

High-temperature test of tin-lithium CPS under deuterium plasma irradiation conditions

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ABSTRACT

This paper is devoted to experiments on testing of the Sn-Li capillary porous system (CPS) under conditions of deuterium plasma irradiation, carried out at a plasma-beam installation (PBI). As a metal CPS matrix, molybdenum mesh was used. The paper presents a detailed description of the development of technology and procedure for the fabrication of the investigated sample, intended for plasma testing, as well as experiments on irradiation of Sn-Li CPS with deuterium plasma. As a result of the experiments performed, the dependences of the temperature of the investigated sample on the energy of the plasma contacting with the CPS, time dependence of the gas phase composition in the PBI chamber during irradiation of Sn-Li CPS with deuterium plasma, optical spectra depending on the temperature were obtained. Post-experimental material science studies with a sample of Sn-Li CPS were also performed and the results of microstructure, thermal and X-ray phase analysis were obtained. Analysis of the experimental data obtained showed that the energy of deuterium plasma precipitated on the Sn-Li alloy is re-emitted into the energy of optical radiation on the evaporating neutral atoms of lithium and tin. A complete evaporation of the alloy from CPS, during interacting with deuterium plasma, leads to partial destruction of the metal matrix of molybdenum.

1. Introduction

An important area of work in creating new fusion reactors (FR) is research to justify the use of liquid metals and alloys as plasma-facing materials (PFM) [1–14]. Lithium, gallium and tin are considered as possible liquid metal components for use in tokamaks. In almost all parameters (small core charge, thermo-physical properties, corrosion interaction with structural materials), lithium is superior to other fusible metals. In addition, lithium is the most studied considering its properties from the point of view of the requirements for the tokamak PFM and compatibility with the structural materials of the listed liquid metals and alloys. Although lithium has a number of advantages in its properties over other low-melting metals, it should be mentioned that there are two problems that have not been fully solved yet, related to the possibility of

accumulation of high concentrations of tritium in lithium and increased vapor pressure over its melt. These factors are important from the point of view of ensuring the safety of future fusion reactor (FR) and the purity of fusion plasma.

Recent studies indicate the prospects of the tin-lithium (Sn-Li) alloy as a new liquid metal for protecting the in-vessel elements of a FR from the energy flows and high-density particles. The main hope for this alloy is associated with the low vapor pressure of the Sn-Li, which raises the upper temperature limit of the alloy use compared to pure lithium (450–500°C) in the terms of the fusion plasma contamination. The majority of studies at plasma-physical facilities use lithium and lithium capillary-porous structures (CPS) [15–21]. Research results show that at lithium temperature above 500°C there is an intense process of metal evaporation, which subsequently condenses on almost all internal

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surfaces of the vacuum chamber of the plasma-physical facilities. Therefore, attention was paid to alloys of lithium with fusible metals that have low vaporization parameters and high heat capacity. A candidate for creating such an alloy is tin. The metal in the liquid phase has a low value of the saturated vapor pressure and, therefore, Sn-Li alloys are perspective for use in the FR.

However, there has been limited work worldwide on the use of Sn-Li alloys as a PFM and, therefore, there are few data on such studies. For example, there are data on the interaction of hydrogen isotopes with liquid tin and Sn-Li alloy, which were carried out both by calculation-theoretical methods [22] and during helium plasma irradiation at the experimental setup [23]. There are also data from plasma experiments with a Sn-Li alloy that were carried out on the ISTTOK tokamak [24,25] and on the TJ-II stellarator [26]. In these experiments, an alloy with a 20 at% Li - 80 at% Sn percentage was used. On the FTU tokamak [27] a limiter was tested, the surface of which was a CPS filled with liquid tin.

Nevertheless, validated experimental data on the tests of this material under extreme heat, radiation, and plasma loads are required in order to fully justify the application of the lithium-tin alloy as PFM in the FR. The present paper focuses on Sn-Li CPS's high-temperature test under deuterium plasma irradiation conditions, with subsequent post-experimental materials science investigations.

2. Materials and methods

2.1. Procedure of Sn-Li CPS target fabrication

During the initial phase of this work, Sn₇₃-Li₂₇ alloy was fabricated. Procedures for alloy fabrication with a given ratio of lithium and tin were implemented at the VIKA facility [28,29]. According to the technological processes outlined in [30], an experimental sample of Sn-Li alloy with 27% lithium atoms was manufactured.

