Ni phases, the formation of the quasicrystalline and crystal-approximant phases occurs as a result of high-speed quenching under pulsed action with a heat load of 0.6 MJ/m^2. The changes in the contents of these phases as well as in their structure and substructure parameters were studied during isothermal vacuum annealing at a temperature of 550 °C and also after irradiation with 5 plasma pulses in the range of heat loads from 0.1 to 0.4 MJ/m^2. The quasicrystalline phase was found to be resistant to irradiation with hydrogen plasma [11, 12].

In this work, the aim is to analyze and compare the behavior of thin Ti-Zr-Ni films containing the quasicrystalline and crystalline phases under low-energy plasma irradiation.

1. SAMPLES AND INVESTIGATION TECHNIQUE

Coatings with a thickness of 5.7 μm were prepared by dc-magnetron sputtering of a Ti41Zr38.3Ni20.7 (at.%) target. Austenitic steel 12H18N10T was used as a substrate. The substrate temperature during deposition did not exceed 40...50 ºC. After deposition, the samples were annealed in a vacuum chamber with a limiting pressure of about $1\cdot10^{-4}$ Pa. Two samples were used in the experiment. Sample 1 was annealed at a temperature of 500 ºC for 4 hours, and sample 2 – at a temperature of 700 ºC for 3 hours. The annealing modes were chosen based on the data of our previous studies [9, 10] so that the quasicrystalline phase was formed in the first sample and crystalline phases – in the second. The samples were irradiated with fluxes of hydrogen plasma on a QSPA Kh-50 quasi-stationary plasma accelerator (NSC KIPT). The main parameters of the QSPA plasma fluxes were as follows: ion energy of about 0.4 keV, a maximum plasma pressure of 0.32 MPa, and a flux diameter of about 18 cm. The pulse duration was 0.25 ms. The maximum number of pulses was 20. The thermal load on the irradiated surface was chosen equal to 0.2 MJ/m^2, which did not imply melting of the sample surface.

The structure and phase composition were investigated by the XRD method. The measurements were carried out on a DRON-type apparatus in filtered Cu-Kα radiation. The spectra were processed using the New_Profile 3.5 software package. The quasicrystalline phase was identified and its quasicrystalline parameter a_q was determined according to J.W. Cahn [13]. Crystalline phases were identified using the JCPDS card index [14] and the PowderCell software package. The study of surface morphology and elemental microanalysis were carried out using scanning electron microscopy (SEM) on a JEOL JSM-6390 device.

2. RESULTS AND DISCUSSION

2.1. CHARACTERIZATION OF THE INITIAL STATE

The X-ray diffraction patterns of samples in the initial state are presented in Fig. 1. Processing of diffraction patterns and subsequent phase analysis showed that sample № 1 (after annealing at 500 °C) consists of a single icosahedral quasicrystalline phase (*i*-phase, *i*-QC) (Fig. 1,a). Its reflections in the figure are marked with two indices by J. Kahn. The quasi-crystallinity parameter is a_q = 0.5234 nm, and the half-width (B) of the (20, 32) reflection is about 17.4 μrad. One of the proofs that this is really a quasicrystalline phase is the presence of multiple orders of reflections, for example (20, 32) and (52, 84), in which the interplanar distances differ strictly by the "golden number" $\tau = 1.618$.

The phase composition of sample № 2 after annealing at a higher temperature turned out to be more complex. It was found that the sample contains three phases. All of them are crystalline. These are the Laves (*L*), $(Ti,Zr)_2Ni$ phase with the C14structural type, the α-Ti(Zr) solid solution phase with approximately equal titanium and zirconium contents, and the 2/1 crystal-approximant phase (Fig. 1,b). The existence of the first two phases is in full agreement with the data of the phase diagram given in [15-17]. The existence of the 2/1 crystal-approximant phase in the system under consideration was first established by us in [9]. The crystal lattice parameters of the phases are: a = 0.310 nm and c = 0.495 nm for the α-phase; a = 0.5282 nm and c = 0.8304 nm for the *L*-phase; and 2.3321 nm for the 2/1 approximant phase. In Fig. 1,b, reflections from phases are labeled as *L*, α, and 2/1 for the Laves phase, solid solution, and approximant respectively.

