https://doi.org/10.46813/2022-139-042

# FACILITY AND RESEARCHES RESULTS OF THE FRICTIONAL CHARACTERISTICS OF METAL – IRRADIATED-CERAMICS PAIR

*V.I. Butenko, S.N. Dubniuk, A.F. Dyachenko, O.V. Manuilenko, K.V. Pavlii, B.V. Zajtsev*
*National Science Center "Kharkov Institute of Physics and Technology", Kharkiv, Ukraine*
*E-mail: kvint@kipt.kharkov.ua*

The technique and facility for studying of frictional characteristics of metal – ceramics pair are presented. Parameters of samples irradiation on the helium ions linear accelerator with 0.12 and 4 MeV energies are resulted. Samples from $TiO_2$ and $Al_2O_3$ at 0.12 MeV beam energy are irradiated. Damageability and occurrence profiles of target atoms along of helium ions range as sputtering ratios are calculated for the irradiated materials. Dependences of the sputtered atoms quantity on samples depth are resulted. Using this data, calculations on change of samples "superficial" density along of ions range are made. Experimental results of a sliding friction factor measurement depending on cycle's quantity, temperature and irradiation dose are presented. On the basis of microscopic researches conclusions on influence of an irradiation on frictional characteristics of a metal – ceramics pair are drawn.

PACS: 29.17.w, 29.27.Bd

## INTRODUCTION

Development of nuclear and thermonuclear power demands creation of the new wear proof 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 hard loaded friction units of nuclear power facilities are the transformational hardened ceramic materials [2, 3] which often work together with metal. Processes of carrying over of metal and its oxides on a ceramics surface take place in a metal – ceramics pair, therefore the so-called transfer layer is formed which carries out protective function, increasing ceramics wear resistance [4].

In atomic power engineering the friction forces are shown at all stages of the fuel assembly (FA) life cycle [5-7]. Friction forces are considered at a justification of thermo-mechanical durability FA, seismic resistance of a protection control system (PCS), vibration strengths FA and etc. [8, 9]. The insufficient attention to them led to fretting-damages of FA covers, to increase in time of falling and sticking of PCS cores, excessive efforts at assemblage FA and loading-unloading FA from the reactor [9, 10]. The basic mechanism responsible for FA destruction at capacity change is mechanical influence of extending fuel on a shell. Such influence is caused by various temperature of the core and a shell and several bigger (in 1.5-2 times) thermal expansions of the fuel ceramic tablets in comparison with a metal shell. 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. There are some more processes which take place at materials irradiation. It is action electrons, photons, neutral atoms or molecules, surface waves, magnetic and electric field and etc., influencing on interacting pairs frictional characteristics.

The work purpose is technique working out, manufacturing of a facility and research of calculated and experimental frictional characteristics of metal – irradiated-ceramics pair.

## FACILITY FOR MEASUREMENT OF METAL – CERAMICS PAIR FRICTIONAL CHARACTERISTICS

Considering specificity of an irradiation (the maximum radiating damage can be reached, irradiating flat samples) and researches (uniform relative movement of samples pair) necessary parameters of a frictional interaction the scheme presented in Fig. 1 has been chosen. Samples from $TiO_2$ and $Al_2O_3$ which were irradiated on the linear accelerator of helium ions are shown in Fig. 2 (samples diameter is 15 mm). The metal sample has been made from alloy steels; its sizes are 50×23×14 mm. This steel is used for production calibrating elements and standards with thermal expansion factor equal to zero.

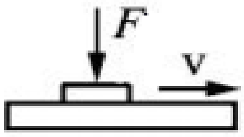


*Fig. 1. Scheme of samples contacting in facility*

*Fig. 2. $TiO_2$ (left), $Al_2O_3$ (right) samples*

Facility for frictional characteristics studying of a metal – ceramics pair is shown in Fig. 3.

