Tests of lithium CPS on basis of carboxylic fabric, reinforced with carbon nanotubes under high thermal and radiation loads

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Abstract. This work is devoted to testing a lithium CPS based on carbon fabric reinforced with carbon nanotubes under conditions of thermal and radiation loads. The paper considers and analyzes: the properties of carbon nanotubes and methods of their synthesis, the nature of the interaction of carbon materials with liquid lithium at different temperatures. A description of all the main stages in the manufacture of lithium CPS based on carbon fabric reinforced with carbon nanotubes is given. Microstructural studies of a manufactured lithium CPS sample based on carbon fabric reinforced with carbon nanotubes are presented. Studies have shown that a carbon fabric with a fiber surface reinforced with carbon nanotubes is completely wetted by liquid lithium. The developed technology is fully suitable for the manufacture of lithium CPS samples for further research. The results of experiments on the interaction of lithium CPS based on graphite fabric reinforced with carbon nanotubes with hydrogen isotopes under thermal and radiation loads are presented.

Keywords: lithium CPS, carbon nanotubes, reactor experiments, deuterium, sorption.

1. Introduction

Materials science studies conducted as part of the implementation of international programs for the creation of fusion reactors ITER and DEMO have shown that one of the best materials for use as a PFM (plasma-facing material) [1] in controlled fusion plants is liquid lithium and materials based on it [2,3,4]. The use of liquid lithium as a plasma-facing material is especially promising if lithium is enclosed in a so-called capillary-porous system (CPS) [5, 6]. Currently, there are many studies in the world conducted with lithium CPS, which once again confirms the interest in this material [7,8,9,10,11,12,13]. Work on this topic is also being carried out at the Kazakhstan Materials Science Tokamak KTM [14,15,16,17,18,19,20]. The installation is currently the only tokamak in the world designed to solve problems in the field of thermonuclear materials science. Kazakh scientists, in cooperation with leading international scientific organizations in this field, are implementing a large-scale project to create and test a model of a lithium divertor (MLD), which is used in the KTM tokamak, as one of the replaceable segments of the receiving divertor device. The conducted research has confirmed the prospects of using lithium and lithium technologies in controlled fusion plants. However, the available technology for manufacturing lithium CPS requires further development in terms of
optimizing the material of the main matrix in which liquid lithium is enclosed. One of these materials is carbon fiber reinforced with nanotubes. An important step for the implementation of this idea is to conduct tests of lithium CPS based on graphite fabric reinforced with carbon nanotubes under conditions modulating the actual operation of thermonuclear reactors.

2. CNT-based lithium CPS sample manufacturing
For CNT-based lithium CPS sample manufacturing a special experimental device was developed (see figure 1) located on the VIKA facility [21]. The manufacturing technology CNT-based lithium CPS samples is described in detail in [22,23].

Subsequently, work was carried out to fill the carbon fabric with lithium. So, using the filling technology described in detail in [17], prototypes of lithium CPS based on carbon fabric with CNT fibers synthesized on the surface were manufactured (see Figure 2).

3. Experiments on the interaction of lithium CPS based on carbon fabric reinforced with CNTs with hydrogen isotopes under thermal loads
Experiments with samples of lithium CPS based on carbon fabric reinforced with CNTs were carried out on the VIKA experimental setup. Experimental setup VIKA [26] makes it possible to study the processes of release of various gases from materials of nuclear, fusion reactors by the TPD method (temperature-programmed desorption). The installation allows carrying out experiments at temperatures of the sample under study in the range from 300 K to 1750 K, with different heating rates. During the heating process, the possibility of mass spectrometric registration of released gases in real time was realized. The installation is equipped with systems for purification and controlled supply of various gases into the working chamber, as well as various experimental ampoule devices mounted on the VIKA installation.

At the beginning, a series of thermal desorption experiments was carried out, in which, during the heating of the sample under study, the partial pressures of residual gases in the volume of the ampoule device were continuously recorded. Figure 3a, b shows the time dependences of the sample temperature and partial pressures of the gases.
Figure 3. Diagrams of changes in the partial pressures of gases released from a lithium CPS sample during heating (a) – experiment 1, (b) – experiment 2

Next, an experiment was carried out on the sorption of deuterium by lithium CPS based on carbon fabric reinforced with CNTs. The experiment itself was carried out using the gas absorption method. The method is based on studying the migration of an impurity from the surrounding atmosphere into a solid and makes it possible to simultaneously determine the solubility constant and the diffusion coefficient. During the experiment, either a decrease in the concentration of diffusant in the gas phase or an increase in its amount in the sample is recorded. Throughout the experiment, changes in the gas pressure in the ampoule, as well as the partial pressure of residual gases, were continuously recorded. As a result of the experiment, data were obtained from which the time dependences (Figure 4 a) of the sample temperature and the partial pressures of the recorded gases in the ampoule device were obtained.

Figure 4. Diagram of monitoring of the gas phase (a) and the absolute pressure of deuterium in the ampoule device (b)

Figure 4 b shows diagrams of monitoring of the gas phase recorded through the leak using an RGA-100 mass spectrometer and the absolute pressure of deuterium in an ampoule device with a sample of lithium CPS based on carbon fabric reinforced with CNT. As a result of the experiments on thermal desorption and sorption of samples, experimental data were obtained that allowed us to proceed to a further stage of testing under reactor radiation conditions.

