SECTION 4

DIAGNOSTICS AND METHODS OF RESEARCHES

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THE DEFORMATION VALUE EFFECT ON THE Ta-W-Ta SOLID-PHASE JUNCTION NATURE WITH INTERMEDIATE Ti (TITANIUM) INTERLAYERS FOR THE NEUTRON PRODUCING TARGET OF THE NEUTRON SOURCE

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The paper presents the quality studies results of the Ta-W-Ta solid phase junctions with Ti interlayers obtained by hot roll bonding in vacuum, that determine the work suitability in the form of neutron producing targets at the research nuclear facility 'Source of Neutrons'. It is shown the layers mutual penetration level of the Ta-W-Ta solid-phase junction with Ti interlayers which provides a high level of clad layers adhesion when using the hot vacuum roll bonding method. The deformation parameters necessary both for the solid phase bond occurrence and for the reliable solid-phase junction formation of tantalum and tungsten refractory metals layers through a titanium interlayer, with a joint strength exceeding the value of a less durable material are determined. The possibility of chemical vapor deposition (CVD) method using for side faces coating of targets was studied.

INTRODUCTION

Tungsten is one of the neutron producing target variants for the subcritical assembly of the nuclear research facility (NRF) 'Neutron Source' of NSC KIPT. It is promising due to the neutrons high yield when irradiated with high-energy electrons. The target represents a set of square-section plates, each of which has 4 spacer protrusions that form cooling cavities when these plates are installed close to each other. During operation, the target plates are cooled with water which will certainly lead to the tungsten corrosion especially under irradiation conditions.

For the chemical corrosion protection and the irradiated material radioactive products ingress into the cooling water prevention, the tungsten target plates must be covered with a protective layer of tantalum [1–5]. The problem in such a target creation is about the appropriate technology development. Among the existing metals joining methods, the most suitable for this task are the following: the method of hot deformation and the method of chemical vapor deposition (CVD). The literature data review shows that the W and Ta materials joining to each other requires the temperature not lower than 1500 °C and high pressure reaching the values of 1450 MPa [1–5].

The vacuum hot roll bonding (VHRB) is one of the most relevant and modern methods of refractory metals joining due to their mutual high-temperature vacuum friction [6–16]. Using a combined way for the solid-phase junctions creation based on such a pair of refractory metals as tantalum-tungsten which includes the CVD and VHRB methods it was possible to obtain high-quality high-strength solid-phase tantalum-tungsten-tantalum junctions with titanium interlayers, at much lower technological parameters, suitable for neutron producing targets at the NRF 'Neutron Source' at NSC KIPT.

1. TESTING PROCEDURE

Ta-W-Ta solid-phase junction specimens were obtained by hot roll bonding in vacuum at the temperature of 1300 °C. In order to do this tungsten and tantalum plates were cut out by the electric spark method on a ZAPbp BP-96ds machine to the dimensions of 65×65×3 mm, after which they were washed with acetone and subsequently chemically etched in the nitric and hydrofluoric acids solution.

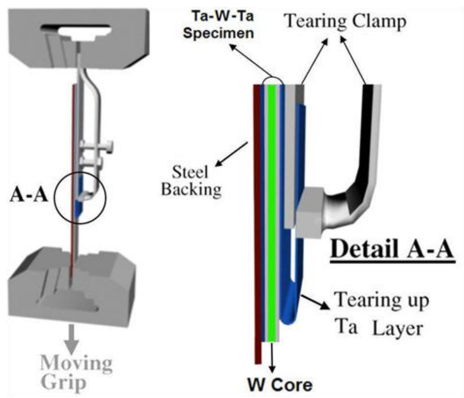

Fig. 1. The peeling test scheme

After that, the plates were assembled into a layered batch and the entire specimen was placed in a vacuum chamber for a subsequent heating to predetermined temperature. After reaching it, the specimen was put under the rolls by manipulator where the roll bonding process took place. The chamber pressure did not exceed $1{\cdot}10^{-6}$ mm Hg. The relative reduction was calculated as follows:

$$\varepsilon_{rel} = \frac{\Delta H}{H_{init}}, \quad (1)$$

$$\Delta H = H_{init} - H_{fin}, \quad (2)$$

where H_{init} and H_{fin} are the initial and the finite heights of undergoing the roll bonding laminate respectively.

