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EFFECT OF THERMOCHEMICAL TREATMENT ON THE Zr-1%Nb ALLOY CHARACTERISTICS

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Data on improving the characteristics of fuel rods made of the Zr-1%Nb alloy by modifying the near-surface layer are presented. For modification, thermochemical treatment (TCT) was used in a controlled gas environment based on the diffuse oxygen saturation of the near-surface layer. It was established that TCT does not reduce the mechanical characteristics of the Zr-1%Nb alloy compared to ones in the initial state, but in some cases increases its. The influence of different regimes of TCT on the features of oxidation in air and hydrogen saturation of the Zr-1%Nb alloy was considered. It was shown that the optimal regime of TCT provides a reduction in weight gain during oxidation at a temperature of 550 °C in the air for 100 h, and prevents saturation with hydrogen of Zr-1%Nb alloy. It was concluded that the TCT is a promising method for improving the performance of Zr-1%Nb alloy fuel rods.

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INTRODUCTION

Zirconium alloys have a unique combination of nuclear physics and mechanical characteristics at room and high temperatures; therefore they are widely used as structural materials for manufacturing fuel rods, fasteners, spacers, sealing, and other parts of the active zone of nuclear reactors [1–3].

The interaction of zirconium alloy products with the working environment significantly worsens their mechanical characteristics, which reduces the service life [4–8]. These processes include hydrogenation and corrosion. In order to reduce their negative impact, intensive research is currently being conducted on the development of methods of forming protective coatings and surface modification of zirconium products [9–19]. A promising method of targeted modification of the surface of metals and alloys is thermochemical treatment (TCT), in particular, the method of diffusion saturation with oxygen and nitrogen, developed at Karpenko Physico-Mechanical Institute of the NAS of Ukraine (Lviv). This method proved to be effective in the enhancement characteristics of titanium and its alloys. [20–26]. Zirconium alloys are very similar in structure and characteristics to titanium alloys, and the first experiments carried out on zirconium alloy rods showed the formation of surface protective layers that reduced the hydrogen saturation of alloys.

The aim of work is to study the influence of TCT in an oxygen-containing gas environment on the oxidation rate, hydrogen saturation, and mechanical-acoustic characteristics during the tensile testing of fuel rods ring samples made of Ukrainian Zr-1%Nb alloy. The choice of the acoustic emission (AE) technique is due to the fact that it is a powerful structurally sensitive method, which is widely used recently to study the processes of plastic deformation and destruction of materials [27–30].

MATERIALS, SAMPLES AND RESEARCH METHODS

Ring samples for thermochemical treatment (TCT), mechanical tests, and measurement of acoustic parameters were machine-cut from rods with a diameter of 9.13 mm and a wall thickness of 0.67 mm; the width of the ring samples was 3 mm [31–33]. The regimes of TCT in an oxygen-containing environment are shown in Table 1 (*P* is the total pressure of an oxygen-containing gas environment).

Tensile testing of ring samples was performed on a 1958 U10-1 universal testing machine with a strain rate of 0.17 mm/min ($2\cdot10^{-4}$ s^{-1}) at room temperature. The AE parameters were recorded using the M400 acoustic complex [31–33]. The transducer of the AE recorder made of TTS-19 ceramics has a resonant frequency of 180 kHz. The transducer was fixed to the ring sample through a special waveguide, which was one of the joining supports of the ring sample in the tensile machine.

Table 1

TCT regime of ring samples

Symbol	Regime
R1	Initial state
R2	T = 580 °C, τ = 3 h, P = $1.33\cdot10^{-2}$ Pa
R3	T = 580 °C, τ = 3 h, P = $1.33\cdot10^{-1}$ Pa
R4	T = 580 °C, τ = 3 h, P = 1.33 Pa
R5	T = 580 °C, τ = 0.5 h, P = 1.33 Pa + +T = 580 °C, τ = 2.5 h, P = $1.33\cdot10^{-2}$ Pa

Oxidation of the ring samples was studied by discrete gravimetric after holding for 5...100 h at a temperature of 550 °C in the air. The ring samples were saturated with hydrogen in a gas environment ("dry" hydrogenation) at a temperature of 425 °C and a hydrogen pressure of 0.8 atm. The hydrogen concentration in hydrated ring samples was determined by gravimetric and metallographic methods [26, 27]. Desorption of hydrogen from the ring samples depending on temperature was studied using a MX-7203 mass spectrometer.

