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MULTILAYER TUNGSTEN/QUASI-CRYSTAL TI-Zr-NI SYSTEMS AS PROMISING MATERIALS OF PROTECTIVE ELEMENTS A FUSION REACTOR

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This paper presents the results of fabricating a model sample of a multilayer coating on a Al₂O₃ substrate, which consisted of 30 periods of alternately deposited 10.5-nm-thick layers of Ti41Zr41Ni18 and 2.5-nm-thick layers of W. The effect of annealing for 1 h at 500, 600, and 700 °C was studied. Characterization of the phase and structural state of the coating by X-ray diffractometry and small-angle X-ray reflectometry was carried out. It was found that during the annealing process, the tungsten layers in the multilayer composition did not undergo significant changes, and all alterations occur only in the Ti41Zr41Ni18 layers. Annealing affected the thickness of the layers, density, and interlayer roughness. It has been experimentally shown that the phase transformation "quasicrystal $\rightarrow 2/1$ crystalline approximant" is accompanied by an 8.3% volume increase compared to the volume of the quasicrystalline phase, but this does not lead to the destruction of the periodic composition. The multilayer structure proved to be resistant to high temperatures and, despite phase changes, did not lose its bond with the substrate. The used combination of materials and the high annealing temperature did not generate significant internal stresses or mechanical damage. The results obtained in this study allow for the further controlled formation of layered quasicrystal/tungsten microsystems of various designs with different layer thicknesses. The next perspective involves conducting practical tests with plasma to study the radiation-thermal impact.

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INTRODUCTION

The evaluation of stable systems and materials operating under high particles and plasma fluxes as well as heat and mechanical loads is major issue for the realization projects of nuclear and thermonuclear reactors. Such problem could be solved on the basis of obtaining new physicochemical regularities of radiationinduced processes in novel materials [1]. The important issue is evaluation of fusion plasma influence on plasma facing components first wall and divertor fusion reactor during transient events such as disruptions, vertical displacement event and edge locales modes [1, 2]. Tungsten was chosen as major reference plasma facing materials due to small sputtering rate, high melting threshold and high heat conductivity provided high level heat exhaust [2]. However, the gradual accumulation of internal macrostresses in tungsten as well as the development of surface cracks contribute to significant macroscopic erosion. The accumulation of light isotopes D+, T+, He+ and heavy impurities also lead to brittleness of tungsten [1–3].

It is proposed to use layered "tungsten/Ti-Zr-Ni quasicrystal" coatings to investigate the possibility of enhancing the radiation resistance of materials for nuclear and thermonuclear energy. In such layered systems, Ti-Zr-Ni quasicrystals (QC) or their structurally closer crystalline approximants are suggested for reducing the impact of hydrogen on tungsten and steel. QS can function as reversible hydrogen absorbers in the form of a solid solution without forming hydrides and then releasing hydrogen. Their lack of translational invariance properties makes QC resistant to high radiation. Such layered systems should be considered as modern nanostructured SMART systems, where each layer performs its specific function.

The development of special structural materials for the first wall and blanket that are resistant to high radiation and thermal fluxes is an important task. As a first step in the study of the new coating, it is proposed to analyze the effect of heating on structural and phase changes in a model multilayer Ti41Zr41Ni18/W coating. For this purpose, a multilayer periodic Ti41Zr41Ni18/W composition was specifically fabricated. The analysis of its X-ray diffraction pattern at small angles, along with traditional X-ray phase analysis, provides additional information about thermal stability of the composition, possible processes at the Ti41Zr41Ni18/W interlayer interfaces, and changes in layers' density. That is the focus of this article.

1. SAMPLES AND INVESTIGATION TECHNIQUE

periodic Multilayer coating (MPC) of Ti41Zr41Ni18/W was manufactured by direct current magnetron sputtering of an alloy target with the composition of Ti41Zr41Ni18 (at.%) and a tungsten target (with a tungsten content of 99.8%) in an argon environment. The working gas pressure was maintained at 0.4 Pa. The chamber was initially evacuated to approximately 10⁻⁴ Pa. Flat sapphire plates with a root mean square roughness < 0.3 nm were used as substrates for the coating. To improve he adhesion of the coating the surface of the substrate was pre-cleaned using an Ar+ beam. The substrate temperature during the coating deposition process did not exceed T = 40 °C.