The specimens mechanical tests were carried out on a ‘Bi-00-201 Nano’ servo-hydraulic testing machine. The layers adhesion quality was studied using the tantalum clad layer peeling tests according to used in work [17] scheme and presented in Fig. 1. The peeling strength was calculated as a load divided by the bond width – N/mm. The target core strength was studied by three-point bending tests according to the Fig. 2 scheme.

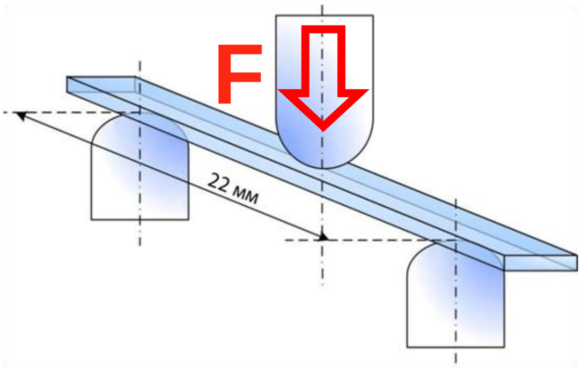

Fig. 2. Three-point bending test scheme

2. RESULTS AND DISCUSSION

2.1. TITANIUM INTERLAYER APPLYING

The titanium interlayer applying for the Ta-W-Ta solid-phase junction creation provides the high-strength and high-tightness characteristics of the resulting joints due to these materials affinity with respect to the created composition that prevents the stress concentration formation at the layer boundaries during the solid-phase joints manufacturing process. This is confirmed by metallographic studies shown in Fig. 3.

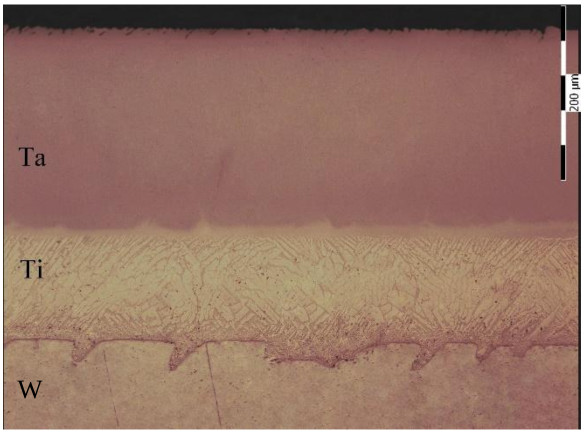

Fig. 3. The microstructure of Ta-W-Ta solid-phase junction with Ti interlayers specimen, ×200

The optical microscopy study of the W-Ti and Ti-Ta joint zones microstructure (see Fig. 3) revealed the absence of defects in the form of pores, delaminations, cracks or inclusions as well as in the body of the specimen components. An X-ray electron probe microanalysis (Fig. 4) made it possible to determine the widths of the diffusion zones for Ta-Ti and Ti-W equal to 2 and 3 µm, respectively. The nature of the concentration curves slope at the Ta-Ti and Ti-W interfaces indicates the titanium predominant penetration into both tantalum and tungsten, which is explained by the high diffusion mobility of titanium.