RESULTS AND DISCUSSION

The influence of TCT on the ring samples mechanical characteristics. Mechanical characteristics of ring samples after TCT by regime R1-R2 are shown in Table 2.

Table 2

Mechanical characteristics of Zr1%-Nb ring samples after various TCT regimes

Regime	Tensile strenght, MPa	Yield strength, MPa	Elongation, mm
R1	450.2	372.6	2.00
R2	449.0	371.4	1.80
R3	444.5	374.4	1.85
R4	432.8	358.0	2.27
R5	451.0	373.0	2.37

According to the obtained results (see Table 2), the TCT has some influence on the mechanical characteristics, especially on elongation. Of particular interest is the regime R5, which slightly influences on strength but significantly increases elongation to failure that is ductility.

The analysis of the stress-strain curves of the ring samples showed that their character is similar both in the initial state and after TCT. However, the acoustic parameters of the initial and surface treated ring samples have significant differences in the activity, the sum of the registered signals, and the amplitude distribution of the signals in the AE radiation spectrum. This is due to additional structural changes led by the TCT of ring samples.

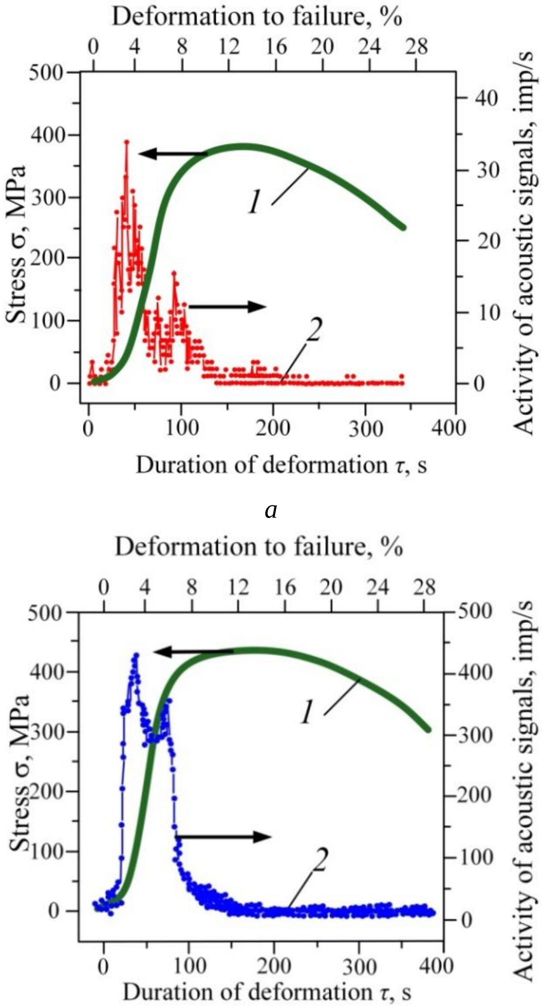
Fig. 1 shows the stress-strain (1) and AE activity (2) curves of the ring samples in the initial state (see Fig. 1, *a*) and after TCT by regime R5 (see Fig. 1,b), depending on the duration of deformation. Three common features in the activity of AE signals for both ring samples are noticeable:

1) acoustic signals begin to register almost immediately after applying the load;

2) the AE activity curves have two well-defined maxima, one in the region of stresses ~ 0.25 of the yield strength, the other in the region of the yield strength;

3) increasing the AE activity at the initial region of deformation, the AE activity decreases and next remains near zero until the destruction of the ring samples.