The layers of the Ti41Zr41Ni18 alloy and tungsten were deposited alternately. For this purpose, the placed substrate was periodically over the corresponding magnetrons using an automatically controlled mechanism. The deposition of MPC was carried out providing high stability of the deposition rate $(\pm 0.1\%)$ and high accuracy of the substrate exposure time over the magnetron $(\pm 5 \text{ ms})$ during the deposition of each layer. This ensured uniform thickness of the layers of each material throughout the entire thickness of the coating and stability of the period (sum of the thickness of one Ti41Zr41Ni18 layer and the thickness of one W layer). The deposition rate for each of the TiZrNi and W layers was approximately 0.5 nm/s. The topmost layer of the coating was a W layer.

The annealing of the samples was performed in a furnace in a vacuum chamber (VUP-5) at a pressure below 10^{-2} Pa. In the furnace, the sample was contained in a tantalum envelope. The temperature was monitored using a chromel-alumel thermocouple connected to the envelope. The sample reached the specified annealing temperature in approximately 30 min. The temperature during annealing was maintained with an accuracy of \pm 5 °C.

A multilayer periodic structure of Ti41Zr41Ni18/W, consisting of alternately deposited layers of Ti41Zr41Ni18 with a thickness of 10.5 nm and W layers with a thickness of 2.5 nm and having 30 periods, was fabricated. The sample was sequentially annealed for 1 h at 500, 600, and 700 °C.

To determine the characteristics of the structure and phase composition of the layers in MPC, X-ray diffraction (XRD) was utilized. Measurements were conducted using the DRON-3M diffractometer with the filtered radiation of the Cu anode. A grazing incidence geometry scheme was employed in the X-ray diffraction measurements (GIXRD), where the sample was set at a constant angle ($\alpha \approx 1.4^{\circ}$) to the primary beam, and scanning was done only by the detector [4]. A graphite secondary monochromator was used to reduce the geometric broadening of reflections.

The identification and indexing of reflections from the quasicrystalline phase were carried out following J.W. Cahn's methodology [5], utilizing two indices (N, M). Additionally, the quasilattice parameter a_q was determined. To construct stripe diagrams of possible crystalline phases, the PowderCell software package was employed, along with data from the International Centre for Diffraction Data (ICDD) Powder Diffraction File (ICPDS).

In the investigation of X-ray interference at small angles (X-ray reflectivity), spectra were recorded using the $\theta/2\theta$ scanning scheme. Monochromatization of the primary X-ray radiation was ensured by a silicon single crystal with (110) orientation. The periodic multilayer structure creates a corresponding diffraction pattern near the primary beam, which is convenient for analysis.

The small-angle X-ray reflectivity curve was modeled and compared with the experimental one using the well-known IMD and XrayCalc software [6, 7]. This softwares are based on the recurrent application of Fresnel formulas. The fitting parameters in the calculation of the model reflectivity curves include the thickness of the layers, their density, and roughness. By varying these parameters, the maximum coincidence of the experimental and calculated curves was achieving. The calculated reflectivity curve is highly sensitive to small changes in these fitting parameters, allowing for precise adjustment. Additionally, the periodicity of the multilayer structure reduces the number of fitting parameters, significantly increasing the reliability of the obtained results. As a result, this modeling allows us to determine the thickness of the layers, the density of the layers, and the rms interlayer roughness for the MPC.

2. RESULTS AND DISCUSSION

The results of the X-ray diffractometric investigation of the Ti41Zr41Ni18/W multilayer coating are shown in Fig. 1. According to the decryption data, all scans contain reflections from the crystal phase of tungsten. They are the most intense. Their intensity remains unchanged with annealing, indicating the constancy of the scattering substance's mass. It should be noted that the half-width of the reflections from the W phase is large, approximately 3 degrees, but it is the same for all registered reflections. Thus, the half-width does not depend on the diffraction angle, indicating the absence of microdeformations in the W layers. The size of the coherent scattering regions (CSR), calculated from the half-width of the reflections using the Seljakov-Sherer formula, ranges from 3.3 nm in the initial state to 3.6 nm after annealing at 700 °C. This value should be close to the thickness of the W layers. Given that the size of CSR was obtained for an inclined cross-section of the layer, simple geometric projection calculations allow us to conclude a good match between the experimental and calculated thickness. An estimation of internal stresses in the tungsten phase was made on based of the GIXRD results. They are insignificant compressive stresses or are absent et al. The lattice period in the unstressed state, both in the initial and post-annealing approximately states. is a = 0.3170...0.3172 nm. This exceeds the reference value $a_{tabl} = 0.3165$ nm possibly due to influence of residual conditions in the vacuum chamber.