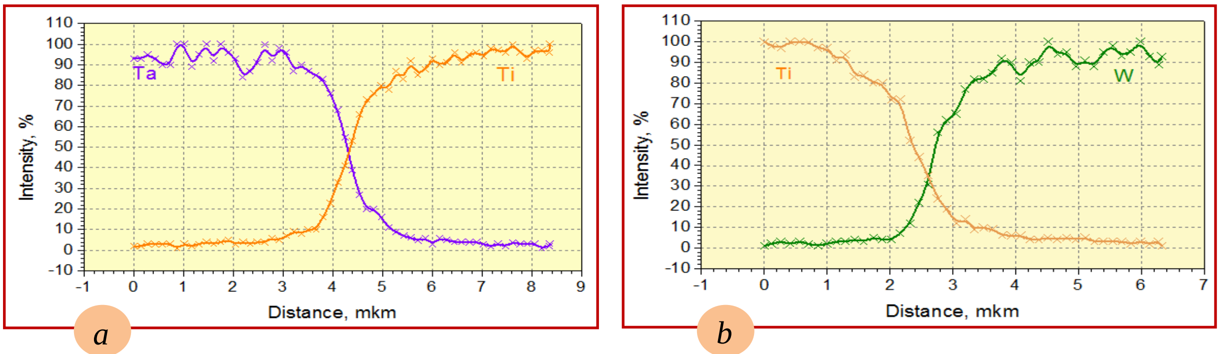

Fig. 4. Electron probe X-Ray analysis of Ta-W-Ta (Ti interlayers) specimens through the interfaces: a) tantalum-titanium and b) titanium-tungsten

The Ta-W-Ta multilayer joints (with Ti interlayers) specimens underwent the three-point bending tests. The results (Fig. 5) clearly show the presence of a plastic deformation region before the tungsten destruction at the room temperature already, which appears due to the deformation of the clad layers of tantalum and titanium. This indicates that the part of the deformation is compensated by the tantalum and titanium clad layers that under the three-point bending conditions work to strain (Fig. 6). The maximum strength during such a test was 65 MPa.

The deposited tantalum coating on the side faces by the CVD method during the deformation near the fracture zone, behaves as follows. Fig. 7 shows the micrographs of the deposited tantalum layer on destroyed specimens. Metallographic studies performed by optical microscopy show that the destruction occurs not along the Ta-W interface, but along tungsten or tantalum themselves. In the body of tantalum and tungsten components there are defects in the form of cracks that develop both in the longitudinal and transverse directions relatively to the interface. Some of them spread beyond the layer boundary and propagate into the plate of the opposite material. Nevertheless, the absence of the tantalum-tungsten interface itself destruction indicates that its strength remains higher than that of the each component.

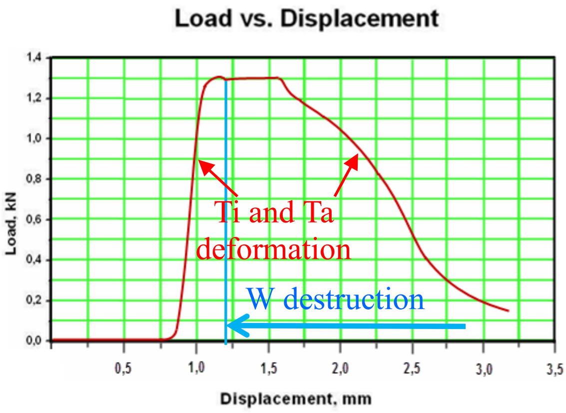

Fig. 5. The result of for three-point bending tests at T_{exp}=20 °C, P=65 MPa

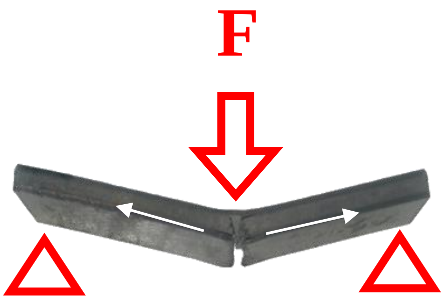

Fig. 6. The appearance of Ta-W-Ta multilayered specimen after three-point bending tests

