THE INFLUENCE OF IRRADIATION BY HYDROGEN PLASMA ON THE STRUCTURE AND PHASE COMPOZITION Ti-Zr-Ni ALLOYS CONTAINING QUASICRYSTALLINE PHASE

S.V. Bazdyreva[1], I.E. Garkusha[2], V.A. Makhlaj[2], S.V. Malykhin[1], A.T. Pugachov[1]

[1]Kharkov Polytechnic Institute, NTU, Kharkov, Ukraine
E-mail: malykhin@kpi.kharkov.ua;
[2]Institute of Plasma Physics NSC "Kharkov Institute of Physics and Technology", Kharkov, Ukraine
E-mail: makhlay@kipt.kharkov.ua

The effect of irradiation by ITER ELM-like hydrogen plasma heat load of 0.6 MJ/m^2 to change of morphology, structure and phase composition of Ti41,5Zr41,5Ni17 alloy is investigated. The initial state of such alloy is characterized by presence of crystal- approximant 1/1 phase (W-phase) with lattice constant a_W = 1.428 ±0.0003 nm and icosahedral quasi-crystalline phase (*i*-phase) with quasi-crystalline parameter a_q = 0.5175 ±0.0003 nm. Plasma irradiation causes creation of re-solidified layer with depth up to 50 µm and phase transformation of crystal- approximant 1/1 to quasi-crystalline phase.

PACS: 61.44.+p; 52.40 Hf

INTRODUCTION

Choice of plasma facing materials and estimation of it response to high heat and particles fluxes remain one of important issues for realization of fusion reactor project. One of interesting class of materials is quasicrystals on the base of Ti-Zr-Ni. They have unique properties concerning reversible multicyclic absorption and recycling of hydrogen.

In generally quasicrystals - naturally ordered aperiodic metal structure with symmetry different from the crystal symmetry [1, 2]. Quasi-crystalline structure can be obtained only from ultra pure metals in two and three-, four-component systems from such processes as: rapid solidification, condensation of steam, alloying of mechanically compacted powders, crystallization of amorphous ribbons and classical process of solidification [1] High-speed quenching can be used as one of the method of modifying the surface of solids by high-density plasma flow.

The results of investigation Ti-Zr-Ni alloy with quasicrystal phase exposed by ITER ELM like heat load are presented in this paper.

1. SAMPLES AND EXPERIMENTAL EQUIPMENT

Original alloy composition Ti41,5Zr41,5Ni17 prepared in an arc furnace with non-consumable tungsten electrode in an argon atmosphere under pressure 10^{-5} Pa. Iodide Ti, Zr and 99.9% Ni - electron beam melting (99.95%) were taken as the initial components. Plate samples of 1 mm thickness and 1 cm^2 area were prepared for the plasma load tests.

The samples have been exposed to hydrogen plasma streams produced by the quasi-steady-state plasma accelerator QSPA Kh-50 [3]. The main parameters of QSPA plasma streams were as follows: ion impact energy about 0.4 keV, the maximum plasma pressure 3.2 bars, and the stream diameter about 18 cm. The surface energy loads measured with a calorimeter were achieved 0.6 MJ/m^2 (near the tungsten melting threshold). The plasma pulse shape was approximately triangular, and the pulse duration was 0.25 ms. A surface analysis was carried out with an MMR-4 optical microscope equipped with a CCD camera and Scanning Electron Microscopy (SEM) of the JEOL JSM-6390 type. To study a micro-structural evolution of the exposed targets, the X-ray diffraction technique (XRD) has been used. The absolute error for lattice parameters measurements is ±3×10^{-4}nm in the experiment. Computer processing of the experimental diffraction patterns were performed using the software package New profile3.5. Quasi-crystalline phase identification was carried out in conformity with the Cahn´s methodology using indices N and M [4]. To characterize the structure of the icosahedral quasi-crystalline phase and its degree of perfection we used quasi-crystalline parameter a_q, whose value is determined by (1)

$$a_q = \frac{\lambda}{4\sin\vartheta}\sqrt{\frac{N+M\tau}{1+\tau^2}} = \frac{d}{2}\sqrt{\frac{N+M\tau}{1+\tau^2}} \quad (1)$$

where *N, M* - index reflections; *d* - the distance between planes; τ = 1.618034 ... - "golden ratio."

The phase of cubic crystal- approximant 1/1 (W - phase) was disclosed by modeling theoretical diffraction pattern using the basis data of the lattice shown in [5]. The lattice constant of crystal- approximant 1/1 was calculated from the position of diffraction peaks.

2. EXPERIMENTAL RESULTS

The initial state of alloy Ti41,5Zr41,5Ni17 is characterized by the presence of two phases (Fig. 1). The main phase is lowly textured crystal- approximant phase (W-phase) with a lattice constant a_W = 1.428 nm. The second phase is an icosahedral quasi-crystalline *i*-phase with quasi-crystalline parameter a_q = 0.5175 nm. I -phase is strongly textured; only planes with 5-fold symmetry locate parallel to the surface.

