



## Material science activities for fusion reactors in Kazakhstan

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### A B S T R A C T

The paper describes the results of fusion reactor materials R&D activities in the Republic of Kazakhstan in the framework of the fusion material national program and the results of the activities on construction of material testing spherical tokamak in the RK.

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### 1. Introduction

The activities in the field of fusion material science have been carried out in accordance with the agreement between Government of Republic of Kazakhstan and Government of Russian Federation on Scientific-Technical Cooperation in the Framework of Development of Technical Project of International Thermonuclear Experimental Reactor (ITER). Main efforts of Kazakhstani scientists in ITER project were aimed at facility safety-related issues (mainly, tritium safety), assessment of beryllium materials application in fusion reactor design, as well as interaction of structural materials with hydrogen isotopes. [1–7]. At present the possibility of Kazakhstan participation in ITER Organization during construction and operation stages is discussed. Main activity directions and the obtained results are described below.

### 2. Assessment of beryllium materials application in fusion reactor design

There were carried out the verification of the computer codes for simulation of the emergencies related to the loss of coolant accidents (LOCA) in ITER reactor. Codes' verification was carried out at the mockup of first wall in a form of three-layer cylinder (beryllium, bronze (Cu–Cr–Zr) and stainless steel). The mockup was produced at Ulba Metallurgical Plant, Kazakhstan (UMP). LOCA accidents may result in hydrogen generation during exothermal reaction of beryllium and steam. In order to verify the codes simulating LOCA accidents the ITER Joint Group stated a task to experimentally assess interaction of beryllium with steam, namely, to define main dependencies of the parameters effecting intensity

of beryllium-steam interaction and to measure quantity of hydrogen generating during beryllium oxidation by steam. During the experiments the following activities were conducted:

- The emission of oxidized beryllium was measured for temperature range of 580–1120 K;
- Time dependences of temperatures of beryllium, bronze and steel were measured. The quantity of released hydrogen was measured;
- Possibility of beryllium– steam reaction was experimentally tested. Under realized experiment conditions the transition to self-sustaining reaction was not observed.

### 3. Interaction of structural materials with hydrogen isotopes under external impacts

Behavior of hydrogen isotopes (diffusion, permeation, and accumulation) was studied for the components of first armor and divertor taking into account temperature, pressure, and reactor irradiation. There were carried out out-of-pile and in-pile (reactors IVG.1M, WWR-K, RA of National Nuclear Center RK) studies of beryllium of various grades produced by the UMP technologies (TV-56, TShG-56, DV-56, TGP-56, TIP-56), graphites (RG-T, MPG-8, FP 479, R 4340), molybdenum, tungsten, steels (Cr18Ni10Ti, Cr16Ni15, MANET, F82H), alloys V-(4-6)Cr-(4-5)Ti, Cu+1%Cr+0.1%Zr, and double Be/Cu and triple Be/Cu/steel structures.

#### 3.1. Study of hydrogen permeation through structural materials under reactor irradiation

##### 3.1.1. Hydrogen permeation through stainless steel Cr18Ni10Ti

The studies were carried out at reactor IVG.1M in temperature range of 573–1073 K. The following results were obtained:

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**Table 1**

Hydrogen diffusivity and permeability in copper alloy obtained during reactor experiments.

	$D_0$ , $m^2/s$	$E_d$ , kJ/mole	$P_0$ , mole/ $(m \times s \times Pa^{1/2})$	$E_p$ , kJ/mole
Pre-reactor experiments	$2.69(\pm 0.3) \times 10^{-4}$	$81 \pm 3$	$1.55(\pm 0.1) \times 10^{-6}$	$88.0 \pm 3.7$
Reactor experiments (W = 2 MBt)	$1.1(\pm 0.1) \times 10^{-4}$	$76 \pm 3$	$2.45(\pm 0.2) \times 10^{-6}$	$82 \pm 3.4$
Reactor experiments (W = 5 MBt)	$1.96(\pm 0.2) \times 10^{-4}$	$79 \pm 3$	$0.53(\pm 0.1) \times 10^{-6}$	$74.4 \pm 3.1$

- Post-radiation effect of irradiation was observed, i.e. values of effective constants of diffusion, permeation, and solubility didn't return to their initial values under ordinary thermal diffusion.
- Increase of effective coefficients of hydrogen diffusion and permeability in steel during reactor irradiation: for example, under temperature of  $T = 773$  K effective diffusion coefficient increases in 3 times due to reactor irradiation, and effective permeability constant increases in 1.7 times in comparison with ordinary thermal diffusion, irradiation effects decrease with increase of the experimental temperature.