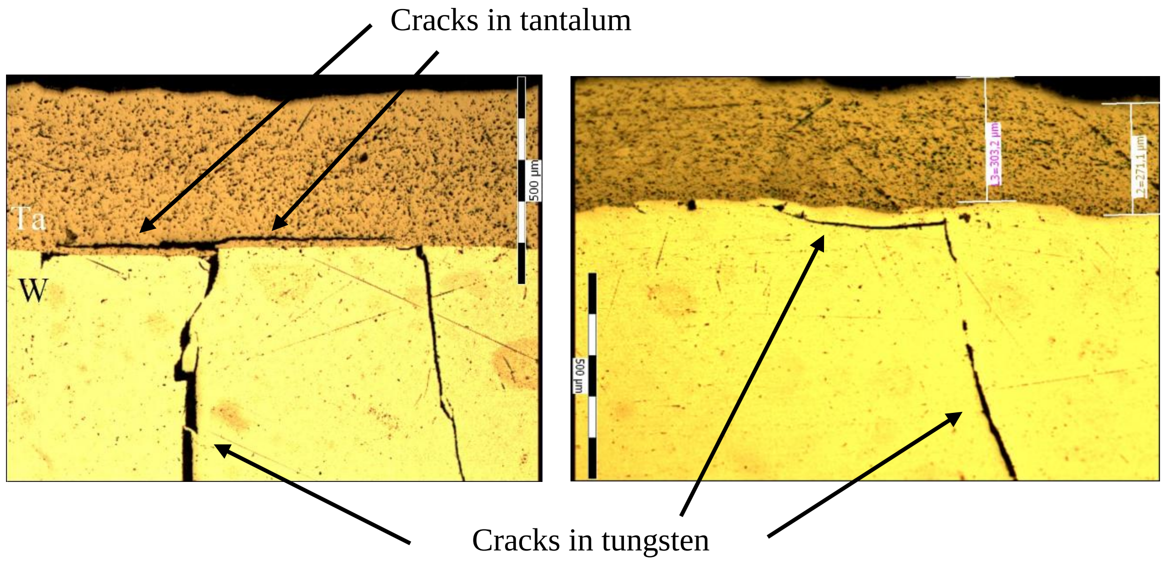

Fig. 7. Metallographic images of tantalum coated tungsten specimens after three-point bending tests near (≈ 2 mm) the fracture zone

2.2. THE REDUCTION IMPACT DURING ROLL BONDING

The layers bonding nature of the multilayer composition was determined by metallographic studies (Fig. 8). The layers bonding strength was determined by the clad layer peel tests tensions. Fig. 9 shows the dependence of the clad layers bonding strength on the batch relative deformation value ε_{rel} (roll bonding at the laminate optimum heating temperature of 1300 °C).

As it follows from the obtained results the threshold deformation of such laminates corresponds to 1.5…1.6%. Such a deformation value during roll bonding can provide the layers adhesion already. The connection area reaches up to 60…80%, that comes from the metallographic analysis results (see Fig. 8,a). Strength bursts during the peeling tests (see Fig. 9,a) also indicated the zonal nature of the layers solid-phase junction. The layers average bonding strength was 10 N/mm, which corresponds to the peak strength values of that when these materials were being roll bonded directly without interlayers. The minimum strength of 7 N/mm corresponds approximately to the average strength of tungsten and tantalum roll bonded without interlayers.

Prior to this deformation value the specimens were destroyed when removing from the unloading chamber of the VHRB mill, or when they were being fixed in a tensile testing machine, ($\varepsilon_{rel} \approx 0.5\%$), or the macroscopic defects and some partial joint occurred with peeling of tantalum and titanium along the solid-phase junction field, detectable during the visual inspection ($\varepsilon_{rel} \approx 1.0\%$).

a – T=1300 °C, ε=1.6%
(ΔH=0.05 mm, H_{end}=3.0 mm)

b – T=1300 °C, ε=3.2%
(ΔH=0.1 mm, H_{end}=3.0 mm)