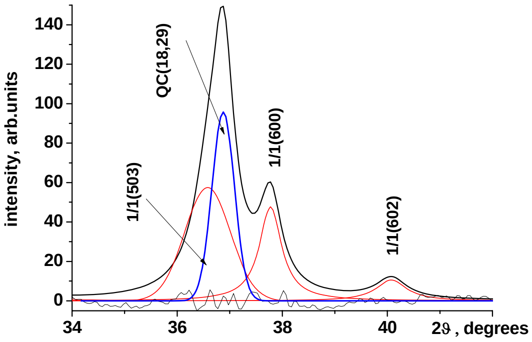

Fig. 1. The result of separation of the complex diffraction pattern fragment for the sample in initial state

The strict analytical relation between the parameters of structure (2) confirms the presence of these two phases [6, 7]

$$a_{p/q} = \frac{2(p+q\tau)}{\sqrt{2+\tau}} a_q \quad (2)$$

where p and q are order of crystal- approximant (for our case $p=q=1$). The deviation from this expression does not exceed 2 %.

After five exposures with plasma heat load of 0.6 MJ/m^2 W-phase disappears, the content of icosahedral phase grows (Fig. 2). It is shown that the texture disappears. Increasing intensity and the number of *i*-phase reflections evidence about this. The quasicrystalline parameter increases to $a_q = 0.5181$ nm. This fact evidences about the uniform distribution of components by volume. Further of number of plasma pulses up to10 did not change phase composition. The quasicrystalline parameter slowly decreased till 0.5170 nm, hypothetically, due to reducing the content of the most low-melting element nickel in the alloy.

Fig. 3 demonstrates the surface modification after multiple plasma loads of 0.6 MJ/m^2 that causes surface melting. Optical microscopy observations show that network of cracks and ripple are formed on the exposed surface (see Fig. 3,a). Such ripple is a set of dodecahedron and triacontahedron asperities of re-solidified material. Cracks with smooth edges are observed also on the exposed surface. Such cracks are typical for fracture of the amorphous material.

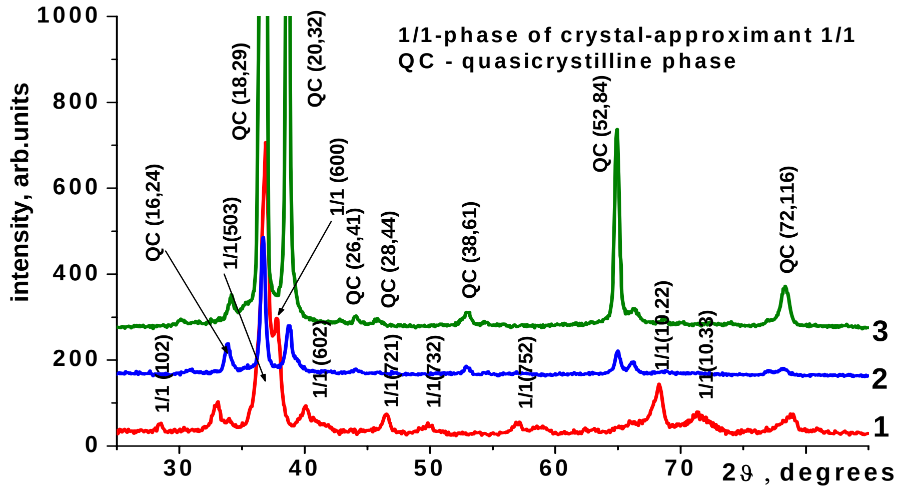

Fig. 2. Diffraction patterns in the initial state (1), (2 exposed areas after 5 (2) and 10 (3) pulses of 0.6 MJ/m^2) (Cu-K_α radiation)

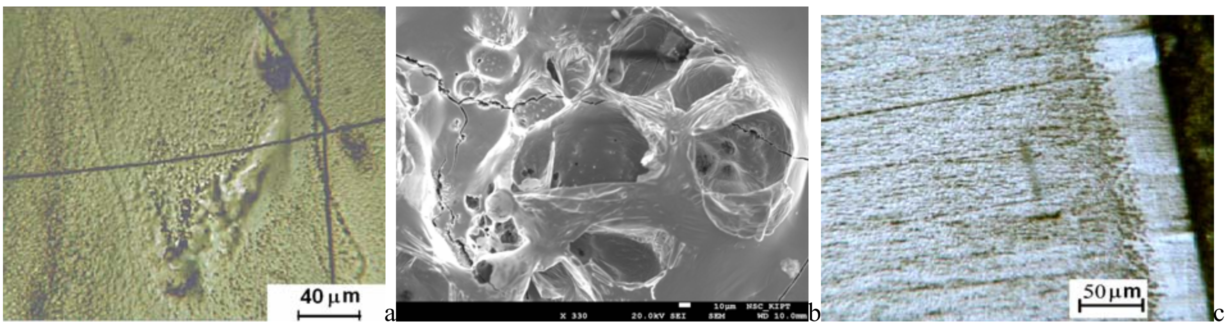

Fig. 3. Views of exposed sample surface after 5 pulses of 0.6 MJ/m^2: optical image (a) , SEM image (b), and cross section (c)

