

V. S. Trush\*, I. M. Pohrelyuk, O. H. Luk'yanenko,  
S. M. Lavrys, T. M. Kravchyshyn

*Karpenko Physico-Mechanical Institute, National Academy of Sciences of Ukraine, Lviv, Ukraine*

\*Corresponding author: trushvasyl@gmail.com

## OXYGEN DIFFUSION SATURATION OF HAFNIUM WITH THE DIFFERENT SURFACE LAYER<sup>1</sup>

Hafnium is used for the production of control rods and protective screens in nuclear reactors after its hot plastic deformation and final thermochemical treatment. This research presents the experimental results of the influence of the surface layer state on the oxygen diffusion saturation of hafnium during thermochemical treatment in an oxygen-containing gas medium. Two cases of the surface layer state were considered: in the initial (delivery) state and after grinding, where 50  $\mu\text{m}$  of the surface layer was removed. Differences between the hardness distributions in the near-surface layer of HFE-1 hafnium after thermochemical treatment in an oxygen-containing gas medium were shown. It was found that thermochemical treatment of hafnium with a surface layer in its initial state leads to an increase of hardness in the near-surface layer compared to the surface: i.e., the near-surface layer hardness increases to a depth of 5 ... 7  $\mu\text{m}$ , and then gradually decreases to the matrix (core) hardness. It was determined that the fatigue life of hafnium after thermochemical treatment depends on the surface layer state. A 6.5 times higher fatigue life after thermochemical treatment of hafnium samples was fixed with a grinded surface compared to hafnium samples without grinding.

*Keywords:* hafnium, thermochemical treatment, oxygen, near-surface layer, hardness, fatigue life.

### 1. Introduction

Hafnium has a large neutron capture cross-section (capture resonance integral is  $\sim 105$  b); a high melting point ( $\sim 2503$  K), exceptional mechanical and anticorrosion properties in the harsh medium of pressurized water reactors [1 - 5]. Therefore, hafnium is used as a neutron absorber in protection control systems, mainly screens designed to reduce the neutron load on the nuclear reactor [6 - 9].

One of the stages in the manufacture of thin sheets of hafnium is rolling at high temperatures. The defects such as residual stress and inhomogeneous structure are formed in the near-surface layer after rolling. Also, hafnium has a high affinity to the interstitial elements (oxygen, nitrogen, carbon, and hydrogen) which can lead to the formation of undesirable oxide, nitride, carbide, and hydride compounds in the near-surface layer of hafnium [10 - 14]. In addition, a near-surface layer of hafnium can be enriched with chromium and iron, which are the main components of the steel rolling rolls [15, 16].

Final thermal treatment (850  $^{\circ}\text{C}$ , 2 h,  $1.33 \times 10^{-1}$  Pa) after rolling provides stress relief and dehydration of sheet hafnium [17 - 19]. However, such thermal treatment does not provide complete dissolution of hafnium oxides and carbides and removes chromium and iron from the surface layer.

As a result, such a defect near-surface layer will significantly affect the phase-structural state and performance, in particular, the fatigue life of hafnium products. Therefore, it would be expedient, after rolling, to remove the near-surface layer by machining, for example, by grinding. However, in the scientific and technical literature, attention is not focused on whether it is necessary to remove the surface layer of hafnium after hot plastic deformation.

Thermochemical treatment (TChT) in a vacuum can be replaced by treatment in a controlled, for example, oxygen-containing gas medium. At the TChT stage of thin-sheet hafnium in a high vacuum, it is possible to provide stress relief, and at the holding stage in an oxygen-containing gas medium – to form a modified near-surface layer based on oxygen, which will have a positive effect on fatigue characteristics [20 - 24].

The purpose of the research is to establish the effect of the pre-removal of the surface layer of the hafnium and treatment in an oxygen-containing gas medium on the physico-mechanical characteristics and fatigue life of thin-sheet hafnium.

### 2. Methodology

Thin-sheet ( $\sim 1$  mm) of HFE-1 hafnium after hot plastic deformation was studied. The samples with

© V. S. Trush, I. M. Pohrelyuk, O. H. Lukyanenko, S. M. Lavrys, T. M. Kravchyshyn, 2023

<sup>1</sup> Report at the XXIX Annual Scientific Conference of the Institute for Nuclear Research of the National Academy of Sciences of Ukraine, Kyiv, September 26 - 30, 2022.

dimensions of  $10 \times 20$  mm were used for metallographic and durometric tests. The first group of them was investigated in the initial state (without grinding). The second group of samples was ground with the removal of a  $50 \mu\text{m}$  surface layer. The samples of two groups were subjected to TChT in an oxygen-containing gas medium at a temperature of  $850^\circ\text{C}$  for 2 h, using laboratory equipment under rarefaction of the gaseous medium (Table 1) without leakage into the reaction chamber of the furnace.