2.2. PLASMA IRRADIATION RESULTS

The change in the shape of the diffraction pattern of sample № 1 as a result of irradiation with hydrogen plasma is shown in Fig. 2. Fig. 2,a represent the section of diffraction patterns obtained for sample № 1 in the initial state, Fig. 2,b – after irradiation with 0.2 MJ/m^2 with 5 pulses, Fig. 2,c – after10 pulses, and Fig. 2,d – 20 pulses. It can be seen from the figure that the number and intensity of reflections from the quasicrystalline phase increase with an increase in the plasma irradiation dose.

In addition, a redistribution of intensity between reflections (18, 29) and (20, 32) is observed. The greater the number of irradiation pulses *N*, the higher the reflection intensity (18, 29). This ratio of intensities is consistent with the theoretical calculation performed in [13], and it is associated with the number of equivalent crystallographic planes (repeatability factor). In the literature [18-20], however, for bulk and ribbon samples, different data are given for the intensity ratio $I_{(18,29)}/I_{(20,32)}$. It can be assumed that this discrepancy is due to the presence of texture in the samples associated with the method of preparation. Also, the redistribution of the intensity between reflections during cyclic heating-cooling can be associated with changes in the preferred orientation of quasicrystalline grains. This is also further evidenced by an increase in the number of reflections and an increase in their intensities. We note that irradiation does not lead to the appearance of reflections not characteristic of the *i*-phase; that is, the *i*-phase turns out to be resistant to irradiation with a thermal load of 0.2 MJ/m^2.

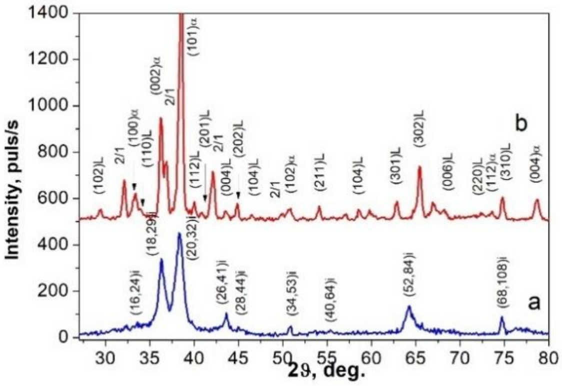

Fig. 1. X-ray diffraction patterns recorded in the Cu-Kα radiation for sample № 1 annealed at a temperature of 500 ℃ (a) and sample № 2 annealed at a temperature of 700 ℃ (b) in the initial state (before irradiation)

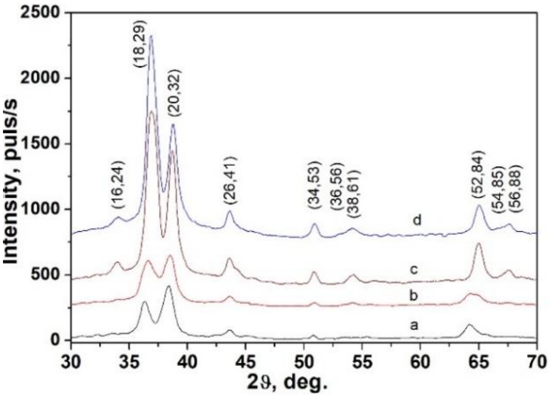

Fig. 2. Section of diffraction patterns from sample № 1 in the initial state (a) and after irradiation with 0.2 MJ/m^2 with 5 pulses (b), 10 pulses (c), and 20 pulses (d)

With the accumulation of the number of irradiation pulses, the positions of the reflections shift and their width decreases. The reflections are displaced towards larger diffraction angles. The value of the displacement, recalculated into the change in the value of the quasicrystallinity parameter, is shown in Fig. 3.

