

TRIBOLOGICAL CHARACTERISTICS OF 'STEEL – CERAMICS-IRRADIATED-BY-HELIUM-IONS' PAIR

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Facility for the tribological characteristics studying of a metal – ceramics pair and parameters of ceramic samples irradiation on the helium ions linear accelerator with energies 0.12 and 4 MeV are resulted. Profiles of damageability and occurrence of target atoms along of helium ions range are calculated for the irradiated TiO₂ and Al₂O₃. Sputtering ratios and dependence of the sputtered atoms quantity on samples thickness are received. Calculations on density change of the irradiated samples are made. Experimental results of a sliding friction factor measurement depending on cycle's quantity, temperature and irradiation doses are presented. On the basis of microscopic researches and calculation data conclusions are drawn about irradiation influence on interacting pair's tribological characteristics.

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INTRODUCTION

Development of nuclear and thermonuclear power demands creation of the new antiwear materials which can to work in the conditions of as much as possible wide range of temperatures, radiating fields, loadings and etc. [1]. Perspective materials for use in the loaded friction units of nuclear power facilities are the transformational hardened ceramic materials [2, 3] which often work together with metal. Transfer of a metal and its oxides on a ceramics surface carries out protective function, increasing its endurance [4].

The friction forces are shown at all stages of the fuel assembly (FA) life cycle [5–9] in atomic power engineering. The insufficient attention to them led to fretting-damages of fuel elements covers with undesirable consequences [8, 9]. The basic mechanism responsible for destruction fuel elements at capacity change is mechanical influence of extending fuel on a cover. Such influence is caused by various temperature of the core and cover and several bigger (in 1.5–2 times) thermal expansions of the fuel ceramic tablets in comparison with a metal cover. This circumstance limits capacity maneuvering possibilities of the reactor facility [5, 6]. The irradiation leads to redistribution of atoms in materials structure, to formation of a blistering and flaking, to breach of the interacting elements surface.

For today the question of the new wearproof materials creation which can to work in severe constraints of nuclear and thermonuclear facilities is very topical. Investigation of tribological characteristics of ceramic materials which are used in various branches of a science and technics the great number works [10–15] is devoted.

The work purpose is research of tribological characteristics of a metal – ceramics irradiated by helium ions pair on the developed and made facility at NSC KIPT.

1. IRRADIATION PARAMETERS AND INITIAL SAMPLES

The helium ion (He⁺) linear accelerator, which has a range of energies from 0.12 up to 4 MeV, operates at

NSC KIPT. This machine is used in a great variety of studies [16, 17].

The chamber for an irradiation of samples and system of experimental parameters measurement [17, 18] is created. The turbo-molecular pump provides the oxygen-free environment in a chamber volume. The temperature of irradiated samples is set by the heating element located directly in the irradiation chamber and it's measured by the thermocouple. The focusing triplet is established in front of the chamber for change of current density of a beam, falling on the sample. It allows changing a beam radius with preservation of the general current, depending on experiment requirements [19]. Beam currents are measured by contactless flying gauges which are established on input and output of the triplet, and as before the irradiated sample. Digital devices which are connected to the computer with the further data recording and their processing are used for registration of irradiation parameters [18]. The basic beam parameters at an irradiation of samples on the helium ions linear accelerator are resulted in Table 1.

Table 1

Irradiation parameters

| Parameter | Value |
|----------------------|--|
| Beam energy | 0.12...4 MeV |
| Pulse current | 700 μA |
| Pulse length | 500 μs |
| Repetition frequency | 2...5 imp./s |
| Average current | 0.7 μA |
| Current density | (0.15...0.44) · 10 ¹³ part./cm ² |
| Temperature | up to 900 °C |

After samples preparation Al₂O₃ and TiO₂ to doses 10¹⁸ part./cm² at 0.12 MeV energy of the helium ions beam have been irradiated. This energy has been chosen for the purpose of the maximum surface damageability. Samples from TiO₂ and Al₂O₃ which were irradiated on the linear accelerator of helium ions are shown in Fig. 1 (samples diameter is 15 mm).