Table 1. TChT regimes of hafnium

Symbol	Rarefaction of the gas medium
Initial state	Without TChT
<i>R1</i>	$P = 1.33 \cdot 10^{-3}$ Pa
<i>R2</i>	$P = 1.33 \cdot 10^{-2}$ Pa
<i>R3</i>	$P = 1.33 \cdot 10^{-1}$ Pa

The weight gain  $\Delta M$  ( $\mu\text{g}/\text{mm}^2$ ) was determined by weighing samples before and after TChT using a Voyager electronic balance with an accuracy of  $\pm 0.1$  mg. The surface roughness was measured by standard profilometer model 176021 with automatic determination of the roughness parameter  $R_a$ . The microhardness was determined using a PMT-3M durometer. Metallographic analysis was carried out via JEOL Superprobe 733 scanning electron microscopy.

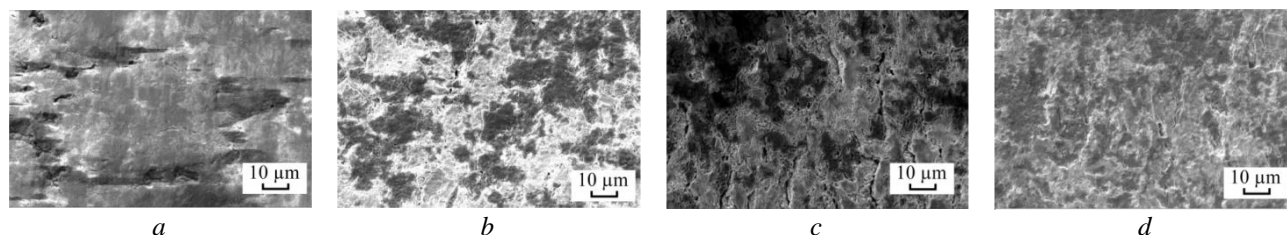
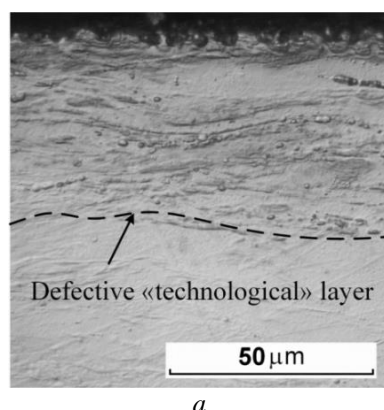


Fig. 2. Surface topography of hafnium in an initial state (*a*) and after TChT according to the regimes: *b* - *R1*, *c* - *R2*, *d* - *R3*.

In the near-surface layer after hot plastic deformation (in an initial state), it is observed a structure with grains elongated in the rolling direction



(Fig. 3, *a*). After TChT, the texture in the near-surface layer disappears (Fig. 3, *b*).

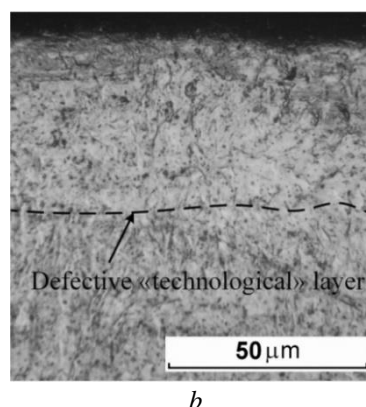


Fig. 3. Microstructure of the near-surface layer of hafnium in the initial state (*a*) and after TChT according to the regime *R2* (*b*).

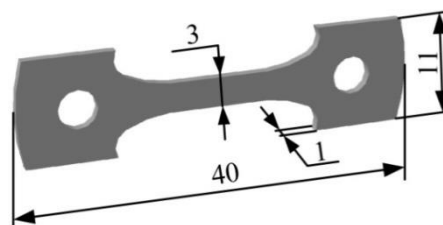


Fig. 1. Sample for fatigue test under pure bending.

The fatigue tests of the samples were performed under the symmetrical pure bending using a UMGU 01 machine manufactured at Karpenko Physico-Mechanical Institute of the NAS of Ukraine [25]. For fatigue tests, flat samples with a working part width of 3 mm and a thickness of 1 mm were used (Fig. 1). The scanning electron microscope was used for the fractographic analysis of the samples.

### 3. Results and discussion

#### 3.1. TChT of hafnium in an initial state (without grinding)

Hafnium surface after hot plastic deformation has defects: looseness, cracks, etc. The reason for their occurrence is related to the contact stress between the roll and thin-sheet hafnium. TChT in an oxygen-containing gas medium under different regimes affects surface relief (Fig. 2).

TChT of hafnium at various rarefactions of the oxygen-containing gaseous medium affects the chemical composition of the surface layer. According to the experimental results (Table 2), treatment both in high vacuum  $P = 1.33 \cdot 10^{-3}$  Pa (regime *R1*)

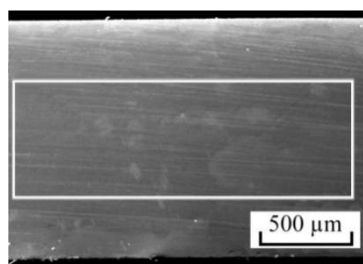
and at lower degrees of rarefaction  $P = 1.33 \cdot 10^{-2}$  Pa and  $P = 1.33 \cdot 10^{-1}$  Pa (regimes *R2* and *R3*, respectively) provides different oxygen content on the surface.

