ALDABERGENOVA TAMARA MUSTAFAEVNA

Study of structure degradation and physical-mechanical properties of graphite and tungsten surface under irradiation with plasma streams and charged particles

ABSTRACT

of the dissertation thesis for the degree of Doctor of Philosophy (PhD)
specialty «6D071000 - materials science and technology of new materials»

General description of paper.

The dissertation paper provides the results of the experimental studies of the influence of pulsed energy release and ion irradiation on the surface structure and the physical-mechanical properties of high-purity tungsten of double hot forging and the reactor graphite.

Relevance of research.

Controlled thermonuclear reaction is one of the promising ways to solve the energy problems of the world community. The best plasma parameters were obtained at the research thermonuclear plants with toroidal plasma confinement, i.e. Tokamak plants. Therefore, at present, the main efforts of nuclear physicists and materials scientists are focused on further development and improvement of these facilities, used as the basis for development of the power TNR projects. The materials science issues play a significant role among the issues of TNR establishment, which include cooling of the first wall, the systems of plasma-cord containment with superconducting magnets, build-up of tritium, and others. These are the tasks of protecting the first wall and materials of divertor devices, as one of the most energy-stressed TNR units from the energy fluxes and charged particles. The issues of creation and selection of structural materials for TNR are the integral part of the issue on radiation resistance of materials for any nuclear facilities, but at the same time have their own characteristics. Thus, in the TNR, the materials are exposed not only to neutron irradiation and contact with the coolant, but also to the synergistic effect of neutron and ion irradiation, which significantly increases the radiation effects, as well as the release of energy on the chamber surface during plasma breakdown. One of the solutions, implemented at the Tokamak facilities, is cladding of the chamber, i.e. the first wall of the TNR, the special protection materials, taking the main thermal loads and protecting the first wall from these impacts. The role of these materials is to maintain operability of the first wall for a long period of operation and, ideally, during the entire “operation period” of the TNR.

Even more strict requirements are imposed on the divertor plates. The main thermal and radiation loads are applied on the divertor plates, since their task is to “clean up” the plasma from the products of the thermonuclear reaction. Therefore, the divertor plates are affected not only by the energy flows during abnormal situations such as plasma breakdowns, but also by the energy flows from charged and neutral

particles during normal operation of the Tokamak facility. Selection of the material for divertor plates is particularly acute today. To date, the series of requirements have been stipulated for protection materials of the first wall and the divertor devices. The main requirements are as follows: low sputtering coefficient of the surface, high thermal conductivity coefficient, low chemical activity, high melting point, high strength at high temperatures, high ductility and crack resistance, high surface radiation resistance to the erosion processes (sputtering and peeling).

Several types of materials, molybdenum, graphite, liquid crystal materials were considered as the materials for divertor fabrication, but tungsten is currently considered to be the highest priority material. Tungsten is deemed as a promising material for the TNR diverter plates due to its properties such as high melting point and thermal conductivity, low surface sputtering coefficient and other characteristics. Tungsten grades differ by the fabrication method, availability of impurities and alloying additives; as a result, they have different physical and mechanical properties. In particular, highly pure tungsten of vacuum melting meets the necessary requirements and is considered as a cladding material for the ITER reactor divertor. Based on these requirements, the dissertation thesis is focused on the studies of highly pure tungsten, as the most promising material of the TNR diverter.

Aim of the dissertation thesis is a comprehensive study of the impact of the pulsed energy flows and ion irradiation on the structure and physical-mechanical properties of high-purity DFW grade tungsten - the promising material for protecting of the divertor plates and the first wall of fusion reactors with toroidal plasma confinement. To achieve this goal, the following tasks were specified.

Main tasks of research:

- adaptation and development of the methods for study of the effects of surface sputtering and degradation of tungsten properties during ion irradiation.
- study of the sputtering effects of DFW tungsten and graphite under ion irradiation.
- study of the effects of surface erosion (measurement of the mass loss during local melting) and the change in the mechanical properties of DFW tungsten and graphite during pulsed energy release (pulsed electron irradiation, pulsed plasma irradiation).
- study of helium and hydrogen build-up and the effects of surface structure degradation (blistering, flecking) in DFW tungsten irradiated with alpha particles and protons.
- development of the theoretical models for estimating the effects of surface erosion (mass loss during local melting) under pulsed energy release.