Fig. 1. TiO_2 (left), Al_2O_3 (right) samples

Surfaces photos of not irradiated samples are given in Fig. 2. Roughnesses of the surfaces measured on a microscope it's not revealed.

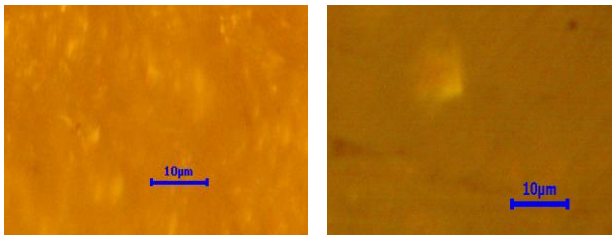


Fig. 2. Al_2O_3 (left) and TiO_2 (right) surfaces of the not irradiated samples

The metal sample has been made from alloy steel, its sizes are $50 \times 23 \times 14$ mm. Structure became in percentage: Fe – 92.087; Na – 3.181; Cr – 1.478; Mg – 1.247; Si – 0.827; Mg – 0.514; Cu – 0.206; Ni – 0.186; Co – 0.155; S – 0.118; class – 1.

2. FACILITY FOR TRIBOLOGICAL RESEARCHES

In consideration of irradiation specificity and research of tribological interaction necessary parameters such scheme has been chosen (Fig. 3).

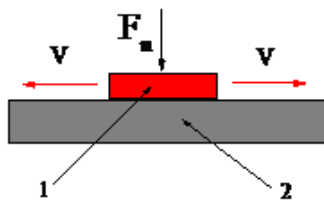


Fig. 3. Scheme of samples contacting in facility, where 1 – TiO_2 or Al_2O_3 , 2 – steel

Facility for tribological characteristics studying of a metal – ceramics pair is shown in Fig. 4.

The reversible asynchronous electric motor RD-09 (see Fig. 4, pos. 1) with rotation frequency of a reducer output shaft to 8.7 rpm is applied to relative moving of interacting samples. The pressure (force) tensometric gauge DYMH-103 (see Fig. 4, pos. 2) is used for definition of a friction force. The basic technical characteristics of a gauge are resulted in Table 2. The gauge spent graduation has linear character.

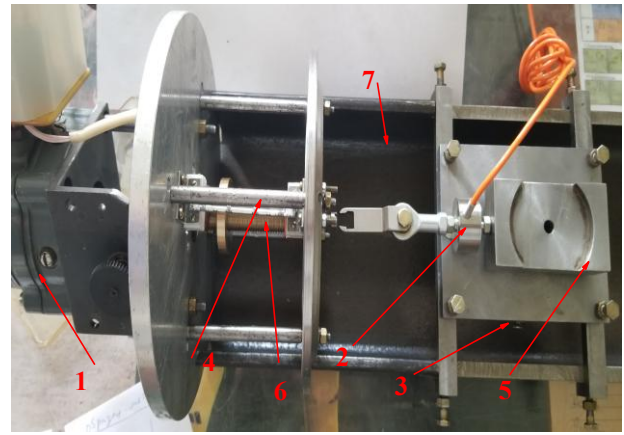


Fig. 4. Facility for tribological characteristics studying: where 1 – reversible asynchronous electric motor RD-09; 2 – pressure (force) tensometric gauge DYMH-103; 3 – infra-red heating element; 4 – resistor with two microswitches; 5 – holder of the ceramic sample and loading weights; 6 – transformation mechanism of a rotary motion in forward; 7 – base

Table 2

Pressure (force) gauge characteristics

| Parameter | Value |
|---------------------|---|
| Measurement range | $(-3) - 0 - (+3)$ kg |
| Measurement error | 0.05 ~ 0.1% |
| Sensitivity | 1.0...1.5 mV/V |
| Temperature error | $\pm 0.5\%$ |
| Working temperature | $(-35\text{ }^\circ\text{C}) - (+85\text{ }^\circ\text{C})$ |

The holder is made (see Fig. 4, pos. 5) for fastening of the ceramic sample and maintenance of vertical loading on a frictional pair. Its appearance with the pressure gauge is shown in Fig. 5.