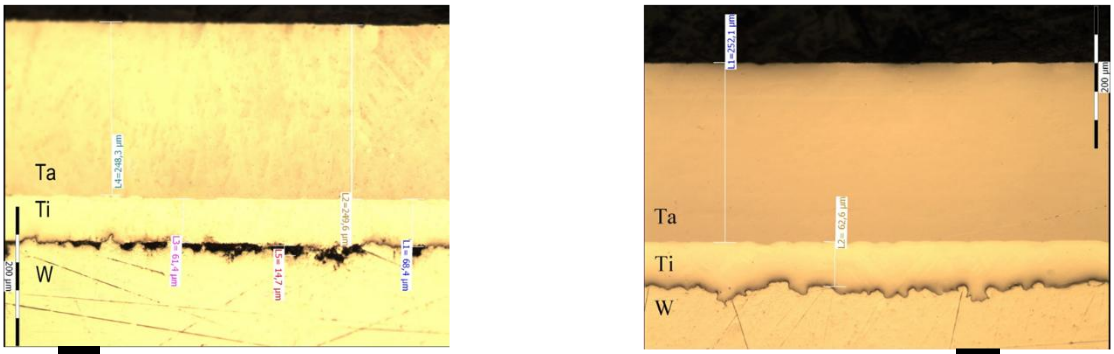

Fig. 8. Microstructure of Ta-W-Ta with Ti interlayer obtained with different reduction values

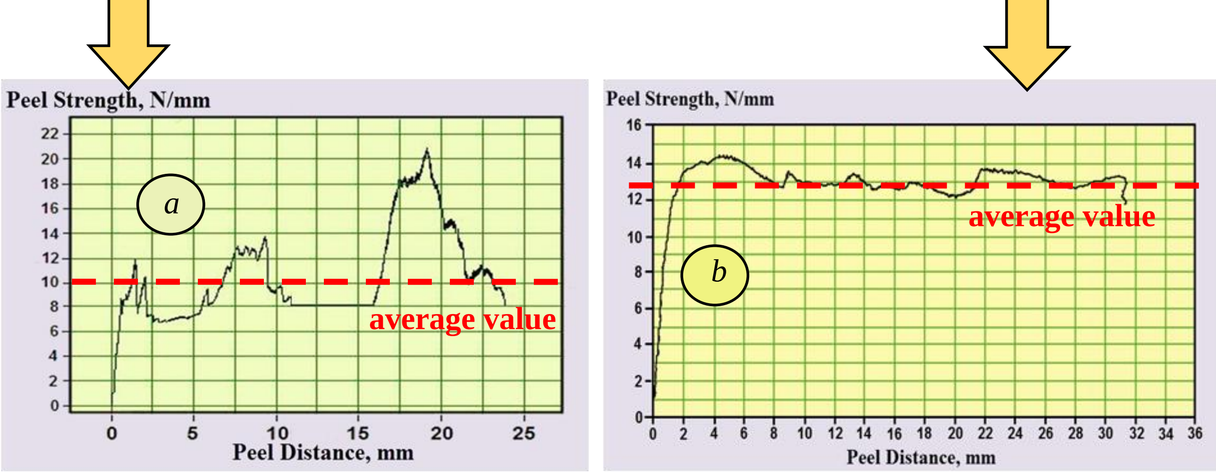

Fig. 9. Peeling tests results of Ta-W-Ta with Ti interlayers

The reduction increasing up to 3.2% ensures the complete adhesion of layers. Metallography analysis of W-Ti and Ti-Ta joints zones revealed the absence of defects in the form of pores, gaps, delaminations, cracks, or inclusions (see Fig. 8,b). The clad layers adhesion strength was 13N/mm according to the peel testing (see Fig. 9,b). The strength graph has the equable extension with no obvious bursts. This can be explained by the uniform fit of the layers along the entire interface which corresponds to 100% of the layers joining area.

A further reduction rising causes the strength of layers joint increasing up to 15 N/mm due to the strain hardening of the solid phase junction make up materials.