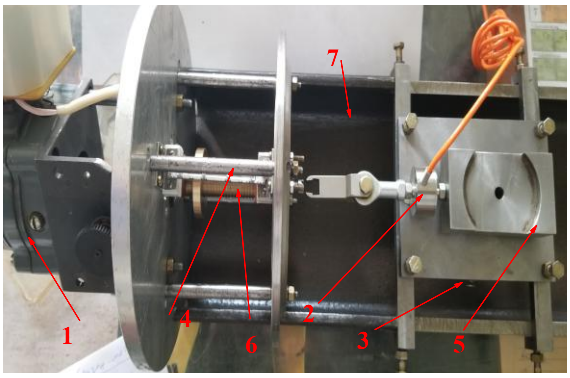


*Fig. 3. Facility for tribological characteristics studying, where 1 – reversive asynchronous electric motor RD-09; 2 – pressure (force) tensometric gauge DYMH-103; 3 – infra-red heating element; 4 – resistor with two microswitches; 5 – holder of the ceramic sample and loading weights; 6 – transformation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

The reversive asynchronous electric motor RD-09 (see Fig. 3, pos. 1) with built reducer, nominal useful capacity of 1 W and rotation frequency of a reducer output shaft to 8.7 rpm is applied for relative moving of interacting samples.

The pressure (force) tensometric gauge (see Fig. 3, pos. 2) is used for definition of a friction force of interacting samples. Its technical characteristics are resulted in Table 1. Overall dimensions and gauge general view are presented in Fig. 4. The gauge spent graduation has linear character.

***Table 1***

*Pressure tensometric 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 | ± 0.5% |
| Working temperature | (–35°C)…(+85°C) |

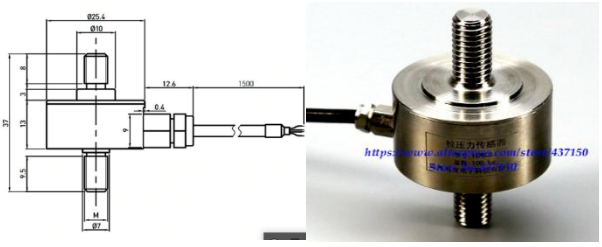

*Fig. 4. Pressure (force) tensometric gauge*

The infra-red heating elements (260 W and 450 W) are used for heating of samples (see Fig. 3, pos. 3). Their view is shown in Fig. 5. The heater in 260 W allows to carry out heating of a metal – ceramics pair to 400°C, and a heater in 450 W – to 900°C. This or that heating element is used depending on an assigned task on a temperature range.

*Fig. 5. Appearance of heating elements. 260 W (60×60 mm) – left, 450 W (80×80 mm) – right*

A resistor with two microswitches (see Fig. 3, 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.

The holder is made (see Fig. 3, 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. 6. This facility provides static loading on a frictional pair. For dynamic loading maintenance it’s necessary to create hydraulic, pneumonic or electromechanical loading unit.

The transformation mechanism of a rotary motion in forward (see Fig. 3, pos. 6) has a step of 1 mm. This allows regulating in a wide interval moving speed of interacting samples. Fastening of the facility all elements it’s carried out on a base (see Fig. 3, pos. 7).

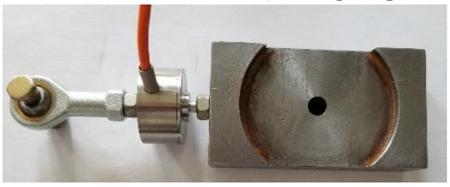

*Fig. 6. Samples holder with pressure (force) tensometric gauge*

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 minutes. During carrying out of measurements it’s possible to receive dependence of sliding and rest friction factors on time, cycle’s quantities and temperature, and as all listed characteristics from of radiating damages doses.

## IRRADIATION PARAMETERS OF SAMPLES ON THE HELIUM IONS ACCELERATOR

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 [11, 12].

The chamber and system of experimental parameters measurement is created for an irradiation and studying of constructional materials characteristics on the accelerator [12, 13]. The vacuum in the chamber is carried out with the help forevacuum and turbo-molecular pumps. It provides oxygen-free environment in volume of the chamber and the same vacuum, as in accelerating structure.