The infra-red heating elements (260 and 450 W) are used (see Fig. 4, pos. 3) for heating of samples. Their appearance is shown in Fig. 6. The heater in 260 W allows to carry out heating of a metal – ceramics pair to $400\text{ }^\circ\text{C}$, and the heater in 450 W – to $900\text{ }^\circ\text{C}$. This or that heating element is used depending on an assigned task on a temperature range.

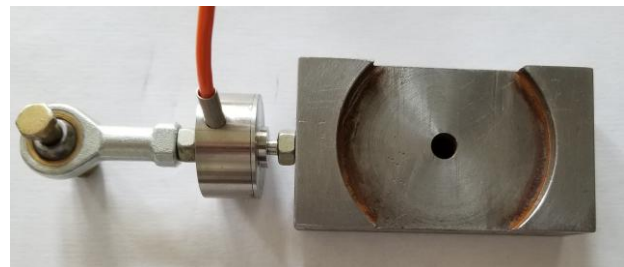


Fig. 5. Holder of samples with pressure (force) tensometric gauge



Fig. 6. Appearance of heating elements. 260 W (60×60 mm) – left, 450 W (80×80 mm) – right

A resistor with two microswitches (see Fig. 4, pos. 4) is used for definition of a relative moving distance of samples and engine RD-09 switching. Its calibrating curve has linear character and completely covers a measurement range of relative moving. This facility provides static loading on a frictional pair. For dynamic loading maintenance it's necessary to create hydraulic, pneumatic or electromechanical loading unit. The transformation mechanism of a rotary motion in forward (see Fig. 4, pos. 6) allows to regulate in a wide interval moving speed of interacting samples. Fastening of the facility all elements it's carried out on a base (see Fig. 4, pos. 7).

Thus, facility provides temperature maintenance and registration on samples to 900 °C, engine RD-09 and the transformation mechanism of a rotary motion in forward supports a full cycle of frictional interaction in a range of 1...9 min. During carrying out of measurements it's possible to receive dependence of a sliding and rest friction factors on time, cycle's quantities and temperature, and as all listed characteristics from of radiating damages doses.

3. CALCULATED CHARACTERISTICS OF THE IRRADIATED SAMPLES

For calculation of ions range in solids software package SRIM [20, 21] was used. This program allows to receive following information about: vacancies distribution in a target; redistribution of irradiated materials atoms; sputtering ratios; phenomena connected with ions energy loss; the distribution of ionization and formation phonons. All listed calculations, taking into account displacement cascades have been carried out for TiO₂ and Al₂O₃ in program SRIM. Energy losses going on ionization, formation phonons and damageability, both helium ions beam, and the displacement cascade are resulted in Table 3.

Table 3
Calculated characteristics of the irradiated samples,
E = 0.12 MeV

| Element | Energy loss, % | | |
|--------------------------------|--------------------------|--------------------------|--------------------------|
| | Ionization | Phonons | Damageability |
| | He ⁺ /cascade | He ⁺ /cascade | He ⁺ /cascade |
| Al ₂ O ₃ | 93.6/1.19 | 0.79/4.16 | 0.08/0.21 |
| TiO ₂ | 93.7/1.10 | 0.78/4.15 | 0.08/0.2 |

The greater part of a beam energy goes on ionization. The basic contribution to phonons formation and damageability is brought by displacement cascades.

On formation phonons it's spent at 17–18 times more energy, than for damageability. The helium ions range in TiO₂ and Al₂O₃ make 0.6...0.7 μm.

Profiles of damageability and occurrence helium and atoms of materials Al₂O₃ and TiO₂, irradiated on the accelerator with energy of 0.12 MeV are resulted in relative units in Figs. 7, 8.