Thickness of modified layer achieved 50 μm (see Fig. 3,c). According to the calculation, the X–ray half-value absorption layer is comparable with light areas on Fig.3,c. Comparing the data of X-ray diffraction and optical microscopy images, we can conclude that the stimulated $W \rightarrow i$- phase transition occurred at the depth ≈ 50 μm. In the "plate" model, the expression describing the time distribution of the plasma power is given by [8]

$W_m(t) = \chi \cdot c_v \cdot \rho \cdot \nabla T$ where χ is thermal diffusivity, c_v is specific heat at constant volume, ρ is density, ∇T - the temperature gradient. Estimations show that the thermal conductivity coefficient of Ti41,5Zr41,5Ni17 alloy is ≈ 48 $W \cdot m^{-1} \cdot K^{-1}$ under 2.4 $GW \cdot m^{-2}$ power and under conditions of changing the temperature from boiling $T \approx 3500$ K to the value of the quasicrystal boundary equilibrium $T \approx 900$ K. This value is in good agreement with the results [9] after extrapolation to high temperatures.

CONCLUSIONS

Behavior of alloy Ti41,5Zr41,5Ni17 was studied under repetitive ITER ELM-like plasma exposures in the QSPA Kh-50 facility. The initial state of Ti41,5Zr41,5Ni17 is characterized by the presence of crystal-approximant phase 1/1 (W-phase) with lattice constant a_W = 1.428 nm and the *i*-phase with quasicrystalline parameter a_q = 0.5175 nm.

The irradiation of alloy surface by pulses of hydrogen plasma with heat load of 0.6 MJ/m^2 and the pulse duration of 0.25 ms leads to create of modified resolidified layer. The phase transformation *W*-phase to *i*-phase with a_q = 0.5181 nm observed in this layer. Such phase transformation is stimulated by fast re-melting and quenching of sample material. *I* - phase remains stable at increase of irradiation dose in two times. At that, average thickness of modified layer achieves 50 μm. The network of cracks typical for fracture of the amorphous material and ripple appear in exposed surface. The estimation of coefficient of thermal conductivity ≈ 48 $W \cdot m^{-1} \cdot K$ carried out on the base of thermophysical properties of Ti41,5Zr41,5Ni17 agrees with received earlier.

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Article received 18.09.12

ВЛИЯНИЕ ОБЛУЧЕНИЯ ВОДОРОДНОЙ ПЛАЗМОЙ НА СТРУКТУРУ И ФАЗОВЫЙ СОСТАВ TI-ZR-NI СПЛАВОВ, СОДЕРЖАЩИХ КВАЗИКРИСТАЛЛИЧЕСКУЮ ФАЗУ

С.В. Баздырева, И.Е. Гаркуша, В.А. Махлай, С.В. Малыхин, А.Т. Пугачов

Исследовано влияние облучения водородными плазменными потоками с тепловыми нагрузками 0,6 $МДж/м^2$, подобными ИТЭР ГЛМ, на изменение морфологии, структуры и фазового состава сплава Ti41,5Zr41,5Ni17. Исходное состояние такого сплава характеризуется присутствием фазы кристалла–аппроксиманта 1/1 (W-фазы) с периодом решетки a_W = 1.428 ±0.0003 нм и икосаэдрической квазикристаллической фазы (*i*-фаза) с параметром квазикристалличности a_q = 0,5175±0,0003 нм. Плазменное облучение вызывает создание повторно затвердевшего слоя с глубиной до 50 мкм и превращение фазы кристалла-аппроксиманта 1/1 в квазикристалическую фазу.

ВПЛИВ ОПРОМІНЕННЯ ВОДНЕВОЮ ПЛАЗМОЮ НА СТРУКТУРУ ТА ФАЗОВИЙ СКЛАД TI-ZR-NI СПЛАВІВ, ЯКІ МІСТЯТЬ КВАЗІКРИСТАЛІЧНУ ФАЗУ

С.В. Баздирєва, І.Є. Гаркуша, В.А. Махлай, С.В. Малихін, А.Т. Пугачов

Досліджено вплив опромінення водневими плазмовими потоками з тепловими навантаженнями 0.6 MJ/m^2 , які подібні до ІТЕР ГЛМ, на зміну морфології, структури та фазового складу сплаву Ti41,5Zr41,5Ni17. Вихідний стан такого сплаву характеризується присутністю фази кристала-апроксиманта 1/1 (W-фази) з періодом решітки a_W = 1.428 ± 0.0003 нм та ікосаедричної квазікристалічної фази (*i*-фаза) з параметром квазікристалічності a_q = 0,5175 ± 0,0003 нм. Плазмове опромінення викликає створення повторно затверділого шару з глибиною до 50 мкм і перетворення фази кристала-апроксиманта 1/1 у квазікристалічну фазу.