The temperature of irradiated samples is set by the heating element located directly in the irradiation chamber and it’s measured by the thermocouple attached to the sample. The focusing triplet is established in front of the irradiation chamber for increase of current density of a beam, falling on the sample, and reduction of an irradiation time. It allows changing beam radius, and, hence, and current density, depending on experiment requirements [14].

Beam currents are measured by contactless flying gauges which are established on input and output of triplet, and as before the irradiated sample. The digital oscillograph ZET-302 and DAC/ADC ZET-210 which are connected to the computer with the further data recording and their processing [13] were used for registration of irradiation parameters. The basic beam parameters at an irradiation of samples on the helium ions linear accelerator are resulted in Table 2.

***Table 2***

*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 grinding and polishing of samples $Al_2O_3$ and $TiO_2$ have been irradiated on the accelerator of helium ions to doses $10^{18}$ ion/cm$^2$ at 0.12 MeV beam energy for researches of frictional characteristics of a metal – ceramics pair. This energy has been chosen for the purpose of the maximum damageability of ceramic samples surface.

## CALCULATED CHARACTERISTICS OF THE IRRADIATED SAMPLES

For calculation of ions range in solids software package SRIM [15, 16] was used which along with possibility of range calculation allows to receive following information about: vacancies distribution in a target; redistribution of irradiated materials atoms; sputtering ratios; phenomena connected with ions energy loss; distribution of ionization and phonons formation. Before an irradiation of samples $TiO_2$ and $Al_2O_3$ in program SRIM all listed processes, taking into account displacement cascades have been calculated. 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 ceramic materials, irradiated at E = 0.12 MeV*

| Element | Energy loss, % | | |
|---|---|---|---|
| | Ionization | Phonons | Damageability |
| | He$^+$/cascade | He$^+$/cascade | He$^+$/cascade |
| $Al_2O_3$ | 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 |

From Table 3 it’s visible that the greater part of energy goes on ionization at the expense of helium ions beam. 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 $TiO_2$ and $Al_2O_3$ make 0.6…0.7 microns.

Profiles of damageability and occurrence helium and $TiO_2$ atoms, irradiated on the accelerator with energy of 0.12 MeV are resulted in relative units in Fig. 7. Also such dependences for $Al_2O_3$ are received.

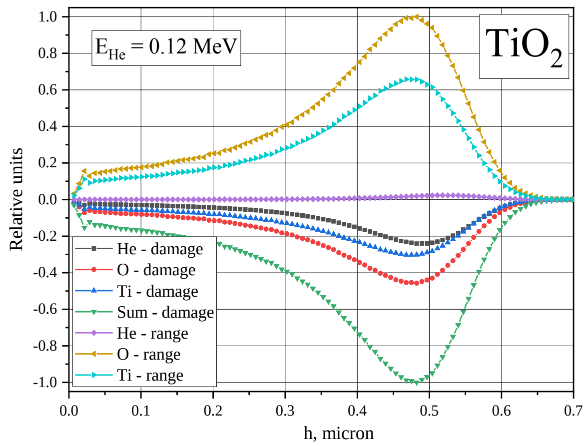


*Fig. 7. Damageability (bottom curves) and occurrence profiles helium, oxygen and titanium (top curves) in $TiO_2$*

From graphs follows that in irradiated samples there is a redistribution of materials atoms along of helium ions range. The material atoms concentration is more than introduced helium. The basic contribution to damageability is brought by displacement cascades (irradiated material atoms) formed at an irradiation by helium ions in dissociation process. Therefore these processes lead to change of the irradiated material density along of helium ions range.

Relations of $Al_2O_3$ and $TiO_2$ materials atoms to He, formed at helium ions irradiation, and the general damageability are resulted in Table 4.

***Table 4***

*Relation of $Al_2O_3$ and $TiO_2$ atoms to He$^+$, formed at helium ions irradiation, and damageability*

| $Al_2O_3$ | | $TiO_2$ | |
|---|---|---|---|
| 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 | |

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 ratio and sputtering average energy for $Al_2O_3$ and $TiO_2$ are presented in Table 5.