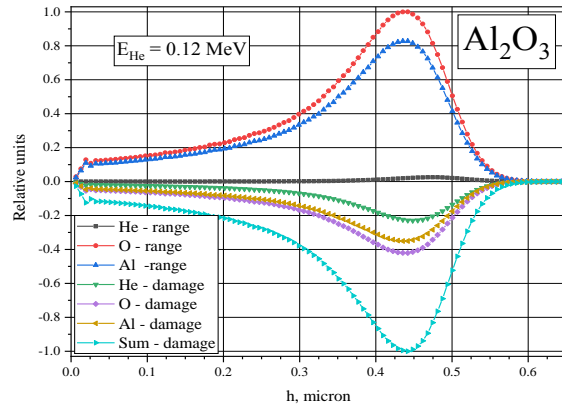


Fig. 7. Damageability (bottom curves) and occurrence profiles helium, oxygen and aluminum (top curves) in Al₂O₃

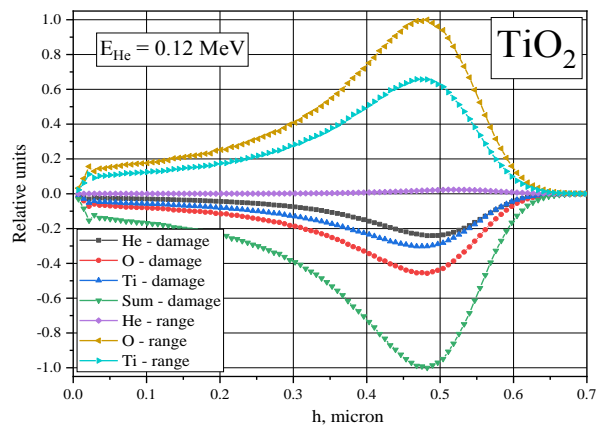


Fig. 8. Damageability (bottom curves) and occurrence profiles helium, oxygen and titanium (top curves) in TiO₂

From graphs follows that in irradiated samples there is a redistribution of materials atoms along of helium ions range. The basic contribution to damageability is brought by displacement cascade formed at an irradiation by helium ions in process of dissociation Al₂O₃ and TiO₂. These processes lead to change of the irradiated material density along of helium ions range.

Relations of Al₂O₃ and TiO₂ atoms to He, formed at an irradiation, and the general damageability are resulted in Table 4.

Table 4
Relation of Al₂O₃ and TiO₂ atoms to He⁺, formed at an irradiation, and damageability

| Al ₂ O ₃ | | TiO ₂ | |
|--------------------------------|-------|-------------------|-------|
| Al/He | O/He | Ti/He | O/He |
| 55.69 | 66.70 | 46.47 | 69.09 |
| Damageability | | Damageability | |
| 118.3 vacancy/ion | | 144.5 vacancy/ion | |

The dose irradiated Al_2O_3 and TiO_2 has made 10^{18} ion/cm². Using Table 4 and Figs. 7, 8 data changes of density along ions range have been recounted. Calculations have shown density minor alteration, no more 2%, through maxima shift of atoms range concerning damageability. Therefore, this influence can ignore at frictional interaction of a metal – Al_2O_3 , TiO_2 pairs. At higher irradiation energies and doses, it's necessary to pay attention to change of materials density.

A sputtering ratio of material atoms is played the important role in a choice of a perspective material for the FR first wall and diverter. Sputtering ratios for Al_2O_3 and TiO_2 are presented in Table 5.

Table 5
Quantity of Al_2O_3 and TiO_2 sputtered atoms

| Beam energy, $E_{\text{He}} = 0.12$ MeV | Quantity of sputtered atoms, atom/ion | |
|--|---------------------------------------|----------------------|
| Al_2O_3 | Al | $1.47 \cdot 10^{-2}$ |
| | O | $2.14 \cdot 10^{-2}$ |
| Sputtering ratio | $\approx 3.61 \cdot 10^{-2}$ | |
| TiO_2 | Ti | $0.63 \cdot 10^{-2}$ |
| | O | $2.33 \cdot 10^{-2}$ |
| Sputtering ratio | $\approx 2.96 \cdot 10^{-2}$ | |

Dependences of Al_2O_3 and TiO_2 sputtered atoms quantity on samples thickness are resulted in Figs. 9, 10.