Table 2. Chemical composition of hafnium surface after TChT

Element	TChT regime							
	Initial state		<i>R1</i>		<i>R2</i>		<i>R3</i>	
	Content of elements							
	Wt.%	At. %	Wt.%	At. %	Wt.%	At. %	Wt.%	At. %
C	2.40	7.24	2.06	9.93	2.82	12.24	4.19	14.59
N	0.89	2.29	–	–	–	–	0.12	0.36
O	28.02	63.36	11.08	40.03	15.07	49.06	19.48	50.88
Cr	0.75	0.52	0.60	0.67	0.57	0.57	1.09	0.88
Fe	24.43	15.83	30.12	31.18	22.36	20.86	30.53	22.85
Zr	10.01	3.97	–	–	–	–	–	–
Hf	33.50	6.79	56.14	18.19	59.18	17.27	44.59	10.44
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

It should be noted that the chemical composition of the core (matrix) of the sample in the initial state differs from the chemical composition of the surface: the metal matrix contains only hafnium, carbon, and oxygen (Fig. 4) and does not contain other elements, namely, chromium, iron, zirconium. The

high content of carbon and oxygen on the surface of the thin section of the hafnium core is associated with its high affinity to these elements, which were diffused to the matrix during the preparation of the metallographic cross-section.



Element	Wt.%	At. %
C	2.12	21.55
O	1.65	12.57
Hf	96.23	65.87
Total	100.00	100.00

Fig. 4. General appearance of the cross-section (*a*) and chemical composition (*b*) of the hafnium matrix in an initial state.

The following results were obtained during studying the linear distribution of chemical elements on the cross-section of hafnium in an initial state (Fig. 5). In the near-surface 50  $\mu\text{m}$ -thick layer from the surface, the distribution of oxygen and iron, zirconium, and chromium elements was recorded. The hafnium content is minimal to a depth of 7 ... 10  $\mu\text{m}$  from the surface, and deeper it increases sharply to a constant value. Therefore, in an initial state, the near-surface layer up to 50  $\mu\text{m}$  can be classified as a defective «technological» layer. The presence of this layer is probably due to the formation of various oxides based on iron, zirconium, and chromium. This chemical composition of the surface may be related to the transfer of the lubricant protective substance to the surface of the sheet material during hot rolling.

The dissolution of interstitial element (oxygen) in hafnium and the formation of a solid solution in the near-surface layer causes an increase in hardness. Therefore, after various treatment regimes, the microhardness of the surface and its distribution in the near-surface layer were determined. According to the obtained results, the surface hardness after different treatment regimes varies in the range from  $H^{\text{surf.}} = 320 \pm 36 \text{ HV}_{0.49}$  to  $H^{\text{surf.}} = 500 \pm 59 \text{ HV}_{0.49}$  (Fig. 6 and Table 3). The largest increase in surface hardness ( $\Delta H^{\text{surf.}} = 260 \text{ HV}_{0.49}$ ) fixed after TChT in a vacuum  $P = 1.33 \cdot 10^{-1}$  Pa (regime *R3*), and the smallest increment ( $\Delta H^{\text{surf.}} = 80 \text{ HV}_{0.49}$ ) was after treatment in a high vacuum (regime *R1*), which is explained by the different concentration (content) of the active components in the gas medium. The increase of surface hardness according to regime *R2*

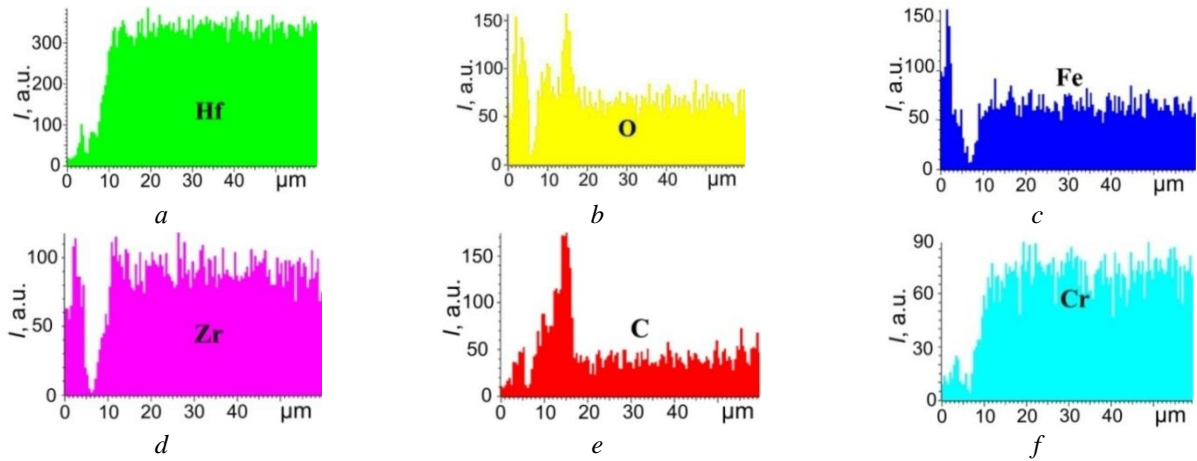


Fig. 5. Distribution of chemical elements in the cross-section of the near-surface layer of hafnium in an initial state. (See color Figure on the journal website.)