### 3.1.2. Study of hydrogen permeation through copper alloy Cu–Cr–Zr during reactor irradiation

Experiments to study hydrogen permeation through copper alloy (Cu+1%Cr+0.1%Zr) were carried out "in situ" at reactor IVG1.M for power levels of 2 and 5 MW in temperature range of 450–600 °C. Experimental results are given in Table 1 and published in [2]. Comparison of the results of reactor experiments (power level of 2 and 5 MW) showed that reactor irradiation results in decrease of diffusion activation energy of alloy Cu–Cr–Zr and in increase of permeability effective constant. Irradiation does not have sufficient effect on hydrogen diffusion coefficient in given copper alloy.

### 3.1.3. Study of hydrogen permeation through vanadium alloy during reactor irradiation

Determination of parameters of hydrogen isotope permeation through vanadium alloys is complicated by the processes taking place on surface of the alloys. Therefore, the diffusion coefficients are determined by using the methods independent of surface state. The experiments showed that use of preliminary treatment of the sample (cleaning of a surface from non-metal impurities) may result in the surface state when results obtained by the permeability technique are comparable with the results obtained by other techniques [4]. To obtain reliable results the studies were carried out for one sample of V4Cr4Ti alloy by using various techniques described in [5]. It was showed that reactor irradiation resulted in increase of hydrogen effective diffusion coefficients that can be explained by cleaning of the surface. Investigation of hydrogen release from V4Cr4Ti alloy under reactor irradiation with different power levels (1, 2 and 6 MW) allowed us to obtain the temperature dependence of the hydrogen pressure over the sample during isochronous heating. The solubility parameters values were de-

**Table 2**

Parameters of hydrogen solubility in V4Cr4Ti (in-pile and out-of-pile experiments).

Experiment	$S_0$ , (%) mole/ $(m^3 \times Pa^{1/2})$	$E_s$ , (%) kJ/mole
In-pile, 6 MW	$3.98E-02 \pm 5$	$-35.4 \pm 5$
In-pile, 2 MW	$3.55E-02 \pm 5$	$-34.6 \pm 5$
In-pile, 1 MW	$3.16E-02 \pm 5$	$-36.9 \pm 5$
Out-of-pile	$1.30E-02 \pm 5$	$-39.2 \pm 5$

rived in Table 2. The results show the visible effect of reactor irradiation on hydrogen solubility in the V4Cr4Ti alloy – reactor irradiation increases the hydrogen solubility in 1.5–2 times and changes the phase transition temperature V4Cr4Ti alloy.

### 3.2. Study of hydrogen accumulation and gas release from irradiated samples of graphites and beryllium

#### 3.2.1. Study of hydrogen accumulation and gas release from irradiated samples of RGT-graphite

Technique of thermostimulated gas release under linear heating was used to study hydrogen accumulation in irradiated and reference samples. Hydrogen concentrations in the samples with various preliminary treatments are given in Table 3. Relative concentrations were calculated according to thermal-desorption curves for temperature range of 300–1700 K. Gas release spectra clearly show that hydrogen retention increases with temperature growth. Important result of the study is first discovered effect of joint impact of hydrogen, temperature and irradiation on characteristics of hydrogen generation in graphite, which shows itself in increase of hydrogen retention in the sample [6].

#### 3.2.2. Hydrogen interaction with beryllium during reactor irradiation

TV-56 grade beryllium samples made by UMP were irradiated at reactor IVG1.M (during 6 h in medium of hydrogen and nitrogen) and reactor RA (during 50 h in deuterium medium). Main results are published in [7]. One of the study goals was to carry out the experiments allowing for determination of correlations between irradiation parameters, such as irradiation spectrum (ratio between fast and thermal neutrons) and its intensity (fluxes of neutrons and gamma-quanta by groups). As a result, the experiments were carried out at the reactor RA, which spectrum sufficiently differs from spectrum of reactor IVG1.M. Hydrogen concentrations in beryllium samples calculated by thermal-desorption spectra for temperature range of 300–1200 K are given in Tables 4 and 5.

The experimental results showed difference of experimental data for the samples irradiated in hydrogen medium for various reactors.

Some important effects were discovered:

**Table 3**

Hydrogen concentration in RGT-graphite samples with various preliminary treatments.

Sample treatment type	Hydrogen content, appm	
	920 K	1150 K
1. Sample irradiated in hydrogen	71.2	94.3
2. Sample irradiated in nitrogen	34.8	33.1
3. Non-irradiated sample saturated in hydrogen	40.3	42.7
4. Sample irradiated in nitrogen and then saturated in hydrogen	–	54.6

**Table 4**

Hydrogen concentration in beryllium TV-56 calculated by gas release spectra in range 300–1200 K (samples irradiated in reactor IVG1.M).