***Table 5***

*Sputtered atoms quantity and sputtering average energy for $Al_2O_3$ and $TiO_2$*

| **Beam energy, $E_{He}$ = 0.12 MeV** | **Sputtered atoms quantity, N $10^{-5}$ atom/ion** | | **Sputtering average energy, eV/atom** | |
|---|---|---|---|---|
| $Al_2O_3$ | Al | O | Al | O |
| | 1470 | 2140 | 168 | 215 |
| Sputtering ratio | $\approx 3.61\cdot10^{-2}$ | | | |
| $TiO_2$ | Ti | O | Ti | O |
| | 630 | 2330 | 256 | 137 |
| Sputtering ratio | $\approx 2.96\cdot10^{-2}$ | | | |

Dependences of $Al_2O_3$ sputtered atoms quantity on a thickness are resulted in Fig. 8 (zero is a sample surface). The similar graph also for $TiO_2$ is received.

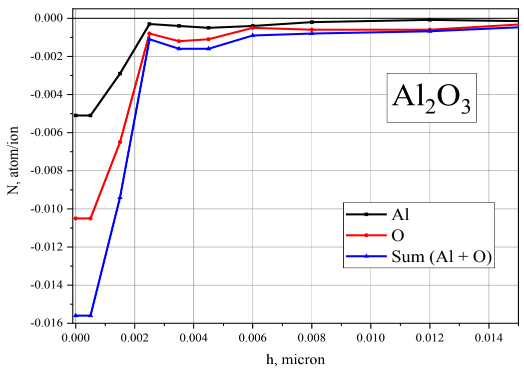


*Fig. 8. Dependence of $Al_2O_3$ sputtered atoms quantity on sample thickness*

From these dependences follows that at density calculation it’s should be considered sputtering of atoms irradiated materials. Sputtering for $Al_2O_3$ and $TiO_2$ it’s necessary to consider on distance 0.006…0.008 microns from samples surface at irradiation energy of 0.12 MeV.

The "superficial" density influences on friction factors of metal – ceramics pairs. In consideration of Table 5 data and sputtered atoms profiles, dependences of "superficial" densities change of $Al_2O_3$ and $TiO_2$ irradiated samples are resulted in Fig. 9.

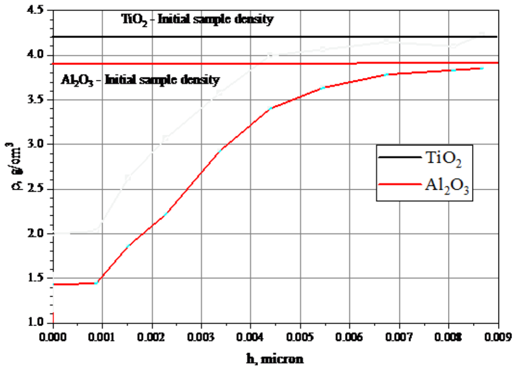


*Fig. 9. Dependence of $Al_2O_3$ and $TiO_2$ "superficial" density on a thickness (zero is samples surface)*

## EXPERIMENTAL RESULTS OF THE FRICTIONAL INTERACTION

After grinding and polishing of $Al_2O_3$ and $TiO_2$ samples their tests were conducted for facility (see Fig. 3) on studying frictional characteristics of steel – ceramics materials at temperatures 20 and 200°C and loadings of 0.5…2 kg. Dependence of a sliding friction factor on experiment time is resulted in Fig. 10. In total about 120 cycles were spent. Dependence of a sliding friction factor on time at one test cycle is resulted in Fig. 11.