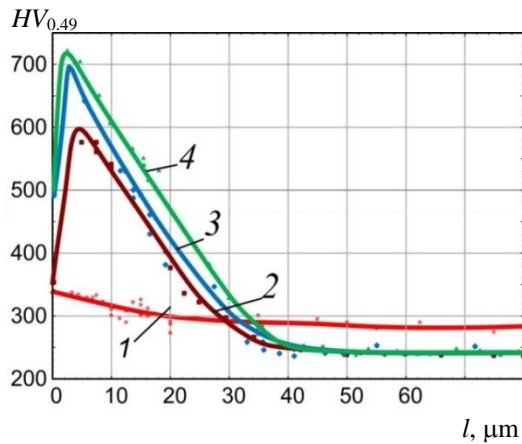


Fig. 6. Distribution of microhardness on the cross-section of hafnium after TChT according to the regimes: 1 - initial state; 2 - R1; 3 - R2; 4 - R3. (See color Figure on the journal website.)

(pressure of the gas medium was  $P = 1.33 \cdot 10^{-2}$  Pa) was  $\Delta H^{surf.} = 255 HV_{0.49}$ . The depth of the hardened zone after various treatments is almost the same and is equal to 40 ... 45  $\mu m$  (see Table 3), which corresponds to the same time-temperature parameters of treatments. It should be noted that in the initial state, the hafnium near-surface layer is hardened to a depth of  $\sim 15 \mu m$ , and the increment of surface hardness compared to the core is  $\Delta H^{surf.} = 80 HV_{0.49}$ .

The microhardness distribution in the near-surface layer (see Fig. 6) after TChT has certain features. In particular, in the case of the formation of a diffusion layer, the surface hardness should be greater or at the level of the hardness of the matrix. However, as follows from the curves (see Fig. 6), the hardness after various treatments increases from the surface to a depth of 5 ... 7  $\mu m$ , and then gradually decreases to the value of the matrix. A certain loosened layer with a depth of 7 ... 10  $\mu m$  is also fixed by the durometric method.

Table 3. Durometric and profilometric analysis of hafnium before and after TChT

Regime	Microhardness $HV_{0.49}$			Depth of hardened layer $l, \mu m$	Surface roughness $Ra, \mu m$		Weight gain $\Delta M, mg/mm^2$
	Surface		core		before	after	
	before	after					
Initial state		–	$260 \pm 10$	15	0.238		–
R1	$340 \pm 32$	$320 \pm 36$	$240 \pm 15$	40 ... 45	0.230	0.233	0.708
R2		$495 \pm 56$			0.227	0.220	0.937
R3		$500 \pm 99$			0.219	0.216	0.961

The surface roughness of hafnium after TChT regimes remain unchanged, and the weight gain increases with a decrease in the rarefaction of the oxygen-containing medium (see Table 3).

For a more detailed study of the hardness distribution of the near-surface layer of hafnium, it was decided to check whether this effect would be observed under other loads during micro-indentation. For this purpose, the microhardness distribution

under different loads during micro-indentation was additionally investigated on the sample after treatment according to regime R2 (Fig. 7): 0.049, 0.098, 0.196, 0.98, 1.96, and 2.94 H.

It was found that at indenter loads from 0.049 to 1.96 H, the character of the hardness distribution is the same. However, at the high indenter load (2.94 H) the character of the hardness distribution is different: there is no “peak” of hardness at a depth of

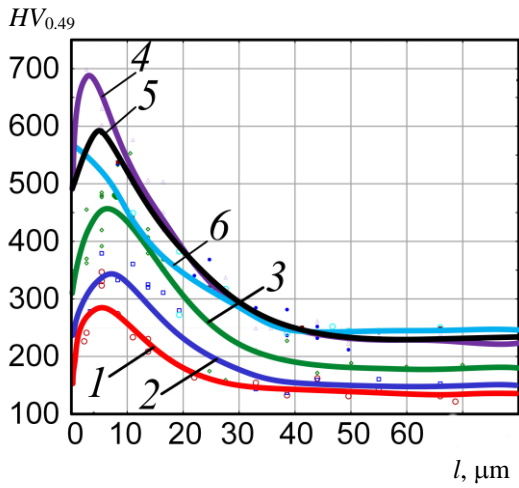


Fig. 7. Distribution of microhardness on the cross-section of hafnium after TChT according to the regimes R2 at different loads during indentation: 1 - 0,049 H; 2 - 0,098 H; 3 - 0,196 H; 4 - 0,98 H; 5 - 1,96 H; 6 - 2,94 H. (See color Figure on the journal website.)

5 ... 7 μm, where the hardness gradually decreases from the surface to the matrix. It is probably caused by a larger imprint area than the defective “technological” layer during the determination of the hardness under a load of 2.94 H.

**3.2. TChT of grinding hafnium samples with the removal of 50 μm surface layer**

To reduce the influence of the defective “technological” layer on the properties of hafnium after rolling and TChT, a near-surface layer with a thickness of 50 μm was removed from each side of the sheet. Such treatment contributes to a significant improvement in the surface quality (Fig. 8). The surface roughness decreased (Table 4).