Objects of research are:

Extremely pure DFW grade tungsten, produced by hot forging (double forging high purity tungsten fabricated by firm Plansee AG), as one of the most promising materials for protection of the Tokamak divertor plates;

Graphite of ARV grade, which is a structural material of nuclear facilities.

Subjects of research are:

The surface structure and mechanical properties of tungsten irradiated with low energy alpha particles, the sputtering coefficients of the surface of tungsten and graphite, the weight loss of tungsten and graphite irradiated by pulsed electrons and plasma.

Methods of research:

- Optical microscopy - to study the morphology of surface;
- Raster electron microscopy - to study the "fine" structure of surface;
- Atomic force microscopy - to study the structure and surface roughness;
- Thermal desorption spectroscopy - to determine the accumulation of implanted helium in the samples irradiated with alpha particles;
- Measurement of micro- and nano-hardness,
- Rutherford backscattering - for measuring the thickness of the sputtered layer during ion irradiation.

Scientific novelty of the dissertation research results

The dissertation paper provides the results of the complex study of the effects of helium and hydrogen ions and pulsed energy release on the structure and properties of DFW tungsten and graphite.

- the dependence of the tungsten sputtering coefficient on the fluence of argon ions irradiation was measured experimentally for the first time. It is shown that the sputtering coefficient increases with the growth of ions fluence.
- the mass loss of DFW tungsten was measured for the first time experimentally during irradiation with the high power pulsed electron beam. It is shown that the mass loss of tungsten is approximately two times less than the mass loss of molybdenum, under the same irradiation conditions.
- the theoretical model was developed for estimating the mass loss during the pulsed energy release on the surface. The novelty of the model implied consideration of the phase transformation, i.e. melting and evaporation, as well as thermal radiation from the irradiated surface. The calculated mass losses of DFW tungsten, molybdenum and graphite, irradiated by pulsed electrons, are in agreement with the experimental results.
- formation of blisters and peeling of the DFW tungsten surface, irradiated with low-energy alpha particles to high fluencies, was experimentally studied. It has been shown that under the irradiation fluence of 1.5×10^{18} cm^{-2}, blisters are formed on the surface, i.e. the pores, filled with helium, are released to the surface, some of them are open, i.e. there is a peeling of the surface.
- Based on the results of the studies on kinetics of helium desorption from the DFW tungsten, irradiated with low-energy alpha particles, the forms of helium occurrence were first determined and the relationship between blisters evolution and the strength properties of tungsten was established for the first time.

Main provisions for the defense:

1. The sputtering coefficient of tungsten increases by 20% with the growth of the fluence of argon ions (100 keV energy) from $5 \cdot 10^{+17}$ cm^{-2} to $1 \cdot 10^{+18}$ cm^{-2}. The experimental measurements of the sputtering coefficient for metallic materials were performed using the developed original technique based on determination of the sputtered layer thickness by the Rutherford backscattering method.

2.The experimentally measured mass loss during irradiation of DFW tungsten with the pulsed electron beam of 2 × 10-6 with the energy flux J = 4×1011 Вт×m-2 leads to the mass loss of 1.6 mg. Calculation of the mass loss according to the developed theoretical model is consistent with the experimentally measured mass loss.

3. Irradiation with alpha particles of 45 keV energy to the fluence of 11.5·10+18cm-2 results in formation of helium-filled pores, blistering and peeling in the surface layer of ~ 100 nm. It is established that ~ 25-30% of implanted helium is contained in the helium filled pores in the near-surface region. The main part of the implanted helium is contained in the helium-vacancy complexes that are stable to high temperatures (> 1100 ºC), leading to hardening in the near-surface region.

4. The experimentally measured mass loss during irradiation of graphite with the pulsed plasma beam is 4 mg at 13 pulses of 2 μs duration with the energy release of 10 J/cm2. Calculation of the mass loss according to the developed theoretical model is consistent with the experimentally measured mass loss.

Relationship of the dissertation paper with the plans of research programs.