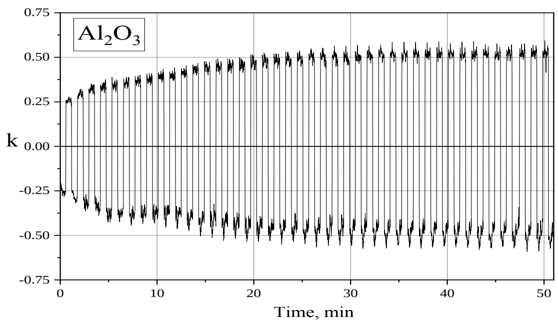


*Fig. 10. Dependence of a sliding friction factor on experiment time*

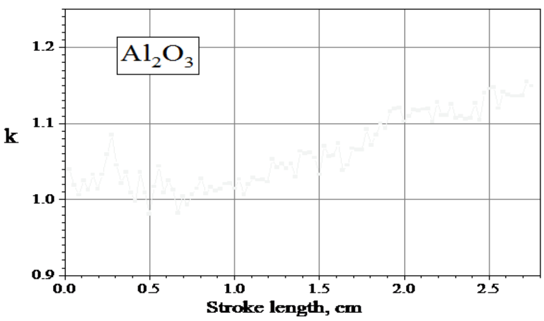


*Fig. 11. Dependence of a sliding friction factor on a stroke length of interacting samples (one cycle)*

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 $Al_2O_3$ and $TiO_2$ samples interacting with steel are resulted in Figs. 12, 13.

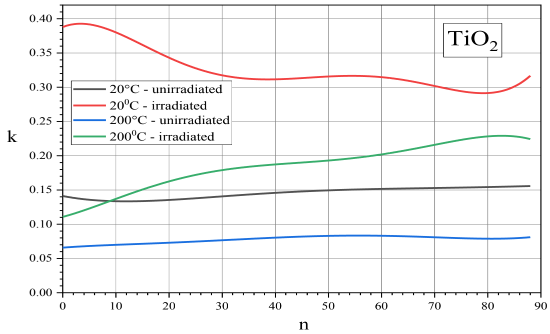


*Fig. 12. Dependence of a sliding friction factor on interaction cycle's quantity of steel – $TiO_2$ pair*

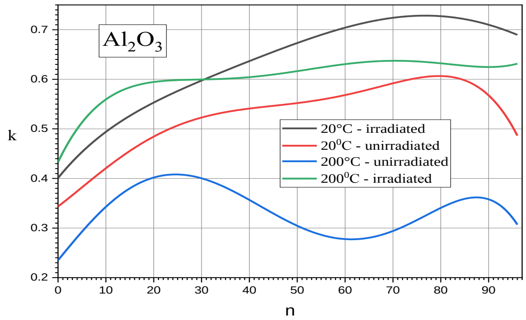


*Fig. 13. Dependence of a sliding friction factor on interaction cycle's quantity of steel – $Al_2O_3$ pair*

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 metal – $TiO_2$ 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. 14.

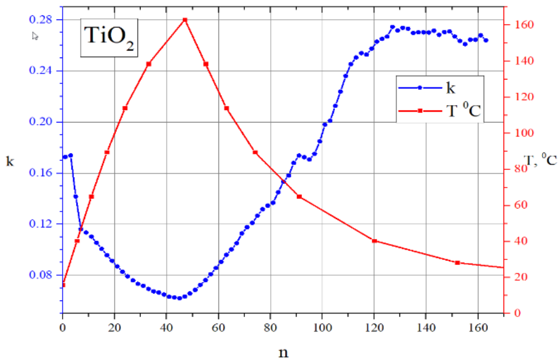


*Fig. 14. Dependences of a sliding friction factor and temperature from cycle's quantity*

Properly from Fig. 14, at temperature growth a sliding friction factor decreases, and at reduction it increases. It coincides with conclusions by tests results at stationary temperature.

## MICROSCOPIC INVESTIGATIONS AND RESULTS DISCUSSION

A MMU-3 microscope and 5 and 18 megapixel ZZCAT chambers are used for microscopic studies. Surfaces of interacting samples influence on frictional characteristics. Surfaces photos of not irradiated samples are resulted in Fig. 15.