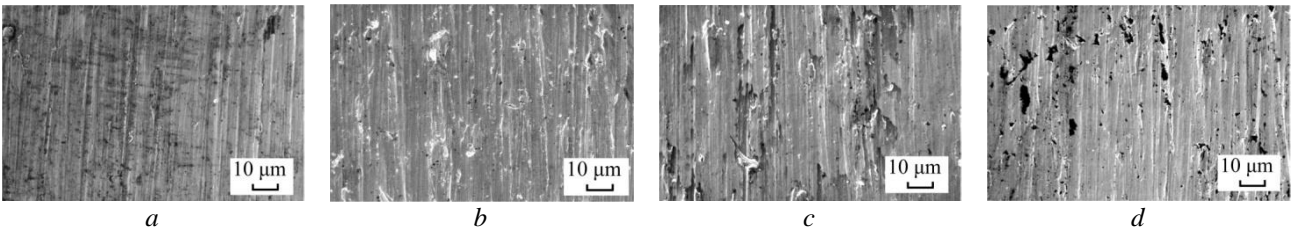


Fig. 8. Surface topography of the grinded hafnium (a) and after TChT according to the regimes: b - R1, c - R2, d - R3.

Table 4. Durometric and profilometric analysis of ground hafnium before and after TChT

Regime	Microhardness $HV_{0,49}$			Depth of hardened layer $l, \mu m$	Surface roughness $Ra, \mu m$		Weight gain $\Delta M, mg/mm^2$
	Surface		core		before	after	
	before	after					
R1	$275 \pm 20$	$270 \pm 22$	$240 \pm 15$	15 ... 20	0.039	0.041	0.116
R2		$340 \pm 10$			0.040	0.038	0.226
R3		$345 \pm 12$			0.031	0.030	0.282

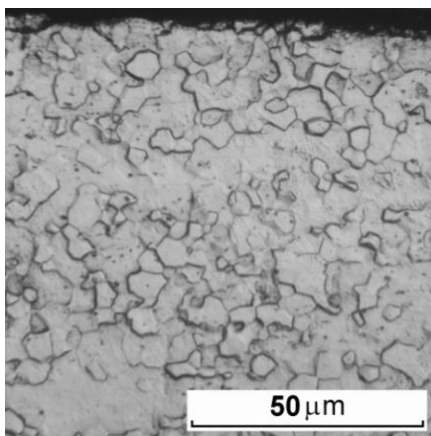


Fig. 9. Microstructure of the near-surface layer of ground hafnium after TChT according to the regime R2.

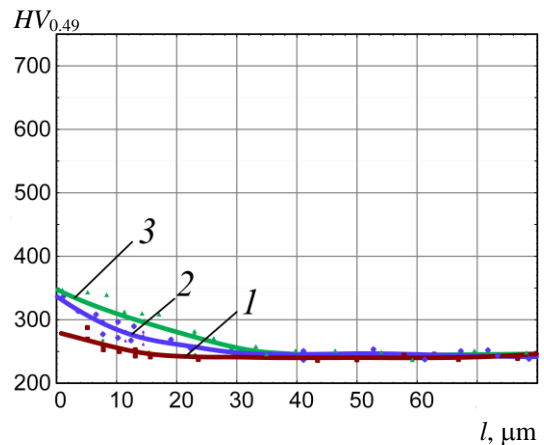


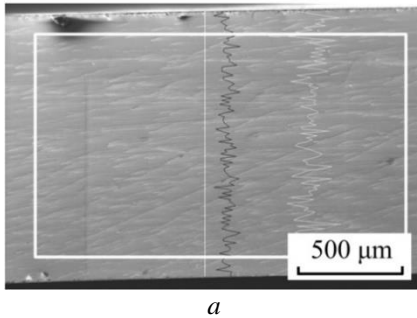
Fig. 10. Microhardness distribution on the cross-section of ground hafnium after TChT according to the regimes: 1 - R1; 2 - R2; 3 - R3. (See color Figure on the journal website.)

In the near-surface layer of ground hafnium after TChT, polyhedral grains size 7 - 15 μm are observed (Fig. 9).

TChT of ground hafnium according to regimes *R1*, *R2*, and *R3* ensured the formation of a near-surface layer with a cross-sectional hardness distribution (Fig. 10) different from that of hafnium without grinding (Fig. 6). After TChT by all regimes *R1*, *R2*, *R3*, the hardness monotonically decreases from the surface to the core, i.e., the distribution

corresponds to classical diffusion hardening by the interstitial elements.

According to the analysis of the chemical composition of the ground hafnium with the removed (50 μm) defective surface layer, the following elements were found: hafnium, oxygen, and carbon (Fig. 11). The reason for the presence of carbon and oxygen on the surface of the cross-section is the same as in the previous case.



Element	Wt. %	At. %
C	2.20	22.72
O	1.34	10.38
Hf	96.45	66.90
Total	100.00	100.00

Fig. 11. General appearance of the cross-section (*a*) and chemical composition (*b*) of ground hafnium.