It was found that the AE activity for the surface treated ring samples is an order of magnitude higher compared to the untreated ones. This allows us to assume that the presence of a hardened layer leads to an increase in the AE activity in the elastic and plastic deformation zones. Perhaps, such a significant increase in AE activity is due to several factors simultaneously. Firstly, the interstitial elements (oxygen) present in the near-surface layer fix some of the dislocations, and upon reaching a certain stress level, the dislocations break away from the fixations, which leads to an increase in the number of mobile dislocations and, accordingly, the number of registered AE signals. Secondly, this may be related to twinning, because in the initial stages (up to 10%) of tensile deformation in zirconium, twins appear and the changes in the location of the elementary prisms of the hcp lattice directly depend on the stress state.



a

b

Fig. 1. Dependence of tensile strength (1) and AE activity (2) on the duration of deformation of ring samples in initial state (a) and after TCT by regime R5 (b)

The influence of TCT on the ring samples oxidation resistance. Fig. 2 shows the dependence of the increase in weight of ring samples that were treated by various regimes on the duration of oxidation at a temperature of 550 °C. According to the results, it can be seen that at the initial stage of oxidation, was fixed high and almost the same rate of weight increase of the ring samples. Then the rate slightly decreases (by 1.5–2 times), and after 60…70 h of oxidation, the starting of an increase in the rate of weight increase is observed. These three stages are particularly visible in the untreated ring sample. It should be noted that after holding for 100 h, the ring samples that were treated by TCT had a smaller increase in weight compared to untreated samples. Thus, for the samples treated by regimes R4 and R5, the weight increase is less by ~ 20%. That is, TCT significantly changes the character of the dependence of weight gain on time and increases the oxidation resistance.

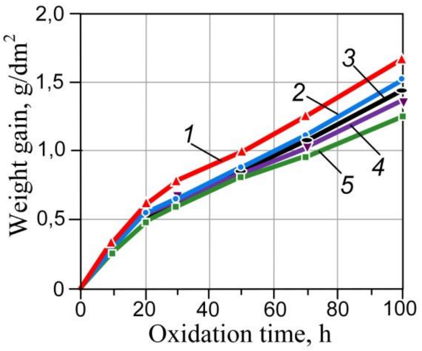
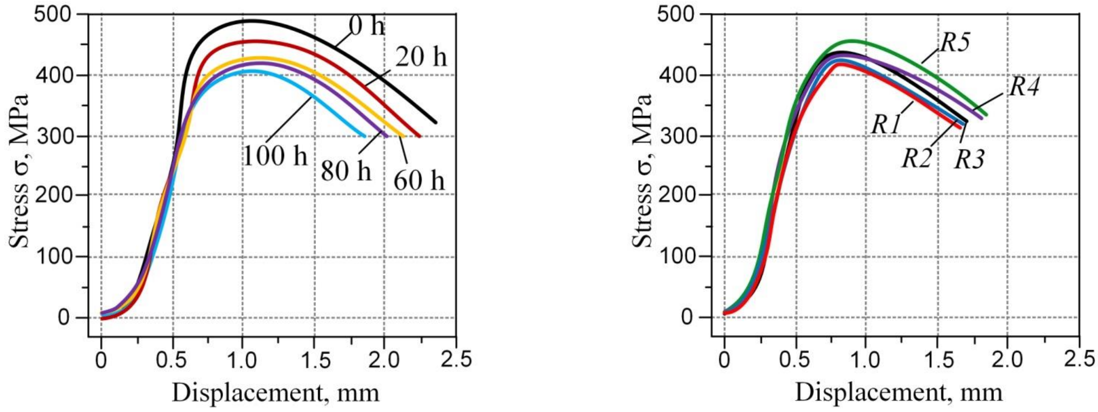


Fig. 2. Dependence of weight gain of ring sample on oxidation time at a temperature of 550 °C (TCT regimes are presented in Table 1)

Fig. 3 shows characteristic changes in the deformation curves of untreated and surface treated ring samples after pre-oxidation at a temperature of 550 °C and holding for 100 h.