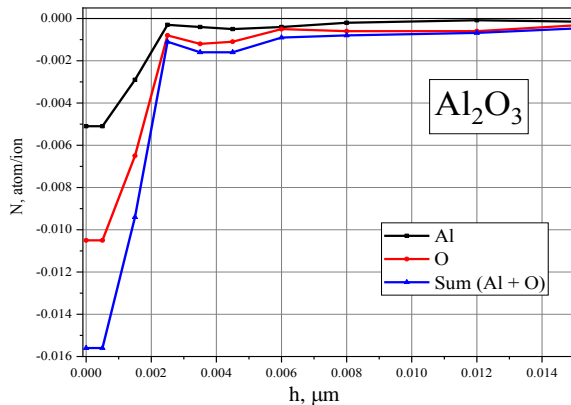


Fig. 9. Dependence of Al_2O_3 sputtered atoms quantity on sample thickness

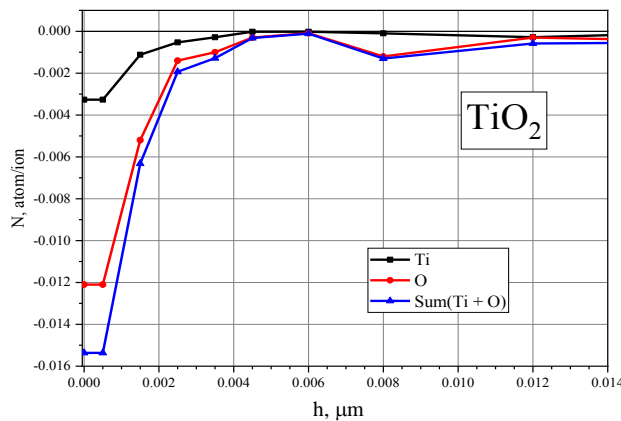


Fig. 10. Dependences of TiO_2 sputtered atoms quantity on sample thickness

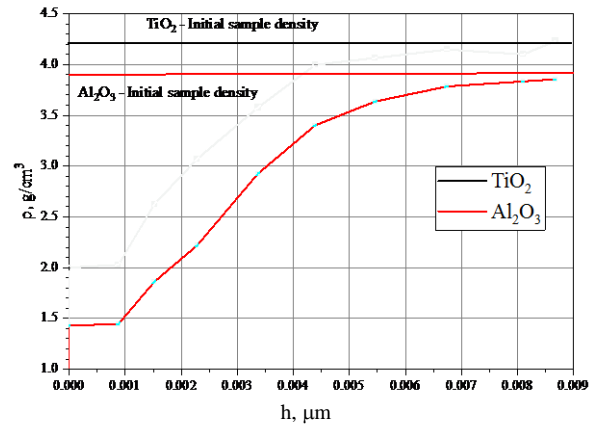


Fig. 11. Dependence of Al_2O_3 and TiO_2 "superficial" density from a thickness (zero is samples surface)

From these dependences follows that sputtering for Al_2O_3 and TiO_2 should be considered on distance no more than 0.006...0.008 μm from samples surface at irradiation energy of 0.12 MeV. In consideration of Table 5 and Figs. 9, 10 data, dependences of "superficial" densities change of Al_2O_3 and TiO_2 irradiated samples are resulted in Fig. 11. Change of "superficial" density should influence on friction factors of the metal – Al_2O_3 and TiO_2 pairs.

4. EXPERIMENTAL RESULTS OF THE FRICTIONAL INTERACTION

After preparation of Al_2O_3 and TiO_2 samples surfaces their tests were conducted for facility on studying tribological characteristics (see Fig. 4) at temperatures 20 and 200 °C and loadings of 0.5...2 kg from 100 to 180 cycles. Dependence of a sliding friction factor on time at one test cycle is resulted in Fig. 12. Dependence of a sliding friction factor on complete experiment time is resulted in Fig. 13.