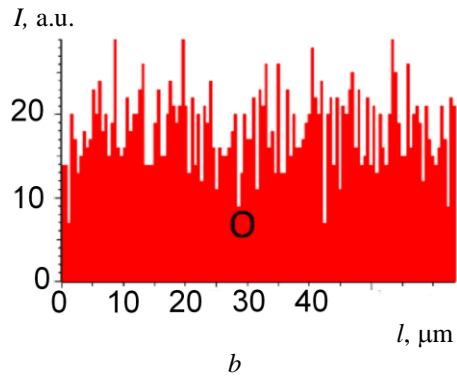
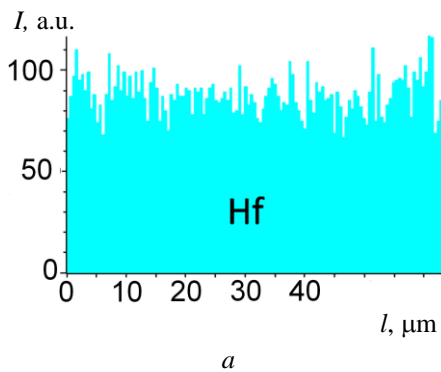


Fig. 12. Distribution of chemical elements on the cross-section of the near-surface layer of ground hafnium with the removed surface layer (50 μm). (See color Figure on the journal website.)

The analysis of the distribution of chemical elements on the cross-section of near-surface layers of ground hafnium with the removed surface layer (50 μm), proved the absence of the impurities (Figs. 11 and 12).

### 3.3. Fatigue life under pure bending

The fatigue life of hafnium under pure bending at the deformation amplitude  $\pm \epsilon_a = 0.6\%$  after surface treatment by various regimes in the initial state (Fig. 13, *a*) and with the removed surface layer

(Fig. 13, *b*) have significant differences. The fatigue life of the ground hafnium after TChT was higher than that of treated hafnium without pre-grinding. For example, the fatigue life of grinding hafnium after TChT according to the regime *R2* was 980 thousand cycles, and before grinding – 150 thousand cycles. In addition, the deviation of the number of cycles to failure from the average fatigue life after grinding hafnium after TChT according to the regime *R2* was only 4 - 7 %, but before grinding hafnium was 40 - 45 %.

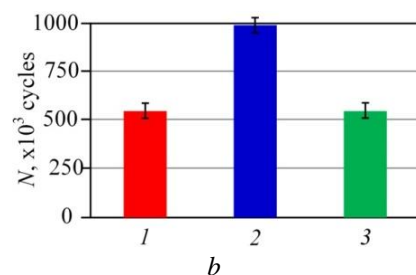
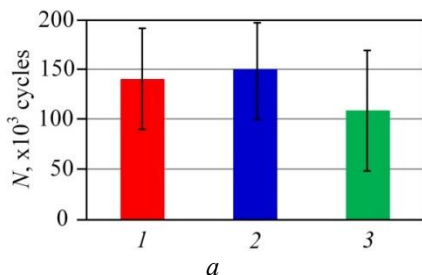


Fig. 13. Fatigue life under pure bending ( $\pm \epsilon_a = 0.6\%$ ) of before grinding (*a*) and after grinding (*b*) hafnium after TChT according to the regimes: 1 - *R1*; 2 - *R2*; 3 - *R3*. (See color Figure on the journal website.)

Therefore, TChT in an oxygen-containing gas medium (regimes *R2* and *R3*) provided an increase in fatigue life compared to treatment in a vacuum (regime *R1*) after grinding and before grinding hafnium. However, pre-grinding allowed for improving the fatigue life and significantly increases the number of cycles to failure compared to before grinding

hafnium after the proposed TChT regimes.

According to the fractographic analysis, in the surface layer of the before grinding hafnium, an inhomogeneous and loose defective “technological” 5 - 7  $\mu\text{m}$  thick layer was observed (Fig. 14, *a, b, c*). This probably causes a large deviation in the fatigue life of hafnium.