Beryllium sample treatment type	Released hydrogen, molecule/cm <sup>2</sup>
Initial samples cut across spinning axis	$1.20 \times 10^{17}$
Sample irradiated in nitrogen and then saturated in hydrogen, T = 920 K	$1.05 \times 10^{17}$
Sample irradiated in hydrogen, T = 920 K	$1.14 \times 10^{17}$
Sample irradiated in hydrogen, T = 1150 K	$2.49 \times 10^{17}$
Sample irradiated in nitrogen and then saturated in hydrogen T = 1150 K	$1.12 \times 10^{17}$

**Table 5**

Deuterium concentration in beryllium TV-56 calculated by gas release spectra in range 300–1200 K (samples irradiated in reactor RA under temperature of  $T = 1150$  K).

Beryllium sample treatment type	Released deuterium, molecule/cm <sup>2</sup>	
<i>Samples cut across spinning axis</i>		
Irradiated	D <sub>2</sub>	$0.14 \times 10^{17}$
	DH	$0.13 \times 10^{17}$
Non-radiated	D <sub>2</sub>	$0.12 \times 10^{17}$
	DH	$0.13 \times 10^{17}$
<i>Samples cut along spinning axis</i>		
Irradiated	D <sub>2</sub>	$0.23 \times 10^{17}$
	DH	$0.17 \times 10^{17}$
Non-radiated	D <sub>2</sub>	$0.22 \times 10^{17}$
	DH	$0.19 \times 10^{17}$

- Hydrogen retention in beryllium samples irradiated in hydrogen is two times larger than that for the samples saturated after irradiation and for the reference samples. This effect suggests that it is improper to estimate retention and accumulation of tritium in ITER reactor on the basis of data obtained for the samples saturated after irradiation.
- It was shown that relatively thin surface layer of beryllium samples answered for gas release. For the first time it was detected that reactor irradiation with low fluences under simultaneous hydrogen impact and high temperature may result in splitting of beryllium surface, which was verified by the data of the studies carried out by using the electronic-probe microanalyzer JXA 733 (Superprobe 733). X-ray spectrum microanalysis (shooting of raster pictures in secondary electrons) was used for analysis of the surface of the samples. Microstructure studies showed that surfaces of the samples irradiated in hydrogen, in comparison with the samples irradiated in nitrogen, had open porosity; beryllium oxide layer of irradiated samples became fine-dyspersated, loose. The increase of BeO layer was observed for all the irradiated samples, but the samples irradiated in hydrogen medium in reactor IVG1.M had macroscopic effect of surface bursting.

#### 4. Study of promising materials of fusion liquid-metal blanket

##### 4.1. Study of tritium penetration from Pb–Li eutectic (Li17Pb83) through stainless steel and low-activated steels MANET and F82H, including ones with protective coatings

The reactor and post-reactor experiments were aimed to estimate tritium accumulation rate in diffusion cell, to estimate tritium discharged in a volume over eutectics and to define parameters of tritium permeation from Pb–Li eutectic through stainless steel Cr18Ni10Ti. There was also studied tritium permeability from eutectic Pb+17%Li through steel MANET taking into account protective coating effects. The container with the eutectics of various temperatures was irradiated at the reactor IVG1.M. Irradiation was carried out under various reactor power of 0.5, 1 and 2 MW. Irradiated eutectic volume was  $\sim 10$  cm<sup>3</sup>. During experiment the gas composition over eutectics was registered. Diffusion cell with two tube samples is given in Fig. 1.

Three types of MANET samples were studied: tube sample without coating, and the samples with two types of Al<sub>2</sub>O<sub>3</sub> coating–melt dipping at the enterprise FZK, Germany, and chemical deposition from gas phase at the enterprise GEA, France; the samples of F82H low-activated steel with coating Cr<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>+P<sub>2</sub>O<sub>5</sub> was developed in JAERI (Japan) and applied by using method of gas phase precipitation. It was obtained that under irradiation:

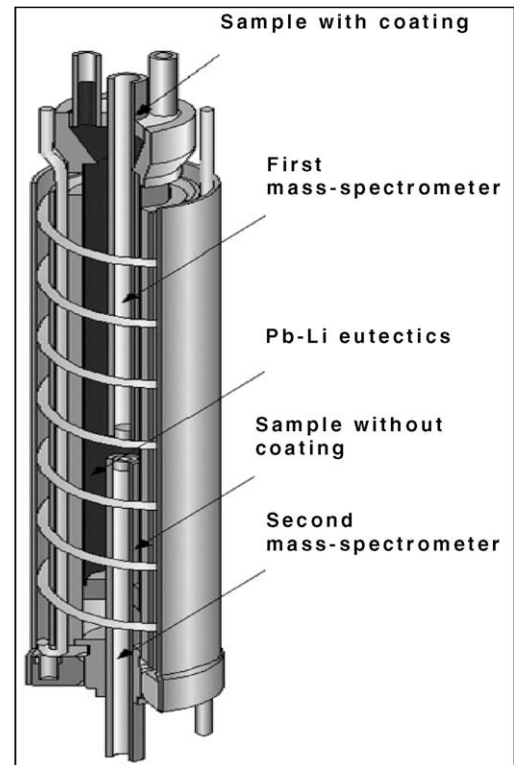


Fig. 1. Scheme of diffusion cell with tubular sample.