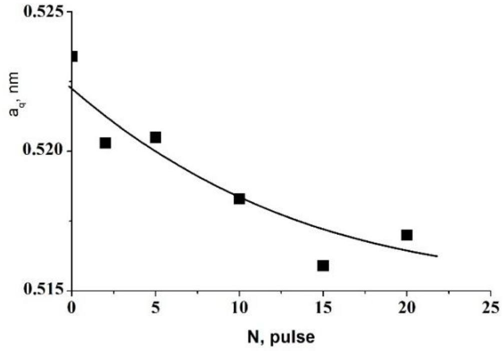

Fig. 3. Change in the quasicrystallinity parameter with accumulation of the number of pulses under irradiation with hydrogen plasma of sample № 1

It can be concluded that, with the accumulation of the number of irradiation pulses, a monotonic and rather significant decrease in the quasicrystallinity parameter a_q occurs. One of the possible reasons for the decrease in the parameter could be a change in the elemental composition of the phase. However, according to the results of microprobe analysis, changes in the elemental composition of the films do not exceed ± 2 at.%, which is comparable with the measurement accuracy. Another reason for the decrease in the parameter a_q may be the accumulation of vacancies of thermal and deformation nature [21]. According to calculations [22, 23], the reason for their formation may be the thermal cycling of samples in the temperature range from room temperature to 850...900 °C. We believe that the radiation component does not play a significant role, and this issue was studied earlier [11].

The changes in the width of (18, 29) and (20, 32) reflections are shown in Fig. 4, squares and circles respectively. It can be observed that for a small (up to 5) number of irradiation pulses *N*, the width practically does not change, and at larger values of *N*, the width of the reflections decreases. Since the chosen reflections are located at small diffraction angles and, therefore, weakly depend on the content of phason defects [13, 24], we assume that the decrease in width is associated with an increase in the coherence length (CL) as a result of thermal action. The attainable absolute reflection width of ≈ 11 μrad corresponds to CL of approximately 15 nm.

The changes in the diffraction patterns of sample № 2 with an increase in the number of plasma irradiation pulses, are shown in Fig. 5, in particular Fig. 5,a – in the initial state, Fig. 5,b – after irradiation with 0.2 MJ/m^2 with 2 pulses, Fig. 5,c – after 15 pulses, and Fig. 5,d – 20 pulses. For the present phases, the irradiation leads to a relative change in the intensity of reflections, a shift in their positions, and a change in width. Reflection indices are given in Fig. 1. The largest increase in the reflection intensity and width is observed for the α-Ti (Zr) solid solution phase. For the Laves phase (*L*), a relative decrease in the reflection intensity is observed, while for the phase of the crystal approximant 2/1, there is a slight increase. Changes in the positions of the reflections, recalculated into the parameters of the crystal lattices, are shown in Fig. 6.

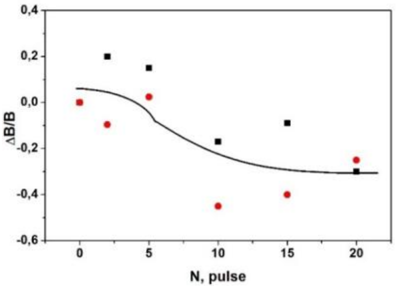

Fig. 4. Relative change in the width of reflections (18, 29) – (squares) and (20, 32) – (circles) as a result of plasma irradiation of sample № 1

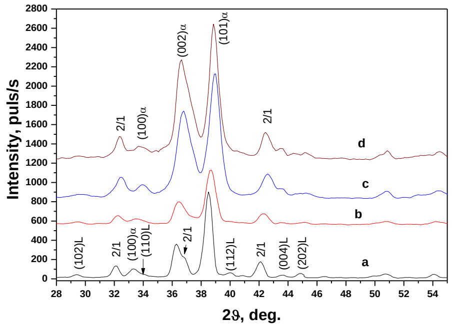

Fig. 5. Sections of diffraction patterns from sample № 2 in the initial state (a) and after irradiation with 0.2 MJ/m^2: 2 pulses (b), 15 pulses (c), and 20 pulses (d)