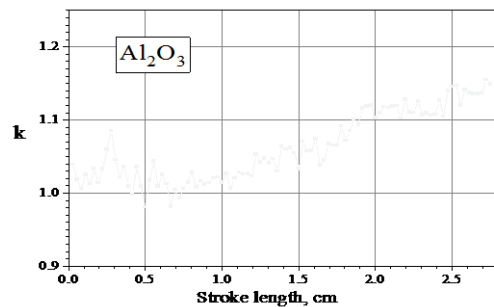


Fig. 12. Dependence of a sliding friction factor on a stroke length of interacting samples (one cycle)

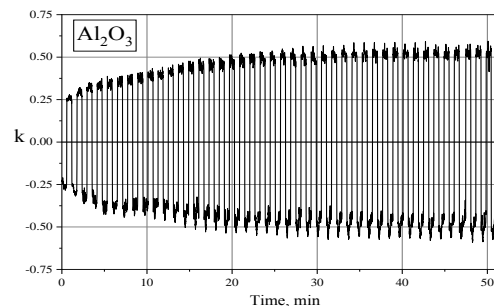


Fig. 13. Dependence of a sliding friction factor on experiment time

It's necessary to notice that the rest friction factor in 2.7–2.9 times more, than sliding friction factor at all tests.

Dependences of sliding friction factors on cycle's quantity at temperatures 20 and 200 °C of irradiated and not irradiated Al₂O₃ and TiO₂ samples interacting with a steel are resulted in Figs. 14, 15.

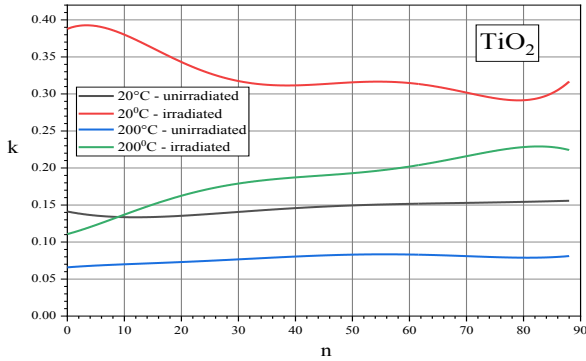


Fig. 14. Dependence of a sliding friction factor on interaction cycle's quantity of a steel – TiO₂ pair

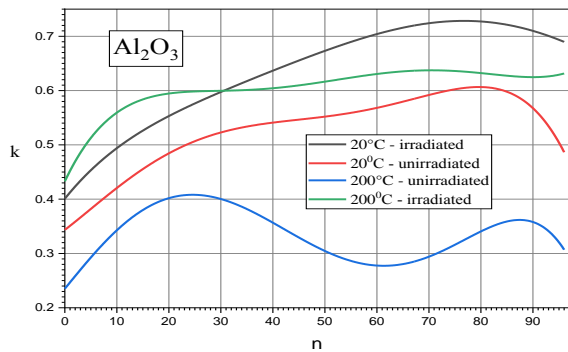


Fig. 15. Dependence of a sliding friction factor on interaction cycle's quantity of a steel – Al₂O₃ pair

From the resulted graphs follows that at temperature 200 °C a sliding friction factor are less, than at 20 °C. The irradiation leads to increase of a friction factor.

As experiment at a non-uniform temperature change mode of a metal – TiO₂ frictional pair has been made. Dependences of a temperature and sliding friction factor change from cycle's quantity of frictional interaction are shown in Fig. 16.

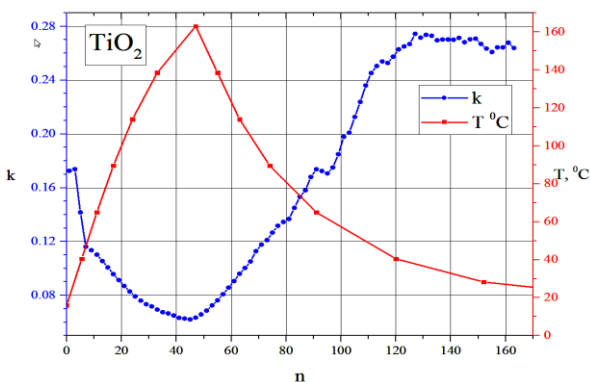


Fig. 16. Dependences of a sliding friction factor and temperature from cycle's quantity

A temperature in the given experiment (stage 1) raised for ~ 50 cycles to 160 °C after that there was a temperature fall (stage 2) for ~ 120 cycles from 160 to 25 °C. Thus the sliding friction factor at the first stage decreased from 0.17 to 0.06, at the second stage was observed growth to 0.27 and at 130 cycles approach on a stationary mode. From the analysis of experimental data (see Figs. 11–15) follows that defining role at non-stationary thermal loading of pair is played by temperature change. The role of cycles in the given temperature range is secondary.