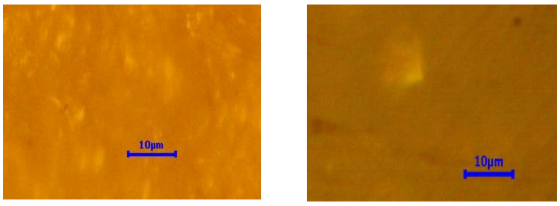


*Fig. 15. $Al_2O_3$ (left) and $TiO_2$ (right) initial surfaces*

The same photos have been made after an irradiation of $Al_2O_3$ and $TiO_2$ samples and their 3d profiles are resulted in Fig. 16.

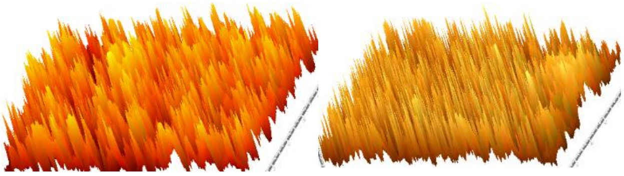

*Fig. 16. 3d profile of $Al_2O_3$ (left) and $TiO_2$ (right) irradiated samples*

A surface linear profile of irradiated sample $TiO_2$ in red, green and dark blue spectra and as on brightness is shown in Fig. 17.

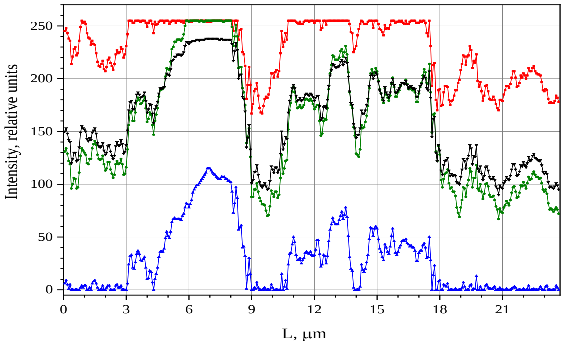


*Fig. 17. Surface profile of $TiO_2$ irradiated sample*

Apparently from these figures the surface roughness increases after an irradiation. It leads to increase of metal with irradiated-ceramics friction factors. The effect of superficial metallization is observed in the process of $Al_2O_3$ and $TiO_2$ samples irradiation, Fig. 18.

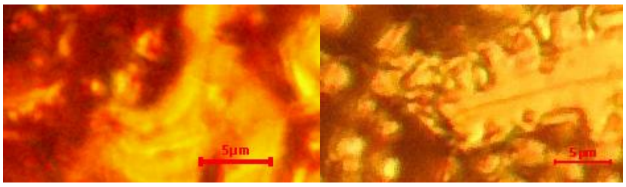


*Fig. 18. Metallization of $Al_2O_3$ (left) and $TiO_2$ (right) irradiated samples*

This process takes place in connection with $Al_2O_3$ and $TiO_2$ dissociation. Also there is oxygen and helium diffusion, 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, on the one hand, increase ceramics wear resistance and with another, increase friction factors at the expense of an interaction of more viscous metal connections.

Ceramics swelling influences on frictional interaction characteristics. $TiO_2$ areas of not irradiated and irradiated parts are resulted in Fig. 19.

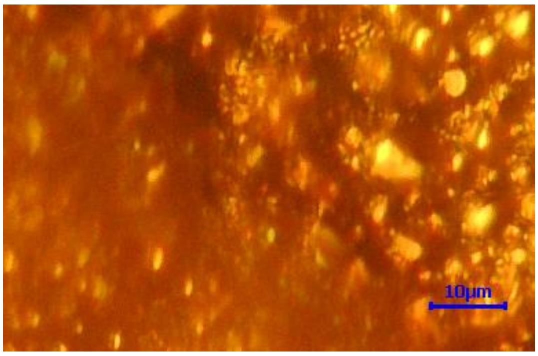


*Fig. 19. $TiO_2$ areas of not irradiated (left) and irradiated (right) sample*

The initial size of $TiO_2$ grain makes 1…3 microns, after an irradiation the grain size makes 4…6 microns. The surface profile of $TiO_2$ from not irradiated part in the irradiated part of the sample is resulted in Fig. 20.