Exceeding the reduction value above $\varepsilon_{rel} = 6\ldots7\%$ led to the entire solid-phase joint curvature. In this case the laminate specimens required the additional processing.

Metallographic studies of the peel tested specimens showed the cladding layer detachment nature. Fig. 10 shows that the specimens destruction occurred not along the solid-phase interface, but along the more brittle metal that is tungsten in this case. Moreover, the destruction of the specimens occurred at the distance of at least 30…50 μm from the joint boundary. This fact was also confirmed by the X-ray fluorescence analysis of such specimens performed from the side of the solid-phase junction area: Table presents the results which confirm the presence of a high tungsten component concentration. In other words, the solid-phase junction specimens obtained by the VHRB method possess the higher joint interface strength than tat of the less durable constituent material.

Strengthening of thin interlayers is explained first of all by the specific nature of the volumetric-stress state occurring in the interlayer under loading. The elementary volume inside the interlayer is in a triaxial tension state, that is, in a stressed state which is harder than in uniaxial tension one. This is a consequence of the rigid contact surfaces reaction between the core plate and the cladding material [18]. As a result, the transverse plastic deformations are formed and the effect of interlayer hardening occurs.

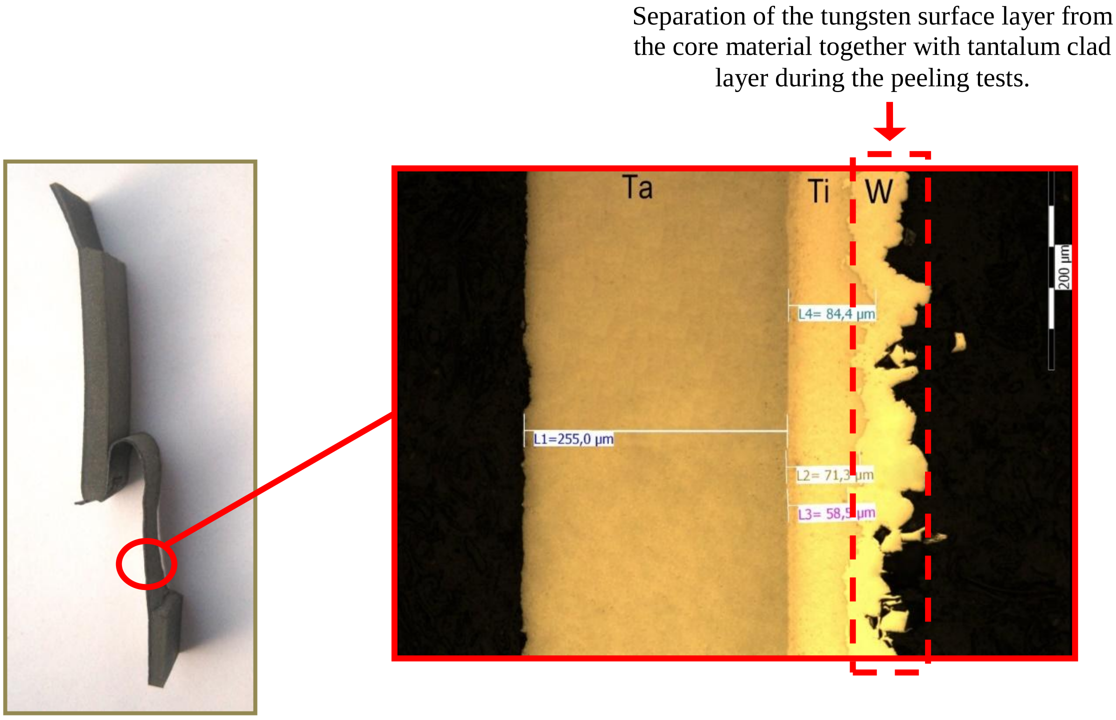

Fig. 10. Appearance and microstructure of the clad layer after the peeling test

X-ray fluorescence analysis of the clad layer after the peel test

At. number	Elem.	Concentration, %
22	Ti	0.2515 ± 0.0162
23	V	0.0282 ± 0.0046
26	Fe	0.0321 ± 0.0026
28	Ni	0.0167 ± 0.0015
73	Ta	0.6933 ± 0.0152
74	**W**	**97.9190 ± 0.0258**
75	Re	1.0593 ± 0.0127

CONCLUSIONS

1. By applying the method of vacuum hot roll bonding, it was possible to obtain high-quality Ta-W-Ta solid phase junctions using Ti interlayers at such temperature and force parameters that are significantly lower than those when using other joining methods.