It is noticeable that the parameters of the crystal lattice of the Laves phase do not practically change with the accumulation of the number of irradiation pulses (Fig. 6,a). The nature of the change in the lattice parameters of the solid solution and the 2/1 crystal-approximant phase turned out to be the same and nonmonotonic (seFig. 6,b and Fig. 6,c respectively). A decrease is observed until the number of impulses is 10...15, and then an increase follows. The relative decrease in $\Delta a/a$ was -0.013 for the 2/1 approximant phase, which coincides with $\Delta c/c$ = -0.014. At the same time, $\Delta a/a$ = -0.023 for α-Ti (Zr), which is significantly higher than the previous values. At the same time, $\Delta a/a$ = -0.023 for α-Ti (Zr), which is significantly higher than the previous values. We believe that the changes in the reflection intensities and structure parameters of the phases indicate the occurrence of phase transformations stimulated by radiation-thermal action. In addition to phase changes typical for sample № 2, irradiation causes a change in the half-width of reflections from the phases present. The relative change in the width $\Delta B/B$ with an increase in the number of irradiation pulses is shown in Fig. 7. It follows from the figure that, in contrast to sample № 1, with increasing the number of irradiation pulses *N*, the width of reflections increases for all phases (see Fig. 7 curve 1–4).

The largest change in $\Delta B/B$ is characteristic of the Laves phase (see Fig. 7 curve 1 and curve 4), the smallest – for the solid solution (see Fig. 7 curve 2). Since the analyzed reflections are located at small diffraction angles, all broadening is due to the effect of phase crystallite fragmentation.

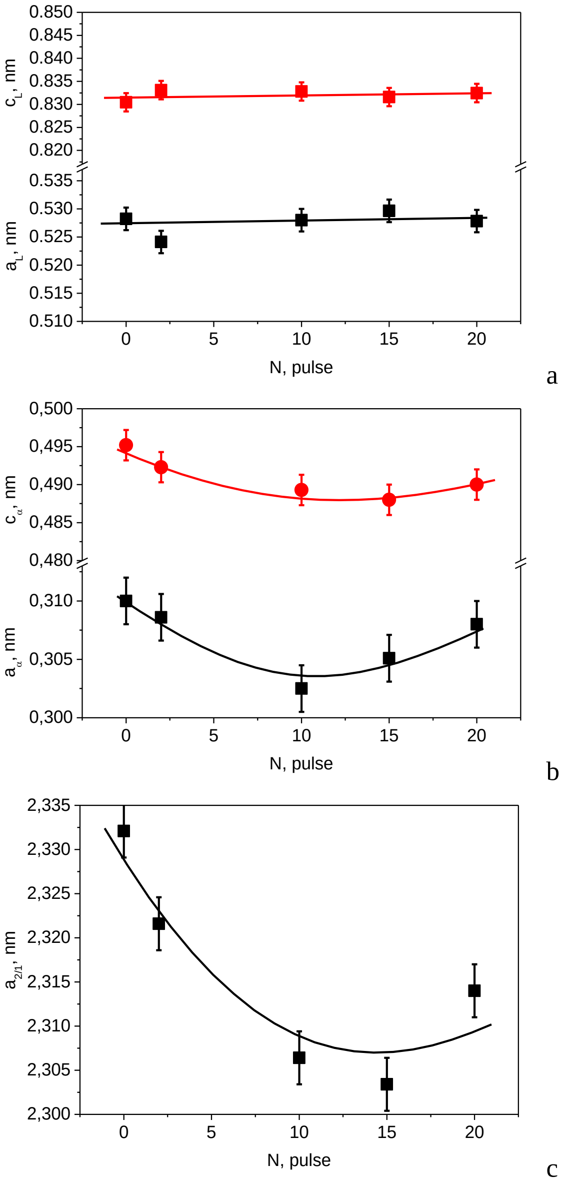

Fig. 6. Changes in the lattice periods of the L-phase (a), phase α-(Ti, Zr) of a solid solution (b), and phase 2/1 – approximant (c) of sample № 2 depending on the number of plasma pulses