- The tritium fluxes through MANET steel sample with FZK coating were failed to be measured, i.e. the fluxes were smaller than sensitiveness of mass-spectrometer apparatus (less than  $1E-13$  mole/s);
- Coating CEA reduces tritium flux from Pb–Li eutectic under reactor irradiation (MANET steel) in 2.5 times.
- During experiments there were not observed visible degradation of the coatings under study.
- It was established that tritium flux through sample of low-activated steel F82H with coating Cr<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>+P<sub>2</sub>O<sub>5</sub> was practically on the background level under following experimental conditions: neutron-flux density  $\sim 1 \times 10^{14}$  cm<sup>2</sup> s<sup>-1</sup>, 12 h of reactor irradiation, temperature range of 673–1000 K. The result is evidence of high stability of this glass–ceramic coating.

##### 4.2. Radiation tests of lithium ceramic Li<sub>2</sub>TiO<sub>3</sub> enriched with <sup>6</sup>Li (96%) up to 20% burn-up by <sup>6</sup>Li with “in situ” registration of generated tritium under various temperatures

Project goal is the radiation tests of lithium ceramic Li<sub>2</sub>TiO<sub>3</sub> (pebbles and pellets) up to 20% burn-up by <sup>6</sup>Li with in situ registration of generated tritium under various temperatures (400–900 °C) during reactor irradiation with post-radiation studies of irradiated samples of lithium ceramics. The irradiation was carried out in two irradiation channels of reactor WWR-K. The active ampoules (A-type) were equipped with the system for continuous control of tritium release “in situ”, passive ampoules (P-type) were equipped with tritium accumulation system. Passive ampoules were meant to provide 20% burn-up in lithium ceramic samples (by <sup>6</sup>Li) under constant sample temperature. Universal loop facility allowed for control and maintenance of operating temperature of each ampoule during all the time of irradiation (220 days or 5350 h) [8]. During long-term irradiation the unique samples with all-time high lithium burn-up 18% for pellets, 23% for A-type pebbles and 20% for P-type pebbles were received.

Main results are

- As a result of long-term irradiation lithium ceramic softens, at that this effect is more for irradiation temperature of 490 °C, than for 650 °C.
- Quantity of residual tritium is greater in the samples irradiated under lower temperatures.
- Radiation change of phase composition of lithium ceramics  $\text{Li}_2\text{TiO}_3 + 5 \text{ mol}\% \text{TiO}_2$  with formation of new phases –  $\text{LiTi}_2\text{O}_4$ ,  $\text{LiTiO}_2$  and  $\text{Li}_4\text{Ti}_5\text{O}_{12}$ .
- After irradiation pebble density decreased and the effect was greater for irradiation at higher temperatures.
- After reactor irradiation the pebbles acquired dark color, accordingly, quantity of dark component (containing lesser density and microhardness) became greater.

### 5. Creation of experimental complex on the basis of Tokamak KTM for material testing

At present Kazakhstani Tokamak for Material Testing (KTM) is created in National Nuclear Center of Kazakhstan in cooperation with Russian Federation organizations. KTM facility mounting is planned to be completed in 2009. Start-up is scheduled for 2010. KTM is the spherical tokamak with aspect ratio  $A = 2$ , that allows for performance of unique studies of boundary magnetic configuration of extremely compact toroids and classical Tokamaks. Moreover, tokamak KTM may be used for unique studies of plasma–surface interaction by simulation of neutron loads with great heat loads up to 10–20 MW/m<sup>2</sup> like ITER conditions. Main parameters, systems and facility opportunities are given in [9–10]. The main stages of Scientific-Research Program for KTM experimental complex are:

- Physical studies of tokamak plasma, including ohmic mode and modes with plasma additional RF-heating.

- Material studies and tests for first wall armor materials and receiving divertor tiles.
- Studies of systems for control over processes at first wall, in boundary plasma, in divertor, equilibrium plasma, as well as fluxes in divertor area.
- Creation of physical and technological diagnostics of plasma column, processes in divertor area, boundary plasma.
- Creation and work-out of demonstration mockups of divertor intrachamber components plasma facing by using lithium technologies.

Tokamak KTM has an opportunity of operative access to vacuum chamber to replace divertor components without loss of vacuum. Parameters of energy loads, wide range of used methods and diagnostics allow for high level studies and tests in divertor volume and at first wall, which is highly important for study of plasma facing materials in ITER and DEMO programs.

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