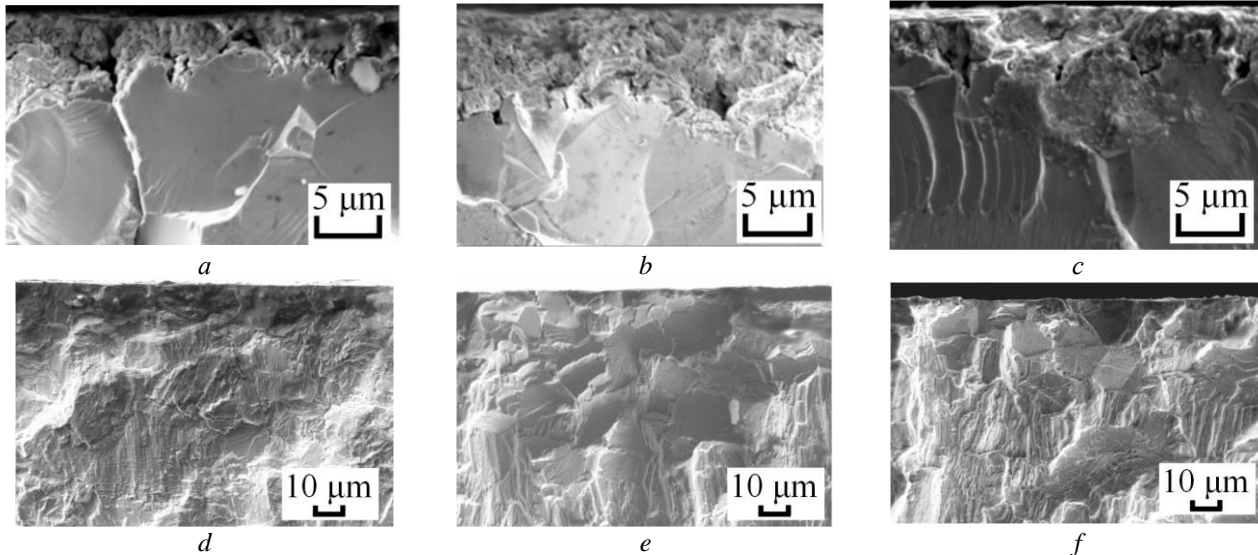


Fig. 14. Fracture surface of before (*a, b, c*) and after (*d, e, f*) grinding hafnium after fatigue tests by pure bending at  $\pm \varepsilon_a = 0.6\%$  after TChT according to the regimes: *a, d* - *R1*; *b, e* - *R2*; *c, f* - *R3*.

Such a defective technological layer is not observed on ground hafnium (Fig. 14, *d, e, f*). The investigated material in the bulk (regimes *R1, R2, R3*) is destroyed according to a mixed ductile-brittle mechanism, where the characteristic lamellar fracture structure is associated with the anisotropy of the structure of the rolled material (see Fig. 14, *d, e, f*). The presence of areas with fatigue striations (regime *R1*) testifies to the micro-viscous nature of the fracture.

The structures of the fracture surfaces in the subsurface zones are absolutely different. After the TChT is carried out in a high vacuum (regime *R1*), the subsurface layer saturated with oxygen fails at a distance of  $\sim 15\ \mu\text{m}$  from the surface according to the mechanism of fatigue quasi cleavage with the appearance of isolated brittle intergrain facets (see Fig. 14, *d*).

As the degree of rarefaction of the TChT medium (regime *R2*) decreases, the subsurface zone up to  $\sim 30\ \mu\text{m}$  in size mainly undergoes brittle fracture, and we observe clearly visible cleavages of intergrain facets (see Fig. 14, *e*).

As a result of the low-vacuum TChT (regime *R3*), the size of the oxygen-saturated layer remains practically invariable ( $\sim 35\ \mu\text{m}$ ) but the intensity of brittle intergrain fracture increases, which is confirmed by a considerable number of secondary intergrain microcracks (see Fig. 14, *f*).

### 3. Conclusions

In this research, the differences in the distribution of hardness in the near-surface layer and fatigue life of the hafnium after TChT in an oxygen-containing gas medium with the removed defective near-surface layer (after grinding) and not removed were shown.

It was found that for hafnium without removal of the surface layer after hot plastic deformation as a result of TChT in an oxygen-containing gaseous medium ( $T = 850\ \text{°C}$  for  $\tau = 2\ \text{h}$ ) in the near-surface layer, the hardness increases from the surface to a depth of 5 ... 7  $\mu\text{m}$ , and then decreases to the matrix stiffness values. Therefore, after the hot plastic deformation of hafnium, a defective “technological” layer of hafnium should be removed from the surface due to its chemical and structural heterogeneity, which can lead to premature failure of the product.

It was shown that TChT in an oxygen-containing medium (regime *R2* and *R3*) provided an increase in fatigue life compared to TChT in a vacuum (regime *R1*) for hafnium without removal and with a removed surface layer. Moreover, the TChT in the oxygen-containing medium after grinding hafnium specimens significantly increases the number of cycles to failure.