Based on the absolute value of the reflection width, we find the fragmentation of crystallites of the Laves phase from the initial 30 nm to 8 nm; the size of the crystallites of the phase 2/1 approximant reduced from 25 to 12 nm, and the crystallites of the α-phase – from 20 nm to 10 nm. Fig. 7 curve 4 also shows the change in the width of the (302) Laves phase reflection located at medium diffraction angles, for which the total broadening includes the contribution of microstrains.

It can be stated that even the first irradiation causes a sharp jump in $\Delta B/B$ and then the increase becomes flattered in comparison with $\Delta B/B$ for the L-phase (202) reflection (see Fig. 7 curve 1). This means that at the initial moment of impact, microstresses accumulate, and, consequently, dislocations randomly located inside the crystallites; and when the crystallites are fragmented, they can go into the sinks. According to [25], such a change in the width, as in Fig. 7, indicates the actual accumulation of defects of the second class, such as dislocations and large dislocation loops, the deformation fields from which propagate over considerable distances. It is possible that the accumulation and accompanying annealing of defects is the reason for the formation of a system of cracks on the surface of the samples (Fig. 8). The formation of cracks upon irradiation of materials with hydrogen plasma in modes simulating transient phenomena in a fusion reactor is a common phenomenon, and was studied earlier on tungsten samples [26].

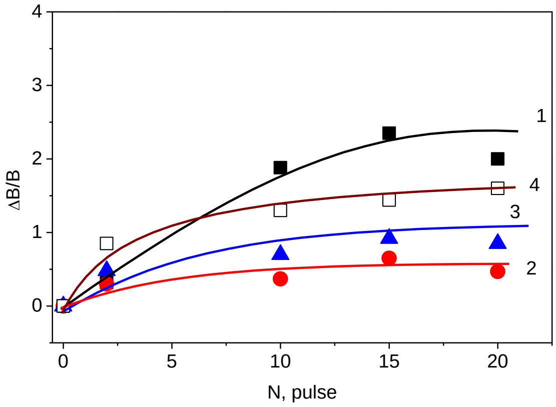

Fig. 7. Relative changes in the width of reflections: (202) of the L-phase (1); (101) of the α-(Ti, Zr) solid solution phase (2) ;(821) of the 2/1-approximant phase (3), and (302) of the L-phase (4) of sample № 2 depending on the number of plasma pulses

Fig. 8 shows that in sample № 2 (Fig. 8,c,d) containing crystalline phases, the number of cracks per unit area is greater than in the sample № 1 (Fig. 8,a,b). The cracks have a sinuous, broken shape, characteristic of crystalline materials. In sample № 1 the cracks have a smooth appearance, typical for glass. We observed similar cracks earlier when coatings were irradiated with a heat load of up to 0.6 MJ/m^2 [27]. The relief observed in Fig. 8,b,d qualitatively resembles the phenomenon of ablation during pulsed thermal action on materials with poor thermal conductivity.

DISCUSSIONS

According to calculations performed in [22, 23], the surface of tungsten samples is heated up to 850 °C at a thermal load of hydrogen plasma of 0.2 MJ/m^2. In the Ti-Zr-Ni system, the quasicrystalline phase is stable up to 660 °C, and then undergoes a reverse eutectoid transformation with the formation of the Laves phase and the solid α-Ti (Zr) solution [16]. However, according to the above results, the quasicrystalline phase in sample № 1, like the phases of sample № 2, turned out to be resistant to the radiation-thermal action of plasma, and the phase composition did not qualitatively change after irradiation even with 20 pulses.