5. MICROSCOPIC INVESTIGATIONS AND RESULTS DISCUSSION

A MMU-3 microscope and 5 and 18 megapixel ZCAT chambers are used for microscopic studies. Surfaces of interacting samples influence on tribological characteristics. After grinding and polishing Al₂O₃ and TiO₂ microstructural photos of surfaces have been made (see Fig. 2). The same photos have been do and after irradiation of these samples. Photo of TiO₂ surface after irradiation is resulted in Fig. 17.

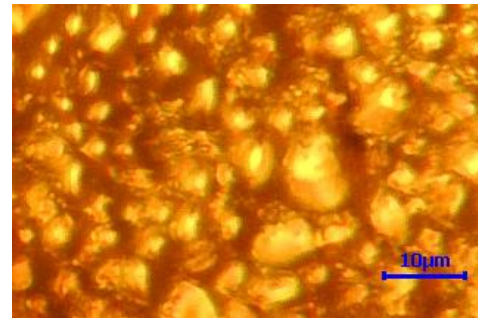


Fig. 17. TiO₂ irradiated sample

A surface linear profile of irradiated sample TiO₂ in red, green and dark blue spectra and as on brightness is shown in Fig. 18.

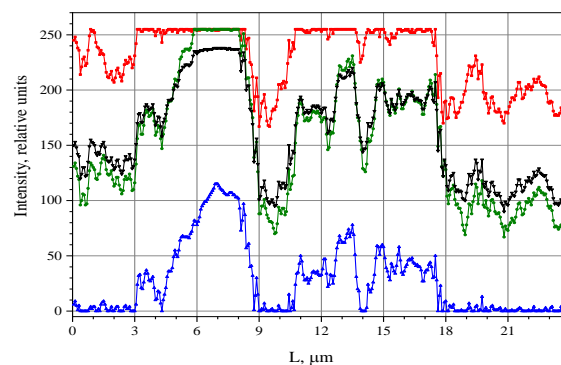


Fig. 18. Surface linear profile of irradiated sample TiO₂ in red, green and dark blue spectra and on brightness (black curve)

From Figs. 17, 18 follows that the irradiation leads to increase of a surface roughness i. e. to reduction of “superficial” density. It confirms the carried out calculations, (see Fig. 11). Formation blistering and flaking is not observed, obviously oxygen and helium diffusion to samples surface take place. In the process of Al₂O₃ and TiO₂ samples irradiation the effect of superficial metallization is observed (Fig. 19).

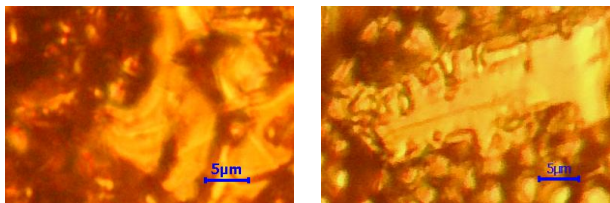


Fig. 19. Metallization of Al_2O_3 (left) and TiO_2 (right) irradiated samples

This process takes place in connection with Al_2O_3 and TiO_2 dissociation. After dissociation oxygen diffusion occurs, basically on grains borders with partial restoration of structure atoms. There is a mutual carrying over of metals and their connections at frictional interaction of metal to ceramics. These processes increase ceramics wear resistance and increase friction factors at the expense of an interaction of more viscous metal connections.

The swelling of $\sim 1\text{--}2\%$ in irradiated Al_2O_3 and TiO_2 samples is observed. TiO_2 areas of not irradiated and irradiated parts are resulted in Fig. 20.