2. An appropriate choice of the materials vacuum hot roll bonding temperature and force parameters, allows to achieve such a high layers joint strength, which exceeds the strength of a less durable material, which in the case of the tantalum-tungsten-tantalum laminate is tungsten.

3. The threshold deformation that is necessary for 100% formation of tantalum-tungsten-tantalum refractory materials solid-phase junctions, when using a titanium layer, was established, which is 3.2% of the relative reduction value.

4. Side-deposited tantalum by CVD has high adhesion to the core material, that is tungsten, and can be used for the target side surfaces coating. At the same time the interfaces strength also remains higher than that of a less durable material, which is tungsten.

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ВПЛИВ ВЕЛИЧИНИ ДЕФОРМАЦІЇ НА ХАРАКТЕР ТВЕРДОФАЗНОГО З’ЄДНАННЯ Ta-W-Ta З ПРОМІЖНИМИ Ti (ТИТАНОВИМИ) ПРОШАРКАМИ ДЛЯ НЕЙТРОННО-УТВОРЮЮЧОЇ МІШЕНІ НЕЙТРОННОГО ДЖЕРЕЛА

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Представлено результати досліджень якості твердофазних з’єднань Ta-W-Ta з прошарками Ti, отриманих методом гарячої прокатки у вакуумі, що визначають придатність до роботи у вигляді нейтронно-утворюючих мішеней на дослідній ядерній установці «Джерело Нейтронів». Показано ступінь взаємного проникнення шарів твердофазного з'єднання Ta-W-Ta з прошарками Ti, що забезпечує високий рівень адгезії плакованих шарів при використанні методу гарячої вакуумної прокатки. Визначено параметри деформації, необхідні як для виникнення твердофазного зв'язку, так і для утворення надійного твердофазного з'єднання шарів тугоплавких металів танталу та вольфраму через титановий прошарок, з міцністю з'єднання, що перевищує значення менш міцного матеріалу. Вивчено можливість використання способу хімічного газофазного осадження (ХГО) для покриття бічних граней мішеней.

ВЛИЯНИЕ ВЕЛИЧИНЫ ДЕФОРМАЦИИ НА ХАРАКТЕР ТВЕРДОФАЗНОГО СОЕДИНЕНИЯ Ta-W-Ta С ПРОМЕЖУТОЧНЫМИ Ti (ТИТАНОВЫМИ) ПРОСЛОЙКАМИ ДЛЯ НЕЙТРОННО-ОБРАЗУЮЩЕЙ МИШЕНИ НЕЙТРОННОГО ИСТОЧНИКА

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Представлены результаты исследований качества твердофазных соединений Ta-W-Ta с прослойками Ti, полученных методом горячей прокатки в вакууме, определяющие пригодность к работе в виде нейтронно-образующих мишеней на исследовательской ядерной установки «Источник Нейтронов». Показана степень взаимного проникновения слоёв твердофазного соединения Ta-W-Ta с прослойками Ti, обеспечивающая высокий уровень адгезии плакированных слоёв при использовании метода горячей вакуумной прокатки. Определены параметры деформации необходимые как для возникновения твердофазной связи, так и для образования надёжного твердофазного соединения слоёв тугоплавких металлов тантала и вольфрама через титановую прослойку, с прочностью соединения, превышающей значение менее прочного материала. Изучена возможность использования способа химического газофазного осаждения (ХГО) для покрытия боковых граней мишеней.