According to the results of processing the diffractogram No. 1 in Fig. 1, an icosahedral quasicrystalline phase is recognized in the deposited layers of Ti41Zr41Ni18 in the initial state. They are characterized by the quasilattice parameter $a_q \approx 0.521$ nm. The estimation of the coherence length based on the width of the reflection gives a value of 2.3 nm. It is indicating that the layers of the Ti41Zr41Ni18 quasicrystal are nanostructured in the initial state.

After annealing at 500 °C, the existence of the quasicrystalline phase in the Ti41Zr41Ni18 layers became indisputable. Its reflections became more clearly recognized, and their quantity increased. The quasilattice parameter remained unchanged. The coherence length increased to 5.5 nm.

Annealing at 600 °C slightly reduced the quasilattice parameter to 0.518 nm, and the coherence length remained unchanged. Additionally, the crystalline phase of the 2/1 approximant (2/1AC) appeared. It is characterized by a lattice period $a_{2/1} \approx 2.278$ nm and a coherence length of approximately 4 nm.



Fig. 1. X-ray diffractograms from the MPC Ti41Zr41Ni18/W in the initial state (1) and after annealing in vacuum at temperatures of 500 (2), 600 (3), and 700 °C (4) in Cu-K_a radiation

The relationship between the lattice period and the quasilattice parameter almost precisely matches the known crystallographic relationship given in [8]. Calculations show that the 2/1AC with a period of 2.300 nm should correspond to the QC with $a_q = 0.518$ nm.

After annealing at 700 °C, the QC phase completely disappeared, and only the 2/1AC phase was observed. Its period was 2.320 nm, i.e. it increased compared to the period in the previous state. We can conclude that a phase transformation of QC to 2/1AC occurred. Its numerical characteristics are practically same as in single-layer coatings of Ti41Zr41Ni18 [9] during thermal annealing.

The results of X-ray reflectometry of the MPC in the initial state and after annealing are presented in Fig. 2. Symmetry of the peaks (see curve No.1 in Fig. 2) and the angular position of the last interference maximum $(29 \approx 9)$ degrees indicate on the high perfection of the manufactured MPC in the initial state [10]. Annealing at 500 °C did not significantly affect the perfection structure. Noticeable changes of the periodicity were observed at 700 °C.

Perfect periodicity of the MPC allowed to perform the procedure of mathematical modeling of the smallangle reflectograms. Roughness and density could be used as fitting parameters for the layer's thicknesses. Examples of the modeling for the initial state and after annealing at 500 °C are shown in Figs. 3 and 4.

A good agreement between the measured and calculated curves was achieved through fitting as it shown in mentioned figures. The calculated structure parameters of the MPC received from the modeling are given in Table for the initial state and after annealing.

Annealing led to changes in the multilayer structure of the coating as it shown in Fig. 2. An annealing causes a sequential shift of the peaks in the reflectogram towards larger angles with increasing temperature up to $600 \,^{\circ}$ C. The appearance of additional maxima is observed on the side of smaller angles on curve No.3 in Fig. 2. It is clearly recognized for maximus after the fifth at same times the first ones became asymmetrical

on the side of smaller angles. This indicates the appearance of a new periodicity after annealing at 600 °C. After annealing at 700 °C, the entire MPC transitions to this periodicity. The intensity and number of peaks decrease as well as their width increases.

As noted earlier, quantitative characteristics of these changes can be traced by the data presented in Table. As can be seen, annealing at 500 °C led to a reduction in the MPC period due to a decrease in the thickness of Ti41Zr41Ni18 layers. While the thickness and density of tungsten layers remain unchanged.

After annealing at 600 °C, the period reduction continued. It due to further thinning of the Ti41Zr41Ni18 layers without changes in the tungsten layers. Taking into account the results of phase investigations, we think that the reduction of the Ti41Zr41Ni18 layer thickness at temperatures of 500 and 600 °C is attributed to the improvement of the substructure of the icosahedral phase $Ti_{41}Zr_{41}Ni_{18}$ and changes of its quasilattice parameter.