Fig. 3 shows the deformation curves of Zr1%-Nb ring samples in the initial state and after various TCT regimes (with a modified surface layer). It was established that with oxidation time increasing the strength and ductility characteristics decrease. Qualitatively similar results of changes in the deformation curves and mechanical characteristics during oxidation were obtained for the surface treated ring samples by regimes R2-R5. The deformation curves of the untreated and surface treated ring samples after 100 h of oxidation are shown in Fig. 3,b. The results of changes in the deformation characteristics of the ring samples after oxidation in the air are presented in Table 3.



a *b*

Fig. 3. Deformation curves of untreated and surface treated ring samples before (a) and after (b) oxidation in air at a temperature of 550 °C

It is obvious that surface treated ring samples by regimes R2-R5 lead to a smaller decrease in the mechanical characteristics after oxidation compared to the untreated ones (regime R1). The reduction percentage of tensile strength after oxidation of surface treated ring samples is 1.5–2 times less than for untreated ones. Although the reduction percentage in yield strength remains almost unchanged, the reduction percentage in elongation is almost two times less for surface treated than for untreated ones. Therefore, taking into account the high absolute initial values of the tensile strength and the ductility of ring samples after TCT, the regime R5 appears to be the most promising of those investigated regimes for improving the complex characteristics of fuel rods made of Zr-1%Nb alloy.

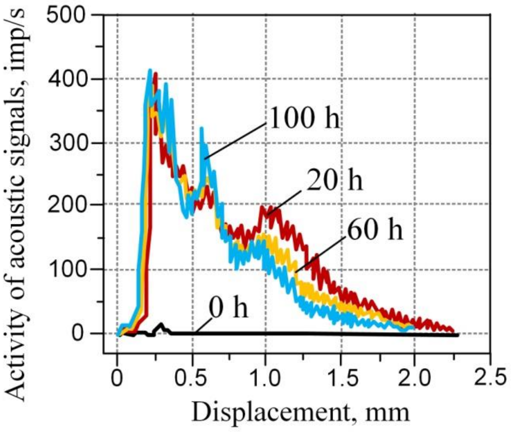
Table 3

The influence of the TCT regimes on the mechanical characteristics of ring samples, oxidized at a temperature of 550 °C holding for 100 h in the air

Regime	State	$\sigma_в$, MPa	Change* $\sigma_в$, %	$\sigma_{0,2}$, MPa	Change* $\sigma_{0,2}$, %	δ, mm	Change* δ, %
R1	Initial state	450.2	–	375.1	–	1.95	–
	after oxidation	406.1	-9.8	344.0	-9.3	1.58	-18.8
R2	after TCT	449.0	–	371.4	–	1.78	–
	after TCT and oxidation	428.0	-4.7	341.9	-7.2	1.60	-10.1
R3	after TCT	444.5	–	371.4	–	1.81	–
	after TCT and oxidation	416.9	-6.2	340.0	-8.0	1.67	-7.7
R4	after TCT	438.2	–	368.0	–	2.2	–
	after TCT and oxidation	411.9	-5.9	343.1	-6.8	2.0	-10.5
R5	after TCT	451.0	–	373.0	–	2.25	–
	after TCT and oxidation	425.6	-6.0	342.5	-8.2	2.03	-9.8

*Decrease in mechanical characteristics after oxidation compared to unoxidized state

In general, when choosing the TCT regime of zirconium alloys in our opinion it is necessary to use such regimes that will provide higher ductility compared to alloy in initial state and the tensile strength matched with it. The dependence of the activity and sum of AE pulses on displacement was revealed for ring samples in an initial state, which were oxidized in air at 550 °C at different times (Fig. 4).



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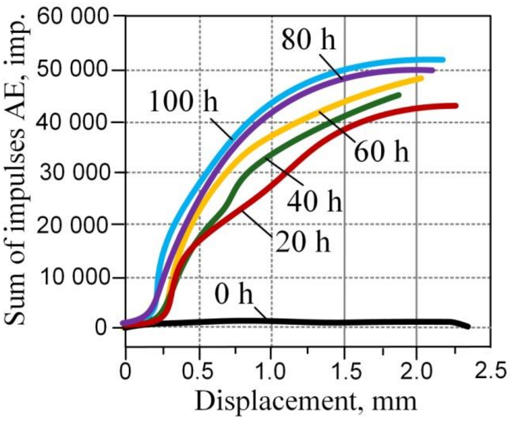
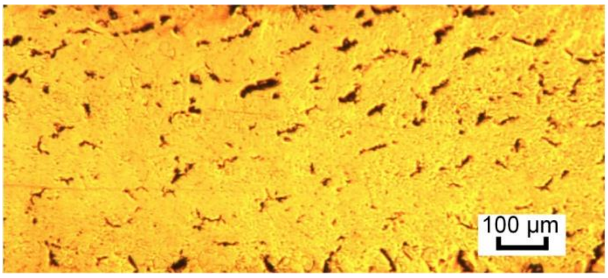


b

Fig. 4. Curves of activity (a) and the sum of AE pulses during deformation to failure (b) of ring samples in an initial state after different oxidation holding in the air at a temperature of 550 °C