The next step was the fabrication of the Sn-Li CPS. For this purpose, a special target, made of MP grade molybdenum was designed (Fig. 1) to perform for experiments on the interaction of Sn-Li CPS with hydrogen isotopes under electron and plasma irradiation conditions.

Further, the fabricated sample was placed in a specially designed (Fig. 2) and constructed experimental device [32]. To provide the necessary gas-vacuum conditions, an experimental facility [33], consisting of a vacuum post, an RGA-100 quadrupole mass spectrometer, pressure sensors, and vacuum fittings was used. Fig. 3

The procedure for filling the CPS matrix (which is a molybdenum mesh with a mesh size of about 0.3 mm) with Li-Sn alloy was as follows: after placing Sn-Li alloy (5.21 g) into the molybdenum target and loading it into the experimental device, high-temperature annealing in hydrogen medium at a pressure of 750 Torr and temperature of 1000 °C for 2 h were held; then the hydrogen was pumped-out, and the target was further annealed at 1000 °C for another 1 h under continuous vacuum pumping. During this heating process, the lithium-tin alloy completely saturated the receiving surface of the CPS matrix. Thus, as a result of the procedures described above, a sample of Sn-Li CPS was fabricated, the appearance of which is shown in Fig. 4.

Further, the fabricated Sn-Li CPS target was placed in the collector

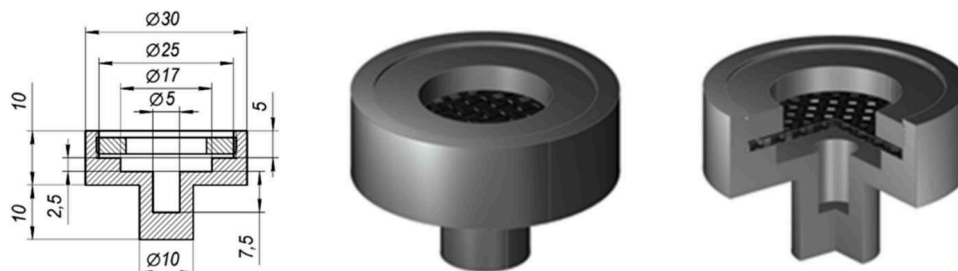


Fig. 1. Drawing and 3D model of the developed target [31].

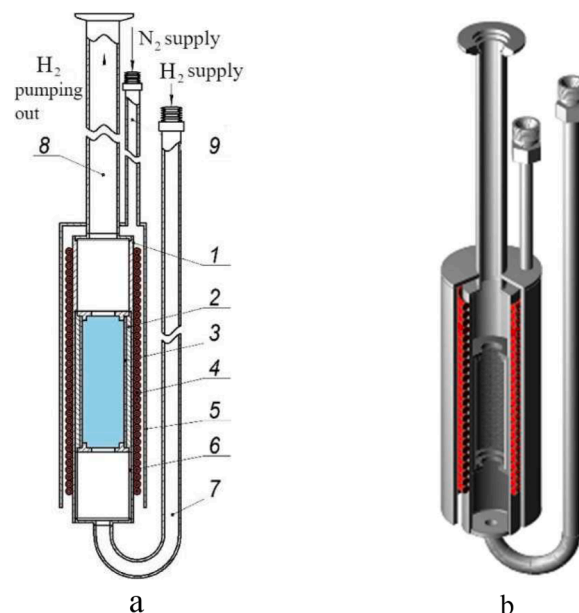


Fig. 2. Drawing (a) and 3D model (b) of the developed experimental device. 1 – casing of the experimental device; 2 – cell for CPS; 3 – molybdenum mesh; 4 – heating element; 5 – cooling jacket; 6 – thrust ring; 7 – path for hydrogen isotope supply; 8 – vacuum pumping path; 9 – path for N₂ supply.

node of the PBI in order to conduct experiments on the irradiation of Sn-Li CPS with deuterium plasma.

2.2. Irradiation of Sn-Li CPS by deuterium plasma

High-temperature tests of Sn-Li CPS under deuterium plasma irradiation conditions were performed on a plasma-beam installation (PBI) [34–36]. The PBI is a linear simulator with electron-beam plasma generation that enables the combination of plasma irradiation with an electron beam with a high heat flux. It is focused on multipurpose and the potential for quick changeover to handle a variety of customized tasks. Depending on the task, both the plasma irradiation mode and the mode of direct heating of materials with an electron beam can be used in PBI.