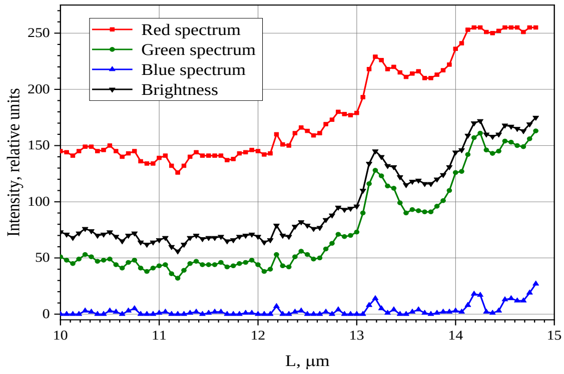


*Fig. 20. $TiO_2$ surface profile from not irradiated part (left) in irradiated part (right)*

## CONCLUSIONS

From the received experimental results and calculating data follows those on friction factors of a metal – ceramics pair an irradiation influences owing to formation on ceramic 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.

Blistering and flaking on irradiated-ceramics surface was not revealed during microscopic investigations. Obviously, there is oxygen and helium diffusion on grains borders.

Atoms sputtering process of irradiated-ceramics reduces materials "superficial" density. $Al_2O_3$ and $TiO_2$ grains growth and swelling is observed. These processes also influence on the frictional interaction characteristics of metal – ceramics pairs.

## ACKNOWLEDGEMENTS

Work was partially supported by the Ukrainian budget program “Support for the most important directions of scientific researches” (КПКВК 6541230), project “Development of linear ion accelerators based on alternating-phase and high-frequency quadrupole focusing for energy and radiation technologies”.

## REFERENCES

1. I.M. Fedorchenko. Antifriction and friction cermet materials // *Powder Metallurgy and Metal Ceramics*. 2002, v. 41, № 9-10, p. 489-497.
2. L. Nettleship, R. Stevens. Tetragonal zirconia polycrystal (TZP) – a review // *Int. J. High Technology Ceramics*. 1987, № 3, p. 1-32.
3. S.W. Lee, S.H. Hsu, M.C. Shen. Ceramic Wear Maps: Zirconia // *J. Amer. Ceram. Soc.* 1993, v. 76, № 8, p. 1937-1947.
4. P.C. Becker, T.A. Libsch, S.K. Rhee. Wear mechanisms of toughening zirconias // *Ceram. Eng. and Sci. Proceed*. 1985, № 7-8, p. 1040-1058.
5. A.A. Enin, A.P. Ustimenko. *Efforts comparative analysis of fuel elements interaction with a fuel rigid skeleton of new generation WCPR.* Gelendzhik, 2010.
6. A.P. Ustimenko, M.A. Shustov. Mechanical characteristics fuel assemblies of the power reactors, defined in experiments on units and small models // *2nd Russian conference "Methods and calculations software on durability".* Gelendzhik. 2002, p. 102-118.
7. I.A. Guschik, V.V. Makarov, A.V. Seleznev, S.N. Komarova. Calculated experimental research of flexible cores passability in tubular channels depending on channels deformation // *Problems of a resource and safety of the power equipment*. Moskow. 1999.
8. Yu.N. Drozdov, V.V. Makarov, A.V. Apfanas'ev, et al. Frictional interaction dynamics of a fuel element cover and distance grating cell of a fuel assembly // *Engineering Bulletin.* 2007, № 3.
9. V.V. Makarov, A.V. Apfanas'ev, I.V. Matvienko, et al. Experimental research of a friction process in the individual contact formed by a fuel element cover and distance grating cells, MNTK-5, Podolsk, 2007.
10. M.V. Puchkov, V.V. Makarov, A.V. Apfanas'ev, et al. Samples tests of fuel elements covers and distance grating cells on a friction with a variation of constructional, technological and operational factors // *Problems of Atomic Science and Technology* (Sarov). 2010, № 27.
11. V.O. Bomko, O.F. Dyachenko, Ye.V. Ivakhno, et al. New prestripping section of the MILAC linear accelerator designed for accelerating a high current beam of light ions // *Proceedings of EPAC 2006 Edinburgh, Scotland.* 2006, p. 1627-1629.
12. S.N. Dubniuk, B.V. Zajtsev. The linear accelerator for radiation structural materials // *Problems of Atomic Science and Technology*. *Series "Nuclear Physics Investigations".* 2014, № 3, p. 172-176.
13. R.A. Anokhin, B.V. Zaitsev, K.V. Pavlii, et al. Experimental complex for investigation of construction materials on the helium ions linear accelerator// *Problems of Atomic Science and Technology*. *Series "Nuclear Physics Investigations".* 2017, № 6, p. 167-171.
14. A.F. Dyachenko, S.N. Dubniuk, A.P. Kobets, et al. The bunch formation and transport system to the target of the helium ions linac // *Problems of Atomic Science and Technology. Series "Plasma Electronics and New Methods of Acceleration".* 2018, № 4, p. 52-55.
15. http://www.srim.org.
16. B. Widrow, J.R. Glover, J.M. McCool, J. Kaunits. Adaptive noise cancelling: principles and applications // *Proc. of the IEEE*. 1976, v. 63, p. 1692-1716.