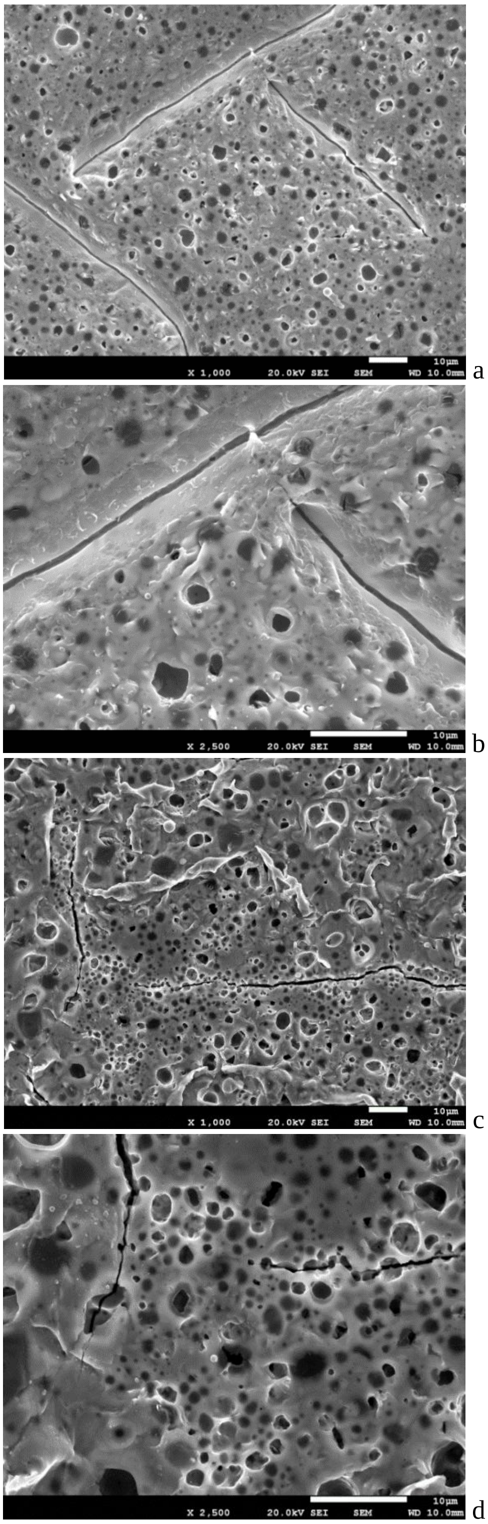

Fig. 8. Change in the surface morphology of the film coating № 1 (a, b) and № 2 (c, d) as a result of irradiation with 20 hydrogen plasma pulses with a load of 0.2 MJ/m^2

This means that the bulk of the sample № 1 material was not heated above 600 °C. The reason may be the low thermal conductivity inherent in quasicrystals [2]. We assume that all the energy of the plasma beam supplied in a short time of the pulse is accumulated in a very thin surface layer and causes its melting.

This can explain the development of the surface relief, which is observed in Fig. 8,b,d. It is known that when a material is heated to the melting temperature, excess vacancies are formed [28], and inhomogeneous heating creates tensile residual stresses in the surface layers [29, 30], which ensure a flow of vacancies into the inner layers of the coating. As a manifestation of this effect, we observe a decrease in the quasicrystallinity parameter as a result of irradiation. As for sample № 2, we also note surface melting. In addition, taking into consideration the changes in the intensities of reflections and the parameters of the crystal lattices of the phases present, we admit the occurrence of partial phase transformations caused by irradiation. In this case, the heating of the sample should have been above the eutectoid equilibrium temperature of ≈ 600 °C. This equilibrium is shown in the polythermal section on the three-component diagram of the Ti-Zr-Ni system given in [16]. According to the Gibbs phase rule, this equilibrium must be established between the four phases. In [16], there are only three of them. We argue that the fourth missing phase is the 2/1 approximant crystal phase. This is an intermediate high-temperature phase, possibly up to the melting of the eutectic. The existence of approximant phases with a number of atoms in the cluster more than in the Bergman cluster, as well as their position on the phase diagram, were primarily considered theoretically in [15]. The straight line of eutectoid equilibrium is actually a section of a certain plane in space. At temperatures above 600 °C, three phases should be in equilibrium, which is what we observe. It is in the heating – cooling mode during irradiation that a partial phase transformation occurs between them. The content of the α-solid solution phase clearly becomes larger. Even according to [16], if at 600 °C the ratio of the masses of α-Ti (Zr) and the Laves phase is 1/3, then already at ≈ 800 °C, it is ≈ 0.9. The content of nickel in the α-solid solution increases. Since the lattice parameters of the Laves phase do not change, we assume that the main redistribution of the components occurs between the α-solid solution and the 2/1 crystal-approximant phases, which is manifested in the behavior of the curves in Fig. 6. We noted earlier that the largest decrease in the $\Delta a/a$ values was observed for the solid solution. Since this is associated with a change in the interatomic distance in the most closely packed plane (000l), we assume that large titanium or zirconium atoms are replaced in it by smaller nickel atoms, which pass from the crystal-approximant phase. The relative content of the solid solution increases.