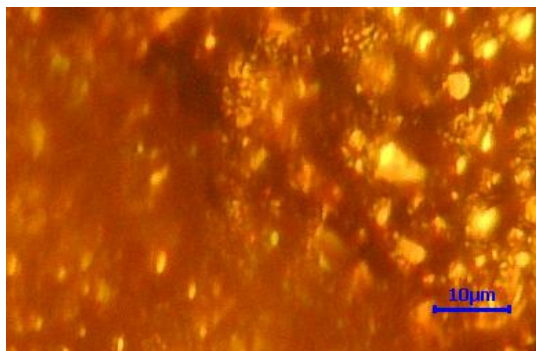


Fig. 20. TiO_2 areas of not irradiated (left) and irradiated (right) sample

TiO_2 surface linear profile from not irradiated part in irradiated part in red, green and dark blue spectra and on brightness (black curve) is resulted in Fig. 21.

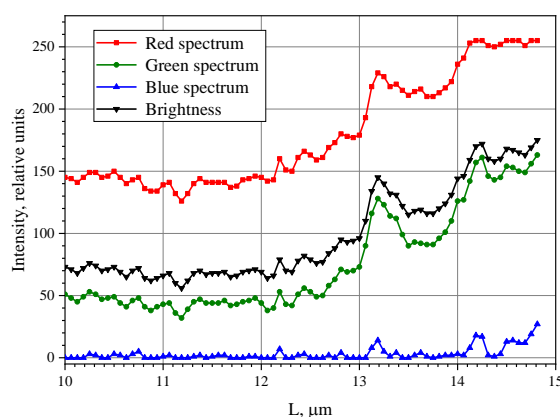


Fig. 21. TiO_2 surface profile from not irradiated part (left) in irradiated part (right) in red, green and dark blue spectra and on brightness (black curve)

CONCLUSIONS

From the received experimental results and calculating data follows those on friction factors of a metal- Al_2O_3 and TiO_2 pair an irradiation influences owing to formation on samples surfaces metallization in

connection with Al_2O_3 and TiO_2 dissociation. At the pair frictional interaction there is a mutual carrying over of metals and their connections. It leads to increase of friction factors of metal with the irradiated samples.

During microscopic investigations it was not revealed a blistering and flaking on irradiated ceramics surface. Obviously that there is oxygen and helium diffusion on grains borders.

Process of atoms sputtering at samples irradiation materials “superficial” density reduces that influences on tribological characteristics.

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ТРИБОЛОГИЧЕСКИЕ ХАРАКТЕРИСТИКИ ПАРЫ СТАЛЬ – КЕРАМИКА, КОТОРАЯ ОБЛУЧЕНА ИОНАМИ ГЕЛИЯ

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Приведены установка для изучения трибологических характеристик пары металл – керамика и параметры облучения керамических образцов на линейном ускорителе ионов гелия с энергиями 0,12 и 4 МэВ. Для облученных TiO_2 и Al_2O_3 рассчитаны профили повреждаемости и залегания атомов мишени вдоль пробега ионов гелия. Получены коэффициенты распыления и зависимости количества распыленных атомов по толщине образцов. Произведены расчеты по изменению плотности облученных образцов. Представлены экспериментальные результаты измерения коэффициента трения скольжения в зависимости от количества циклов, температуры и дозы облучения. На основе микроскопических исследований и расчетных данных сделаны выводы о влиянии облучения на трибологические характеристики взаимодействующих пар.

ТРИБОЛОГІЧНІ ХАРАКТЕРИСТИКИ ПАРИ СТАЛЬ – КЕРАМІКА, ЯКА ОПРОМІНЕНА ІОНАМИ ГЕЛІЮ

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Наведено установку для вивчення трибологічних характеристик пари метал – кераміка та параметри опромінення керамічних зразків на лінійному прискорювачі іонів гелію з енергіями 0,12 і 4 МеВ. Для опромінених TiO_2 й Al_2O_3 розраховано профілі пошкоджуваності та залягання атомів мішені вздовж пробігу іонів гелію. Отримано коефіцієнти розпилення й залежності кількості розпилених атомів по товщині зразків. Зроблено розрахунки щодо змінення щільності опромінених зразків. Подано експериментальні результати вимірювання коефіцієнта тертя ковзання залежно від кількості циклів, температури й дози опромінення. На основі мікроскопічних досліджень і розрахункових даних зроблено висновки щодо впливу опромінення на трибологічні характеристики пар, що взаємодіють.