ABSTRACT
of the dissertation for the Doctor of Philosophy (PhD) degree
in specialty 8D05301 – "Physics"

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Structural phase states of tungsten as a result of carbidization in a beam-plasma discharge

General job description. The dissertation work is devoted to the study of the structural and phase states of tungsten after carbidization using the developed method of carbidization in a beam-plasma discharge. The work established the main patterns of the influence of carbidization conditions on the structural and phase states of tungsten.

Relevance of the research topic. As is known, the use of graphite and carbon fiber composites (CFC) as divertor materials for many years has made it possible to achieve significant progress in increasing the duration of discharges in tokamaks, increasing the temperature and density of the plasma, and in other directions. However, today, the full-tungsten divertor is the most promising for use in several existing and future tokamaks such as JET, ASDEX Upgrade, WEST and ITER due to tungsten's high thermal conductivity, high melting point (W) and low tritium retention. However, recent studies show that the use of W for areas with high ion and heat fluxes does not fully solve the problems accompanying the interaction of plasma with the divertor surface. Thus, an increase in the erosion of W under the influence of intense ion flows, the capture and retention of hydrogen isotopes (H) in W can lead to a reduction in the service life of plasma-facing components of thermonuclear installations. Additionally, another challenge when using a full-tungsten divertor is the weight of the divertor cassette, which is estimated at ~1000 kg and exceeds the weight limit (800 kg) for remote control. Thus, without completely excluding the use of graphite and CFC as divertor materials.

A number of tokamaks use either tungsten coatings deposited on graphite and carbon-graphite materials, such as in the Japanese JT-60SA tokamak, or uncoated graphite materials, such as in the Kazakhstan material testing tokamak KTM. However, erosion and transport of atomized graphite and carbon-graphite materials lead to the simultaneous irradiation of W by H isotopes, carbon ions (C) and hydrocarbon molecules, which can lead to the formation of tungsten carbide (WC and W_2C). Moreover, after conducting experiments with a plasma discharge, it was discovered that WC and W_2C phases are formed on the surface of a tungsten coating deposited on a graphite substrate. At high temperatures, the C atoms of the substrate diffuse into the tungsten coating, ultimately forming tungsten carbide.

However, the formation of carbidized layers on the surface of samples of candidate fusion materials as a result of sputtering C, its distribution into the plasma and into the area of the divertor table during the operation of the KTM tokamak has not yet been studied. To operate the KTM tokamak and obtain correct results, it is necessary to study in advance all possible scenarios for the interaction of plasma with the surface of samples of candidate fusion materials during experiments. Therefore, the relevance of this scientific work lies in the experimental study of the process of surface

carbidization during plasma-surface interaction, which will make it possible to predict the conditions for the formation of tungsten carbides on the samples under study during the operation of the KTM tokamak.

In this dissertation, in order to study the process of surface carbidization during plasma-surface interaction, a method of tungsten surface carbidization using a beam-plasma discharge (BPD) is considered. Although various methods are known for studying the interaction of carbon with tungsten and the formation of mixed layers, such as magnetron sputtering, mixing plasma flows of tungsten and carbon, chemical vapor deposition of carbon or evaporation of carbon using an electron beam evaporator. However, the uniqueness of the chosen method is due to the fact that a BPD seems to be the optimal way to simulate the peripheral plasma of tokamaks for preliminary testing of plasma-facing structural materials under well-controlled conditions. A characteristic feature of BPD is the transfer of significant energy from the beam electrons to the plasma, which is realized in a collisionless mode of beam propagation and is associated with collective interactions in the plasma-beam system. BPD allows you to obtain plasma using any working substances and with unique properties. Therefore, for experiments on the formation of a carbidized layer on the surface of tungsten, methane (CH_4) is used as a working gas. An analysis of literature data showed that as a result of interaction with an electron beam, methane gas decomposes into fragments such as H^+, H^{2+}, C^+, CH^+, CH^{2+}, CH^{3+} and CH^{4+}, which makes it possible to simulate the conditions of local transfer of carbon atoms along plasma-wetted surfaces due to hydrocarbons.

Thus, the topic of the dissertation work is justified and aimed at studying the structural and phase states of the tungsten surface during carbidization using BPD.

The purpose of the dissertation work is to establish the basic patterns of changes in the structural and phase states of tungsten as a result of carbidization in a beam-plasma discharge.

To achieve this goal, it is necessary to solve the following **tasks**:

1. develop a method for tungsten carbidization in a beam-plasma discharge and establish the conditions for its implementation in a plasma-beam installation;
2. determine the temperature-time parameters of tungsten carbidization in a BPD;
3. establish the main patterns of changes in the structural and phase states of the tungsten surface as a result of carbidization in BPD.