CONCLUSIONS

It was experimentally established that the quasicrystalline phase, as well as related crystalline phases (Laves phase, α-solid solution, and 2/1 crystal-approximant phase) of the Ti-Zr-Ni system, turned out to be stable under conditions of radiation-thermal exposure to hydrogen plasma with a heat load of 0.2 MJ/m^2 at the QSPA Kh-50 quasi-stationary plasma accelerator.

It is shown that the inherent changes mainly manifest themselves in the form of a change in the substructure, as well as a partial phase transformation with a redistribution of components between the 2/1 crystal-approximant phase and the α-solid solution phase.

It was found that under radiation-thermal loads in a total of 20 pulses, thin films of the Ti-Zr-Ni system containing a quasicrystalline icosahedral phase are less prone to crack formation than coatings with crystalline phases of the same system.

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ПОВЕДІНКА ТОНКИХ ПЛІВОК КВАЗІКРИСТАЛІВ І АПРОКСІМАНТНИХ ФАЗ СИСТЕМИ Ti-Zr-Ni ПРИ РАДІАЦІЙНО-ТЕРМІЧНОЇ ДІЇ В РЕЖИМАХ ПЕРЕХІДНИХ ПРОЦЕСІВ

С.В. Малихін, В.В. Кондратенко, В.О. Махлай, І.Є. Гаркуша, І.А. Копилець, Ю.С. Борисов, С.С. Геращенко, С.В. Суровицький, С.С. Борисова

На квазістаціонарному плазмовому прискорювачі КСПП Х-50 (ННЦ ХФТІ) досліджено характеристики тонких плівок Ti41Zr38.3Ni20.7 при радіаційно-термічному впливі водневої плазми з тепловим навантаженням на поверхню 0,2 МДж/м^2. Фазовий склад, структурний стан та морфологія поверхні були досліджені методами рентгенівської дифракції та скануючої електронної мікроскопії. Встановлено, що квазікрісталічна фаза, а також споріднені з нею кристалічні фази: фаза Лавеса, α-твердий розчин і фаза 2/1 кристала-апроксиманта Ti-Zr-Ni-системи виявилися стійкими при опроміненні до 20 імпульсів водневої плазми. Фазовий склад якісно не змінюється. Показано, що зміни, які відбуваються в покриттях, в основному проявляються як зміни в субструктурі спостережуваних фаз. Структура квазікристалічної ікосаедричної фази з накопиченням імпульсів впливу вдосконалюється, і розмір областей когерентності збільшується. У плівках, що складаються з кристалічних фаз, спостерігається часткове фазове перетворення з перерозподілом компонентів між фазою 2/1 кристала-апроксиманта і фазою α-твердого розчину. Встановлено, що тонкі плівки Ti-Zr-Ni-системи, що містять квазікристалічну ікосаедричну фазу, при радіаційно-термічних навантаженнях у сумі 20 імпульсами менш схильні до утворення тріщин, ніж покриття з кристалічними фазами тієї ж системи.