In the oxidized state, after a sharp increase in AE activity at the initial part of the deformation, the activity begins to gradually decrease (Fig. 4 shows in contrast to the sharp decrease in AE activity of the samples after TCT in Fig. 1), and this process continues almost until the samples are breaked. At the same time, significant changes in AE activity in the ring samples occur after 20 h of oxidation (see Fig. 4,a). Subsequent oxidation leads to the fact that the curves of AE activity do not change significantly and the dependences have a similar appearance as the total number of pulses (see Fig. 4,b). A similar type of AE activity curves at the initial stage of deformation, in our opinion, is associated with the formation of an oxide film on the surface of the ring samples. When the samples are deformed, the processes of gradual destruction of the oxide film and its delamination occur, which is accompanied by acoustic pulses. As it was shown earlier, in the ring samples in the initial state and after TCT during deformation, the recorded AE activity was not very significant [13]. Thus, the main contribution to the total sum of AE pulses during the deformation of oxidized ring samples is provided by the destruction of the surface film. As increasing of oxidation holding, the curve of the sum of AE signals is located higher (see Fig. 4, *b*). The studied features of the behavior of the activity and the sum of AE pulses of oxidized ring samples at a temperature of 550 °C for 100 h, which surface treated by various regimes, showed that the character of the dependences of these parameters is similar to those presented in Fig. 4. At the same time, the sum of AE pulses for the oxidized ring sample is 1.5–2 times more than that of the oxidized samples that were pre-surface treated. This also indicates more thickness of the surface oxide film of ring samples in the initial state. Consequently, the total sum of AE pulses, which is registered, as well as the increase in the sample weight, can be used to evaluate the oxidation resistance of the Zr-1%Nb zirconium alloy.

The influence of TCT on the ring samples hydrogenation. Hydrogen saturation of the ring samples in the initial state and after TCT was carried out together under the same time-temperature conditions, which ensured the hydrogen concentration in the initial samples at the level of ~ 400 ppm. Then the hydrogen content in the samples was determined and tensile tests were performed with simultaneous recording of mechanical and acoustic parameters. Fig. 5 shows the microstructures of untreated and surface treated by regime R4 ring samples after saturation with hydrogen.



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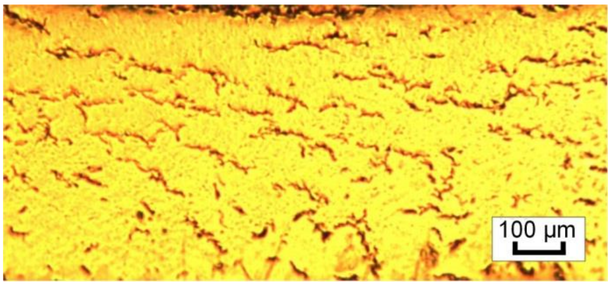

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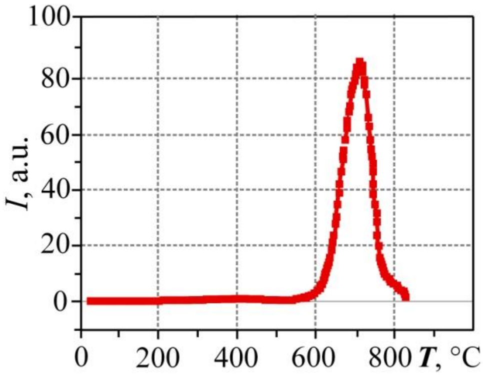
Fig. 5. Microstructures of untreated (a) and surface treated by regime R4 (b) ring samples after saturation with hydrogen

It was determined that the TCT of the samples reduces the absorption of hydrogen by the ring samples and the concentration of the formed hydrides. The most effective was the regime R4, which reduced hydrogen absorption by 3.3 times (to a concentration of ~ 120 ppm) compared to untreated samples.