4. Experiments on the interaction of lithium CPS based on carbon fabric reinforced with CNTs with hydrogen isotopes under conditions of reactor radiation

At the next stage of the work, a reactor experiment was carried out with lithium CPS based on carbon fabric reinforced with CNTs. The experiment was carried out on the LIANA test bench [24,25,26,27,28]
located at the IVG.1M reactor [29, 30, 31, 32] hall. For the experiment, a previously developed irradiation device was used to study the materials of fusion reactors.

The gas absorption method was chosen for the research. The essence of the method was as follows: after all the conditions necessary and determined by the worked-out method for conducting reactor experiments were achieved in the ampoule with the lithium CPS sample, the steady-state temperature of the sample was established. Then, a known amount of deuterium was supplied into the chamber, followed by the saturation of the sample from the gas phase at the selected temperature. Throughout the experiments, the change in the gas pressure in the ampoule was continuously recorded. This was followed by a repetition of the experiment, but at a different temperature studied by the CPS. After carrying out one series of experiments, which consisted in passing the investigated temperature range from 473 to 623 K, all the gas contained in the chamber was pumped and the sample was heated to 773 K for 300 s. In the future, the following series of experiments was carried out.

The research conditions were as follows:
- investigated sample temperature range - from 473 K to 623 K;
- residual pressure in the measuring path – from $10^{-4}$ K to $10^{-6}$ Pa;
- pressure of deuterium supplied to the ampoule – from 80 Pa to 400 Pa;
- thermal power of the IVG.1M reactor – 500 kW, 1 MW and 2 MW.

Figure 5 shows the graphs of the time dependence of temperature of the lithium CPS sample and hydrogen pressure in the saturation chamber during reactor experiments for different reactor power levels (0.5 MW; 1 MW and 2 MW).

![Figure 5](image)

**Figure 5.** Dependences of temperature and pressure of deuterium in an experiment with a lithium CPS sample under neutron irradiation

During the analysis of the results of reactor experiments on deuterium sorption of the lithium CPS, the processes occurring on the surface of liquid lithium enclosed in the CPS when it is saturated with deuterium were considered. Based on the analysis, the constant of the interaction of lithium CPS based on CNT with deuterium was calculated in the Mathcad calculation complex (Figure 6).
From the obtained temperature dependences, the main parameters of the interaction were determined, such as the activation energies of the processes, and the pre-exponents in the Arrhenius dependence. The results are shown in table 1.

\[ K_0 = K_0 \exp \left( -\frac{E_a}{RT} \right) \]  

where \( E_a \) – activation energy J, \( T \) – temperature K, \( K_0 \) – pre-exponents factor mol/(m\(^2\) s Pa), \( R \) – universal gas constant J/(mol·K).

### Table 1 Calculated parameters of the interaction of hydrogen in a lithium CPS

| Experiments | \( K_0 \), mol/(m\(^2\) s Pa) | \( E_a \), kJ/mol | Notes       |
|-------------|-------------------------------|------------------|-------------|
| 1           | \((1.3 \pm 0.7) \cdot 10^{-3}\) | 47 ± 3           | Without irradiation |
| 2           | \((1.6 \pm 0.9) \cdot 10^{-3}\) | 47 ± 3           | 0.5 MW      |
| 3           | \((6.2 \pm 2) \cdot 10^{-4}\)  | 45 ± 3           | 1.0 MW      |
| 4           | \((5.4 \pm 3) \cdot 10^{-5}\)  | 44 ± 3           | 2.0 MW      |

### Conclusion

As a result of the work performed, a literary review was carried out, during which the following were considered and analyzed: the properties of carbon nanotubes and methods of their synthesis; the nature of the interaction of carbon materials with liquid lithium at different temperatures. Lithium CPS based on a carbon fabric matrix reinforced with CNT test samples using the previously developed technology was manufactured. Microstructural studies of the manufactured lithium CPS samples were carried out. The study of the microstructure of the samples surface showed that the matrix of carbon fabric reinforced with CNT was completely filled with metallic lithium. Bench tests of lithium CPS were carried out using a special experimental device, with the help of which thermal desorption experiments and experiments were carried out to determine the sorption properties of lithium CPS in relation to deuterium. Reactor tests of lithium CPS samples based on carbon fabric reinforced with carbon nanotubes were carried out at the stationary IVG.1M reactor by gas absorption method. The analysis of the data obtained during the tests of lithium CPS based on carbon fabric reinforced with CNT was performed, as a result of which the temperature dependences of the deuterium-liquid lithium interaction constant were calculated at different neutron irradiation fluxes and without irradiation, the main interaction parameters such as activation energies of processes and pre-exponents in the Arrhenius dependence were determined from the obtained temperature dependences. As can be seen from the above data, with an increase in the
thermal power of the IVG.1M reactor, an increase in the interaction constant of hydrogen isotopes with a lithium CPS reinforced with carbon nanotubes is observed.

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