The object of the research is HP-grade tungsten (pure tungsten without additives) after carbidization in a plasma beam installation in a methane environment.

The subject of the research is the features of forming a surface mixed layer based on WC, W_2C, and the structural-phase states of tungsten after carbidization in a beam-plasma discharge.

Research methods. In the dissertation work, a plasma beam installation (PBI) was used to experiment on tungsten carbidization in a BPD. To study the structural-phase states of the tungsten surface after carbidization in a BPD, the following experimental research methods were used: X-ray diffraction analysis using the Crystallography Open Database and the PDF-2 ICDD Release 2004 database, scanning electron and transmission electron microscopy. As a diagnostic kit for plasma analysis, methods of probe diagnostics, optical spectrometry, and mass spectrometry of gas composition were used.

The scientific novelty of the work lies in the fact that for the first time

– a method for carbidization tungsten in a BPD was developed and the conditions for its implementation in a PBI were established;

– the features of the carbidization process in a BPD were studied and the temperature-time parameters of tungsten carbidization in a BPD were determined;

– for the first time, structural-phase transformations in the surface layers of tungsten were described and their main principles during carbidization in a BPD in the PBI were established.

The main provisions of the dissertation put on defense are

– **Developed a method for carbidization of tungsten surface in a beam-plasma discharge on a plasma-beam installation.** Carbidization of the tungsten surface in a BPD is carried out in a methane environment by applying a negative potential to a tungsten sample of 0.5 keV through the accumulation and diffusion of carbon in the near-surface region at a working gas pressure of $\sim(1–1.05)\cdot10^{-1}$ Pa and at a temperature sample surface from 1000 °C to 1700 °C with irradiation duration from 600 s to 3600 s.

– **Main temperature-time dependences of tungsten carbidization in a beam-plasma discharge.** Experiments on carbidization of the tungsten surface in SPR at temperatures from 700 °C to 1200 °C lead to the formation of a carbon coating in the form of a continuous film. The formation of WC was recorded after experiments at a temperature of 1000 °C. A change in tungsten surface temperature in the range from 1100 °C to 1400 °C leads to the simultaneous formation of carbide phases WC and W_2C. W_2C becomes the basis of the phase composition of the surface of samples after carbidization at 1500 °C – 1700 °C. At a temperature of 1300 °C, the phase composition of the carbidized layer depends on the duration of carbidization. At a temperature of 1700 °C, the formation of W_2C in the near-surface region of tungsten is completed, regardless of the duration of carbidization. After carbidization of pre-annealed samples, a noticeable increase in the content of the WC phase and a decrease in the content of the W_2C phase are observed relative to the initial tungsten.

– **Structural-phase transformations in the surface layers of tungsten during carbidization in a beam-plasma discharge.** At a temperature of 1000 °C, in the phase composition of the sample, in addition to the cubic phase of metallic W, the WC phase of a hexagonal close-packed structure appears. At temperatures of 1400 °C and above, the metallic W phase disappears from the phase composition, indicating that W is completely consumed for the formation of carbide phases, which occurs in three different ways: $W \rightarrow W_2C \rightarrow WC$, $W \rightarrow WC \rightarrow W_2C$, $W \rightarrow W_2C, WC$.

Practical significance. The obtained results of experimental studies of the process of surface carbidization using a beam-plasma discharge will make it possible to predict the conditions for the formation of tungsten carbides on the samples under study during the operation of the KTM tokamak, and will also have practical application in the formation of research programs for the interaction of plasma with the surface of samples of candidate materials of a thermonuclear reactor on the KTM tokamak. An act on the implementation of the results of the dissertation work into the procedure for conducting scientific and applied research in the field of interaction of plasma with the surface of materials at the branch of the Institute of Atomic Energy of the National Nuclear Center of the Republic of Kazakhstan was received. The obtained results of experimental research were also included in the collection of works of the National Nuclear Center of the Republic of Kazakhstan “Scientific and technical support for experimental research at the Kazakhstan Materials Testing Tokamak KTM. Under the general editorship of Batyrbekova E.G., Skakova M.K. ISBN 978-601-06-7964-1 2021 – 172 p.: ill. 168, Kurchatov, Branch “Institute of Atomic Energy” of the RSE “National Nuclear Center of the Republic of Kazakhstan” of the Ministry of Energy of the Republic of Kazakhstan, 2021».

Author's personal contribution. Setting the goals and objectives of the dissertation research, discussing the results obtained, and formulating the main conclusions were carried out jointly with scientific consultants. The author’s personal contribution consists of conducting an analytical review of literature data and a patent search, participating in the development and implementation of the carbidization method using a beam-plasma discharge, preparing and conducting experimental work, analyzing and processing the experimental data obtained, carrying out calculation work, and studying the patterns of change the composition of near-surface regions during tungsten carbidization using a beam-plasma discharge, the evolution of the morphology and composition of the surface, as well as in the preparation of major publications on the topic of the dissertation work. All experimental work was carried out in the laboratories of the materials science testing department of the branch of the Institute of Atomic Energy of the RSE NNC RK in close cooperation with specialists.