The dissertation work was carried out within the framework of the sub-program 105 “Applied scientific research of technological nature in the area of atomic energy” under the Action “Scientific and technical support of experimental research at Kazakhstan’s materials science facility Tokamak KTM” of the Ministry of Energy of the Republic of Kazakhstan on the topic “Studies of the effects of charged particles radiation and plasma on thermal erosion of surface, accumulation of gas impurities and changes in the mechanical properties of the promising materials for the first wall protection of TNR facility" in 2015 - 2017 State registration №0115RK02433 and in 2018 State registration №0118RK01184.

Personal contribution of the author

The personal contribution of the author of the dissertation thesis, Aldabergenova T.M., is the involvement in determination of the thesis tasks, implementation of the experimental studies, development (in collaboration with the laboratory staff of the applied and theoretical materials science) of the erosion model, discussion of the works results and preparation of the publications.

Scientific and practical value of the work

The results of the completed research have substantial scientific and practical value:

- The obtained results of the complex studies on degradation of the structure and physical-mechanical properties of DFW tungsten under the impact of irradiation with plasma flows, pulsed release of energy and charged particles are the basis for making

the recommendations on the use of DFW tungsten as the material for diverter devices of the ITER reactor and the power TNR.

- The theoretical model for calculating the mass loss and the degree of the materials properties degradation under the effect of irradiation with plasma and charged particles can significantly limit the range of materials suitable for protection of the first wall and fabrication of the diverter devices. The model is also applicable for prediction of surface properties during materials processing by pulsed energy flows to improve their operational properties.

- The developed technique of experimental measurement of the sputtering coefficient of metallic materials, based on thickness determination of the sputtered layer by the Rutherford backscattering method, is applicable to a wide range of materials, including composite multi-component materials.

- The developed experimental techniques will contribute to efficient fulfillment of the materials-science tasks using the Kazakhstan KTM reactor.

Reliability and relevance of the results and conclusions

Reliability of the results obtained in the dissertation thesis is ensured by the fact that the studies were carried out with the correct application of the recommended experimental methods and techniques using the modern equipment. Correctness of the obtained results using the developed theoretical model is proved by comparing with the experimental results obtained for various materials. Relevance of the conclusions is provided by agreement with the fundamental principles of solid state physics and radiation material science. The results and conclusions of the dissertation thesis have been widely discussed at the international and regional conferences and workshops, as well as published in the international peer-reviewed journals.

Approbation of the dissertation paper

Materials of the dissertation paper were presented and reported at the following international conferences:

- Sumposium on Heavy ions fusion “21st Conference (Astana, Kazakhstan, June 18-22, 2016),
- Prospective materials with the hierarchical structure for new technologies and reliable structures” Conference (Tomsk, Russia, September 19-23, 2016)
- International scientific conference of students and young scientists, «Farabi alemi» (Almaty, Kazakhstan, April 9-12, 2018)
- Farabi Alemi” IV- International scientific conference of students and young scientists, «Farabi alemi (Almaty, Kazakhstan, April 10-13, 2017)
- Nuclear and Radiation Physics, Nuclear Science and Its Applications” XI-International scientific conference (Almaty, Kazakhstan, September 12–15, 2017)
- XII - International Conference of ION Implantation and other applications of ions and electrons” аттыХалықаралықконференцияда (Kazimierz Dolny, Poland, June 18-21, 2018)
- XIV - Solid State Physics, Functional Materials and New Technologies" International scientific conference (Bishkek., Kyrgyzstan, 1-5 August, 2018)

Publications

On the topic of the dissertation thesis 15 scientific papers were published, 8 of which were published in the proceedings of the international conferences, 4 articles were published in the journals recommended by CCES MES RK for PhD degree dissertations, two articles are indexed in the Web of Science database and one article with the impact factor is indexed in Web of Science database.

Structure and scope of the dissertation thesis

The dissertation paper includes introduction, three sections, conclusion and the list of references. The work is described on 102 pages of printed text, contains 66 Figures, 14 Tables and the list of references (106 points).

The first chapter provides the literary analysis of works on the main factors limiting the use of materials as protection against ionizing radiation and the energy fluxes. The main tasks of the dissertation paper are indicated at the end of the chapter.

The second chapter describes the main techniques used in the dissertation thesis. The focus is made on the original methods for determination of the coefficient of surface sputtering and measurement of material hardness in depth.

The third chapter provides the experimental and calculated results of the complex studies of the structure and properties of DFW tungsten and graphite under the effect of pulsed flows of energy and charged particles.