The analysis of the influence of TCT regimes on the ductility, tensile and yield strength of hydrogenated ring samples showed that the mechanical characteristics of all samples are similar to each other, i.e. hydrogenation of ring Zr-1%Nb alloy ring samples up to ~ 400 ppm does not lead to a noticeable decrease in their mechanical and acoustic characteristics. Therefore, in order to establish the positive influence of TCT on the characteristics of hydrogenated ring samples, it is necessary to carry out hydrogen saturation of the samples in the initial state (R1) to a concentration of 800…1200 ppm.

It was found that after TCT by regime R5, in addition to a decrease in intensity and the presence of a high-temperature peak (700...800 °C) (Fig. 6,a), another desorption peak at temperatures of 390...420 °C (see Fig. 6,b) was recorded on the hydrogen desorption curves. Such a peak may correspond to the hydrogen desorption from the surface modified layer, in which the energy of hydrogen retention is lower compared to the energy of its retention in the zirconium alloy matrix.



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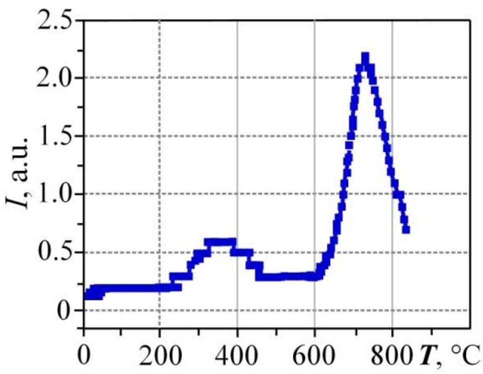

b

Fig. 6. Temperature dependences of hydrogen desorption during heating of pre-hydrogenated ring samples: a – in the initial state; b – after TCT by regime R5.

CONCLUSIONS

1. TCT in an oxygen-containing gas environment by investigated regimes does not reduce the mechanical and acoustic characteristics of the ring samples of fuel rods. Moreover, the ductility of the ring samples increases after TCT by regime R5.

2. The near-surface modified layers, which are formed by TCT change the character of the dependence of weight gain during oxidation and increases the oxidation resistance in comparison with untreated samples.

3. TCT of Zr-1%Nb ring samples significantly reduces hydrogen absorption. The two-stage regime R5 reduced hydrogen absorption by 3.3 times compared to untreated ones. It was shown that hydrogen saturation of the Zr-1%Nb alloy ring samples to a concentration of ~ 400 ppm does not reduce its tensile and yield strength.

4. It was established that TCT is a promising method of influencing the mechanical characteristics of fuel rods made of zirconium alloys and their ability to absorb hydrogen. Consequently, in our opinion current work on the optimization of the TCT regime should be continued on longer rods samples and with higher hydrogenation levels.

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ВПЛИВ ХІМІКО-ТЕРМІЧНОЇ ОБРОБКИ НА ХАРАКТЕРИСТИКИ СПЛАВУ Zr-1%Nb

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Наведено дані щодо поліпшення властивостей твельних трубок зі сплаву Zr-1%Nb шляхом модифікування приповерхневого шару металу. Для модифікування використовували хіміко-термічну обробку (ХТО) у контрольованому газовому середовищі, яка полягала в дифузійному насиченні киснем приповерхневого шару металу. Установлено, що ХТО не знижує механічні властивості, які мали зразки сплаву Zr-1%Nb у початковому стані, а за деяких режимів підвищує їх. Розглянуто вплив ХТО зразків сплаву Zr-1%Nb за різними режимами на особливості їх окиснення на повітрі та на насичення воднем. Показано, що оптимальний режим ХТО зразків сплаву Zr-1%Nb зменшує приріст їх маси при окисненні за температури 550 °C на повітрі протягом 100 год і запобігає насиченню воднем. Зроблено висновок, що застосування ХТО є перспективним методом підвищення експлуатаційних характеристик твельних трубок зі сплаву Zr-1%Nb.