*Article received 22.11.2021*

## УСТАНОВКА ТА РЕЗУЛЬТАТИ ДОСЛІДЖЕНЬ ФРИКЦІЙНИХ ХАРАКТЕРИСТИК ПАРИ МЕТАЛ – ОПРОМІНЕНА КЕРАМІКА

***В.І. Бутенко, С.М. Дубнюк, О.Ф. Дьяченко, О.В. Мануйленко, К.В. Павлій, Б.В. Зайцев***

Представлено методику та установку для вивчення фрикційних характеристик пари метал – кераміка. Наведено параметри опромінення зразків на лінійному прискорювачі іонів гелію з енергіями 0,12 і 4 МеВ та опромінені зразки з $TiO_2$ і $Al_2O_3$ при енергії пучка 0,12 МеВ. Для опромінених матеріалів розраховано профілі пошкоджуваності й залягання атомів мішені вздовж пробігу іонів гелію, а також коефіцієнти розпилення. Наведено залежності кількості розпилених атомів від глибини зразків. Використовуючи ці дані, зроблено розрахунки щодо змінення «поверхневої» щільності зразків уздовж пробігу іонів. Представлено експериментальні результати вимірювання коефіцієнта тертя ковзання залежно від кількості циклів, температури й дози опромінення. На основі мікроскопічних досліджень зроблено висновки щодо впливу опромінення на фрикційні характеристики пари метал – кераміка.

## УСТАНОВКА И РЕЗУЛЬТАТЫ ИССЛЕДОВАНИЙ ФРИКЦИОННЫХ ХАРАКТЕРИСТИК ПАРЫ МЕТАЛЛ – ОБЛУЧЕННАЯ КЕРАМИКА

***В.И. Бутенко, С.Н. Дубнюк, А.Ф. Дьяченко, О.В. Мануйленко, К.В. Павлий, Б.В. Зайцев***

Представлены методика и установка для изучения фрикционных характеристик пары металл – керамика. Приведены параметры облучения образцов на линейном ускорителе ионов гелия с энергиями 0,12 и 4 МэВ и облучены образцы из $TiO_2$ и $Al_2O_3$ при энергии пучка 0,12 МэВ. Для облученных материалов рассчитаны профили повреждаемости и залегания атомов мишени вдоль пробега ионов гелия, а также коэффициенты распыления. Приведены зависимости количества распыленных атомов от глубины образцов. Используя эти данные, произведены расчеты по изменению «поверхностной» плотности образцов вдоль пробега ионов. Представлены экспериментальные результаты измерения коэффициента трения скольжения в зависимости от количества циклов, температуры и дозы облучения. На основе микроскопических исследований сделаны выводы о влиянии облучения на фрикционные характеристики пары металл – керамика.