The appearance of additional periodicity in the multilayer periodic structure after 600 °C is well described by a model with changes in the upper 4 periods, as shown in Table. The mentioned top 4 periods have an increased period in compared to the main multilayer package. Those are also characterized by a significant increase of layer roughness, that appeared in the decreasing of the intensity of peaks.

After annealing at 700 °C, a high roughness of layers is causes by a decrease of the intensity and the number of peaks in the small-angle reflectogram (see Fig. 2), a decreasing of the reflectivity down to 20% in the region of total external reflection (angles less than 1°) as well as an increase of the peaks' width. Such a reflectogram is difficult to model and does not allow us to carefully determine the density and roughness of the layers. However, based on the angular position and the height of the peaks, it was found that the period of the MPC was 13.53 nm as well as the average thickness of the Ti41Zr41Ni18 layers was 10.96 nm. In this case, the density and RMS roughness are estimated values.



Fig. 2. Small-angle X-ray reflectivity from the MPC in the initial state (1) and after annealing in a vacuum at temperatures of 500 (2), 600 (3), and 700 °C (4), measured using Cu- $K_{\alpha l}$ radiation



Fig. 3. Measured and calculated small-angle X-ray diffraction from the MPC in the initial state



Fig. 4. Measured and calculated small-angle X-ray diffraction from the MPC after annealing at 500 $^{\circ}$ C

The appearance of additional periodicity at 600 °C is associated with the formation of the 2/1AC phase in the four upper periods of the MPC. At a temperature of 700 °C, the 2/1AC approximant phase becomes the only one throughout the volume. We assume that during the previous annealing at 600°C, there was a non-uniform temperature distribution in depth. This non-uniformity can be explained by two factors. The first factor is the short annealing time in this experiment -1 h. Studies with single-layer coatings in previous works [11–13] showed that annealing durations of 25...60 h are needed for complete phase transformations at temperatures of 470...600 °C. The second factor is the very low thermal conductivity of QC ($\approx 5 \text{ W/(m \cdot K)}$) [14]. Since a heating have been occurs from the surface than a temperature gradient across the depth of the coating is established due to the low thermal conductivity. Such gradient disappears only at long annealing times. Therefore, in this experiment, at 600 °C, the internal layers of the MPC were insufficiently heated. The phase transformations in the upper four layers proceeded significantly further compared to the rest of the composition. Confirmation of this assumption is the fact that after annealing at 700°C, all layers of the 30-period composition acquired thickness and roughness similar to the four upper periods after annealing at 600 °C (see Table).

As noted above, a quasicrystalline phase is present in the initial state of the nanostructured MPC. While only an amorphous phase was observed previously in single-layer micron coatings [11–13]. In this work, a single-layer sample with a thickness equal to 30 layers of Ti41Zr41Ni18 was prepared. It also turned out to be amorphous. The QC phase usually appeared after annealing at 450 and 500 °C. We assume that specific of the magnetron sputtering technology and the accompanying processes cause QC formation.

A large number (up to 60% of the bombarding ion quantity) of highly energetic neutrals of argon are reflected from the target surface with an average energy of 160 eV during sputtering of materials surfaces with an atomic weight greater than that of the argon [15, 16].

Parameters of the model structure of the MPC Ti41Zr41Ni18/W in the initial state
and after annealing, for which the best match of the calculated and measured smallangle diffraction curves
of this coating was obtained

Number of	Material of the layer	Layer thickness,	Density, g/cm ³	Roughness,	
layers/periods		nm		nm	
Initial state					
1 layer	W	2.56	16.9	0.47	
30 periods	Ti41Zr41Ni18	10.44	6.1	0.39	
	W	2.57	17.8	0.29	
Annealing at 500 °C					
1 layer	W	2.57	16.9	0.44	
30 periods	Ti41Zr41Ni18	10.26	6.3	0.37	
	W	2.57	17.8	0.32	
Annealing at 600 °C					
1 layer	W	2.43	16.9	0.68	
4 periods	Ti41Zr41Ni18	10.71	6.0	0.58	
	W	2.45	17.8	0.58	
	Ti41Zr41Ni18	10.12	6.4	0.51	
26 periods	W	2.57	17.8	0.43	
Annealing at 700 °C					
1 layer	W	2.57	16.9±0,5	1.0±0,2	
30 periods	Ti41Zr41Ni18	10.96	6.0±0,5	1.0±0,2	
	W	2.57	17.8±0,5	1.0±0,2	