Connection of the dissertation topic with plans for research programs.

A significant part of this work was carried out with the financial support of the State Institution “Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan” within the framework of Agreement No. 281 dated November 16, 2020 on the topic “Study of the formation of a carbidized layer on the surface of tungsten under plasma irradiation” for 2020-2021. (AP08955992), and part of the work was carried out within the framework of the scientific and technical program “Scientific and technical support for experimental research on the Kazakh materials testing tokamak KTM” (BR09158585) on the topic 02.01.01. "Study of physical processes of surface carbidization of tungsten".

The degree of validity and reliability of the results obtained in the work is ensured by the correctness, accuracy, and originality of the tasks posed, the mutually consistent results of transmission and scanning electron microscopy, mass spectroscopy, and X-ray diffraction analysis. The main results of the dissertation were published in publications recommended by the Committee for Quality Assurance in the Field of Science and Higher Education of the Ministry of Science and Higher Education of the Republic of Kazakhstan, as well as peer-reviewed foreign scientific journals included in the Scopus and Web of Science databases.

Approbation of work results. The main results of the dissertation were reported and discussed at twelve international conferences:

1. 14th International Conference «Gas Discharge Plasmas and Their Applications» GDP, (Tomsk, Russia, September 15-21, 2019);
2. XXIV Conference on Plasma-Surface Interaction (Moscow, Russia, February 4-5, 2021);
3. International online conference «Advanced manufacturing materials and research: new technologies and techniques AMM&R2021» (Ust-kamenogorsk, Kazakhstan, 2021, February 19);
4. 15th International Conference «Gas Discharge Plasmas and Their Applications»; GDP, (Tomsk, Russia, September 5-10, 2021);
5. IX International Conference "Semipalatinsk Test Site: Heritage and Prospects for the Development of Scientific and Technical Potential" (Kurchatov, Kazakhstan, September 07-09, 2021);
6. III International Scientific Forum "Nuclear Science and Technology", dedicated to the 30th anniversary of Independence of the Republic of Kazakhstan (Almaty, Kazakhstan, September 20-24, 2021);
7. 10th International Conference on Nanomaterials and advanced energy storage systems (INESS-2022), (Astana, August 4-6, 2022);
8. International scientific and practical conference "Uvaliev Readings-2022", (Ust-Kamenogorsk, Kazakhstan, September 16-17, 2022)
9. 32^{nd} Symposium on Fusion Technology (SOFT2022), (Dubrovnik, Croatia, 18^{th} – 23^{rd} September 2022).
10. International Conference "ABDILDIN READINGS", (Almaty, Kazakhstan, April 12 - 15, 2023)
11. XXIV Conference on the interaction of ions with surfaces (Yaroslavl, Russia, August 21-24, 2023);
12. X International Conference "Semipalatinsk Test Site: Heritage and Prospects for the Development of Scientific and Technical Potential" (Kurchatov, Kazakhstan, September 12-14, 2023).

And also at three conferences-competitions:

1. XIX R&D Conference-competition of young scientists and specialists of the RSE NNC RK, National Nuclear Center of the Republic of Kazakhstan (Kurchatov, Kazakhstan, September 29 - October 1, 2020);
2. XX R&D Conference-Competition of Young Scientists and Specialists of the RSE NNC RK, Institute of Atomic Energy NNC RK (Kurchatov, Kazakhstan, October 6-8, 2021);
3. XXI R&D Conference-Competition of Young Scientists and Specialists of the RSE NNC RK, Institute of Atomic Energy NNC RK (Kurchatov, Kazakhstan, October 12-14, 2022).

In addition, the main results of the dissertation work were reported and discussed at scientific seminars of the Department of Physics, at joint scientific seminars of the Faculty of Natural Sciences and Technologies and the Scientific and Technical Council of the NJSC "East Kazakhstan University named after S. Amanzholov ", as well as at the Scientific and Technical Councils of the RSE NNC RK and Branch "Institute of Atomic Energy" RSE NNC RK.

Publications. The main results of the dissertation work were published in 18 printed works, of which: 3 articles in journals indexed in the Scopus and Web of Science databases, 3 articles in peer-reviewed scientific publications recommended by the Committee for Quality Assurance in the Field of Science and Higher Education of the Ministry of Science and Higher Education of the Republic of Kazakhstan.

Structure and scope of the dissertation. The dissertation consists of an introduction, four sections, a conclusion and a list of sources used. The work is presented on 108 pages, contains 60 figures, 9 tables and a list of used sources of 168 titles**.**