## REFERENCES

1. R.H. Nielsen, G. Wilfing. Hafnium and Hafnium Compounds. In: *Ullmann's Encyclopedia of Industrial Chemistry* (Weinheim, Wiley-VCH Verlag GmbH & Co. KGaA, 2012) p. 191.
2. H.W. Keller et al. Development of hafnium and comparison with other pressurized water reactor control rod materials. *Nuclear Technology* 59(3) (1982) 476.
3. V.D. Risovany, E.P. Klochkov, V.B. Ponomarenko. *Hafnium in Nuclear Engineering* (American Nuclear Society, 2001) 101 p.
4. M.M. Pylypenko et al. Pure hafnium for nuclear power. *Problems of Atomic Science and Technology* 1(125) (2020) 3.
5. M.L. Kotsar et al. Iodide hafnium. Receipt, composition, properties and application in materials of regulating units of nuclear reactors. *Problems of Atomic Science and Technology* 2(90) (2014) 78.
6. A.A. Afanas'ev, Yu.F. Konotop, N.P. Odeichuk. Hafnium is a promising absorber for absorbing elements of the systems of control and protection at VVER-1000 reactors of the NPP of Ukraine. *Problems of Atomic Science and Technology* 4 (2000) 80. (Rus)
7. H.W. Huang et al. A Hafnium Material Control Rod Design and Manufacture. *Advanced Materials Research* 338 (2011) 136.
8. Yu.F. Konotop, N.P. Odeichuk, V.S. Krasnorutskii. Current State of the Problem of Neutron-Absorbing Materials and Products Made on the Basis of these Materials for the VVER-1000-Type Reactors (Kharkiv, Kharkiv Physical and Technical Institute, 1998) p. 68. (Rus)
9. R.J.H. Clark, D.C. Bradley, P. Thornton. *The Chemistry of Titanium, Zirconium and Hafnium* (Oxford, England: Pergamon Press, 1975) 151 p.
10. D.A. Negodin et al. Commercial production of metal hafnium and hafnium-based products. *Problems of Atomic Science and Technology* 78 (2012) 97. (Rus)
11. D.E. Thomas, E.T. Hayes (Eds.) *The Metallurgy of Hafnium*. (Naval Reactors, Division of Reactor Development, U. S. Atomic Energy Commission, 1960) 384 p.
12. J.H. Choi, Y. Mao, J.P. Chang. Development of hafnium based high-k materials - A review. *Mat. Sci. and Eng. R: Reports* 72 (2011) 97.
13. I.M. Neklyudov et al. New technology for the production of nuclear-quality hafnium. *Nauchnye Vedomosti Belgorodskogo Gosudarstvennogo Universiteta. Ser. Fizika* 4(14) (2001) 127. (Rus)
14. H. Jehn, E. Fromm, E. Gebhardt. *Gases and Carbon in Metals*. Transl. from Germ. (Moskva: Metallurgy, 1980) 712 p. (Rus)
15. I.M. Neklyudov et al. Preparation for the production of hafnium rods. *Science and Innovation* 6(6) (2010) 5. (Rus)
16. D.A. Negodin et al. Commercial production of metal hafnium and hafnium-based products. *Problems of Atomic Science and Technology* 2(78) (2012) 97. (Rus)
17. V.S. Krasnorutsky, V.A. Zuyok, N.M. Roenko. Influence of deformation and annealing on structure and texture bar from calciumthermic hafnium. *Problems of Atomic Science and Technology* 3(86) (2005) 108. (Rus)
18. I.M. Neklyudov et al. Preparation for the production of constructional materials from hafnium. *Science and Innovation* 5 (2009) 23. (Rus)
19. R.V. Azhazha et al. Influence of annealing on a structure and properties of high temperature rolled hafnium. *Problems of Atomic Science and Technology* 1(17) (2008) 27. (Rus)
20. V.M. Fedirko, O.H. Luk'yanenko, V.S. Trush. Influence of the diffusion saturation with oxygen on the durability and long-term static strength of titanium alloys. *Mater. Sci.* 50 (2014) 415.
21. V.M. Fedirko et al. Effect of thermochemical treatment in regulated gas media on the thermal resistance of Zr1%Nb alloy. *Mater. Sci.* 52 (2016) 209.
22. V.N. Fedirko, A.G. Luk'yanenko, V.S. Trush. Solid-Solution Hardening of the Surface Layer of Titanium Alloys. Part 2. Effect on Metallophysical Properties. *Metal Science and Heat Treatment* 56 (2015) 661.
23. V.S. Trush et al. Influence of the functional layer on the operating characteristics of Zr - 1% Nb alloy at a temperature of 380 °C. *Mater. Sci.* 57 (2021) 234.
24. I.M. Pohrelyuk et al. Topography, hardness, elastic modulus and wear resistance of nitride coatings on titanium. In: *9th International Scientific Conference, dedicated to 100th Anniversary of Restitution of Lithuania, Aleksandras Stulginskis University, Kaunas, Lithuania, 16 - 17 November 2017. Proceedings* (Kaunas: Aleksandras Stulginskis University, 2017) p. 41.
25. G.G. Maksimovich. *Micromechanical Investigations of the Properties of Metals and Alloys* (Kyiv: Naukova Dumka, 1974) 243 p. (Rus)



**В. С. Труш\*, І. М. Погрелюк, О. Г. Лук'яненко, С. М. Лаврись, Т. М. Кравчишин**

*Фізико-механічний інститут ім. Г. В. Карпенка НАН України, Львів Україна*

\*Відповідальний автор: trushvasyl@gmail.com

### **ДИФУЗІЙНЕ НАСИЧЕННЯ КИСНЕМ ГАФНІЮ З РІЗНИМ ПОВЕРХНЕВИМ ШАРОМ**

Для виробництва регулюючих стрижнів та захисних екранів у ядерних реакторах використовують гафній, який після гарячого пластичного деформування піддають фінішній термообробці. У роботі наведено експериментальні результати впливу стану поверхневого шару на дифузійне насичення гафнію із кисневмісного газового середовища. Розглянуто два випадки стану поверхневого шару: у стані постачання з поверхневим шаром після гарячого пластичного деформування та з видаленими 50 мкм поверхневого шару. Показано відмінності розподілу твердості у приповерхневому шарі зразків гафнію ГФЕ-1 після термообробки у кисневмісному газовому середовищі. Зокрема, виявлено, що на зразках із поверхневим шаром після гарячого пластичного деформування термообробка у кисневмісному газовому середовищі призводить до зростання твердості у приповерхневому шарі, відносно поверхні: тобто твердість до глибини 5 ... 7 мкм збільшується, а потім поступово зменшується до значень твердості матриці. Показано, що втомна довговічність тонколистових зразків гафнію ГФЕ-1 термооброблених у кисневмісному газовому середовищі без зняття поверхневого шару після гарячого прокатування – 150 тис. циклів, а зі зняттям поверхневим шаром становить 990 тис. циклів.

*Ключові слова:* гафній, термообробка, приповерхневий шар, кисень, твердість, втомна довговічність.

Надійшла/Received 20.01.2023