A large number (up to 60% of the bombarding ion quantity) of highly energetic neutrals of argon are reflected from the target surface with an average energy of 160 eV during sputtering of materials surfaces with an atomic weight greater than that of the argon [15, 16]. Therefore, when the coating is deposited on the tungsten target, the applied layer of Ti41Zr41Ni18 and the first few atomic layers of tungsten will be subjected to intense argon bombardment. Stimulated transformation from an amorphous phase to a quasicrystalline one may occur in the thin layers of Ti41Zr41Ni18 under such radiative-thermal conditions. The energy of sputtered atoms is in the range of 10...20 eV at irradiation the coating on the Ti41Zr41Ni18 target. While the energy of reflected argon atoms is an order of magnitude lower Therefore, single-layer coatings [17]. remain amorphous.

CONCLUSIONS

1. The results of the study on the thermal stability of a specially prepared model sample of MPC, consisting of alternately deposited layers of Ti41Zr41Ni18 with a thickness of 10.5 nm and W layers with a thickness of 2.5 nm, and having 30 periods, showed that:

- changes occurred only in the Ti41Zr41Ni18 layers during annealing in the temperature range up to 700 °C;

- the phase transformation "quasicrystal \rightarrow crystalline approximant 2/1" is accompanied by an increase in volume by 8.3% compared to the volume of the quasicrystalline phase;

- tungsten layers in the multilayer composition Ti41Zr41Ni18/W do not change significantly during annealing in the temperature range of 500...700 °C; - the combination of materials and temperature did not lead to the generation of significant internal stresses and did not result in mechanical damage;

- the multilayer construction remained stable at temperature up to 700 °C as well as adhesion of the coating to the substrate did not lose despite phase changes.

2. The results obtained in the work allow to the controlled formation of multilayer nanocomposites and layered microsystems of quasicrystal/tungsten with various layer combinations and thicknesses.

3. The further heat fluxes tests of Ti41Zr41Ni18/W coatings of various constructions need to perform by powerful plasma irradiation to evaluation the radiation-thermal effects.

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БАГАТОШАРОВІ ВОЛЬФРАМОВІ/КВАЗІКРИСТАЛІЧНІ СИСТЕМИ ТІ-Zr-NI ЯК ПЕРСПЕКТИВНІ МАТЕРІАЛИ ЗАХИСНИХ ЕЛЕМЕНТІВ ТЕРМОЯДЕРНОГО РЕАКТОРА

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Викладаються результати виготовлення модельного зразку Al₂O₃ з 30 періодами покриття, яке складалося з почергово нанесених шарів Ti41Zr41Ni18 завтовшки 10,5 нм та шарів W завтовшки 2,5 нм, а також дослідження впливу температурного відпалювання протягом 1 год при 500, 600, 700 °С. Характеристики фазового та структурного станів досліджені методами рентгенівської дифрактометрії та малокутової рентгенівської рефлектометрії. Встановлено, що при підпалюваннях шари вольфраму у багатошаровій композиції суттєвих змін не зазнають, а всі зміни відбувалися лише у шарах Ті41Zr41Ni18. Відпалювання впливають на товщину шарів, густину та на міжшарову шорсткість. Експериментально показано, що фазове перетворення «квазікристал → кристалічний апроксимант 2/1» супроводжується збільшенням об'єму на 8,3% в порівнянні з об'ємом квазікристалічної фази, але це не призводить до руйнування періодичної композиції. Багатошарова конструкція виявилася стійкою до високих температур та, незважаючи на фазові зміни, не втратила зв'язку з підкладкою. Використане поєднання матеріалів та температура відпалювання не призвели до генерації істотних внутрішніх напружень та до створення механічних пошкоджень. Отримані в роботі результати дозволяють надалі кероване формування багатошарових нанокомпозицій та шаруватих мікросистем квазікристал/вольфрам різноманітної конструкції поєднання шарів та товщин. Далі перспективним стає проведення натурних випробувань плазмою вивчення радіаційно-термічного впливу.