After the above-described works the experiment on irradiation of Sn-Li CPS with deuterium plasma was conducted. Parameters of irradiation were the following: ion density - $3 \cdot 10^{17} \text{ D}^+/\text{m}^3$, ion energy - 500 eV; plasma temperature of about 13.5 eV. Experiments on deuterium plasma irradiation of tin-lithium CPS were carried out in the temperature range of 100 °C to 850 °C. All experimental parameters were recorded *in-situ*. Photographs of the target with Sn-Li CPS during the plasma experiment are shown in Fig. 5.

The pressure of residual gasses in the cavity of the interaction chamber was measured using an RGA-100 mass spectrometer. Optical spectra in the interaction chamber of the unit were recorded using an

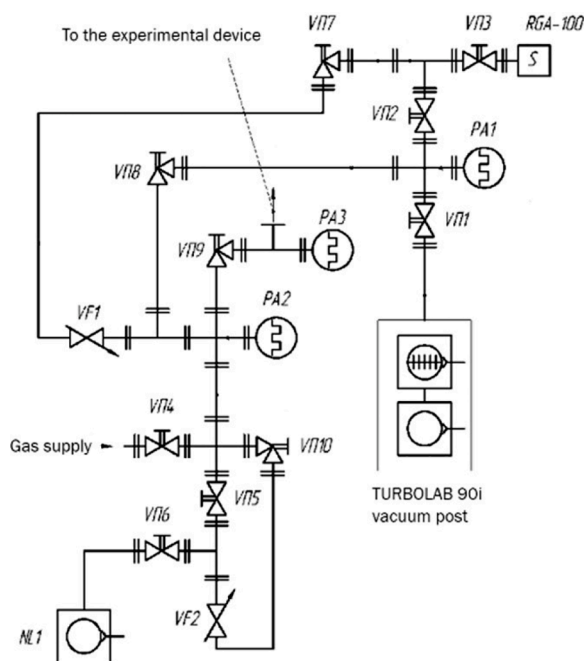


Fig. 3. Gas-vacuum scheme of an experimental facility. NL1 – spiral pump; VII1...VII3 – manual valve KF 40; VII4...VII6 – manual valve KF 16; VII7–manual angle valve KF 25; VII8...VII10 – manual angle valve KF 16; PA1...PA3 – vacuum gage; VF1...VF2 – vacuum inlet valve; RGA-100 – residual gas analyzer.



Fig. 4. Fabricated target with Li-Sn CPS.

optical spectrometer HR2000+ ES (Ocean Insight) with using Ocean-View license software. Deuterium plasma parameters were recorded by probe diagnostics using a Langmuir probe, a Keithley 6487 milliammeter and specialized software. Temperature measurement from the back side of the sample was carried out using a W-Re-5/20 type

thermocouple with connection to a single-channel TRM-201 temperature controller. Also, in addition, the temperature measurement from the front side of the Sn-Li CPS sample was carried out using an optical pyrometer Metis 322. The signal from the pyrometer was transmitted to the information and measurement system of the PBI to provide power regulation by PID controller. As a result of the conducted experiments during irradiation by deuterium plasma, the optical spectra of the test sample at various temperatures were recorded, as well as mass spectra change of the gas phase in the PBI chamber.

3. Results and discussion

3.1. Results of plasma irradiation

The obtained experimental data were processed and the dependence of the change in the composition of the gas phase in the PBI chamber was plotted (Fig. 6).

Optical spectra were also plotted, which recorded the change in the intensity of the emission of resonant lines of deuterium, tin, and lithium from changes in the temperature of the studied sample (Fig. 7).

Sources [37,38] were used to identify the main lines of elements. The spectrum of deuterium is represented by two atomic lines, at 486.1 nm and a resonant one at 656.2 nm (Balmer series of the hydrogen atom H_{α}). Lithium is represented by a strong resonant atomic line at 670.8 nm and a weak atomic line at 610.4 nm. The intensity of optical spectra is given in relative units, without correction for the spectral sensitivity of the setup. Analysis of optical spectra given in Fig. 7 shows, that only both deuterium lines predominate during deuterium plasma irradiation at

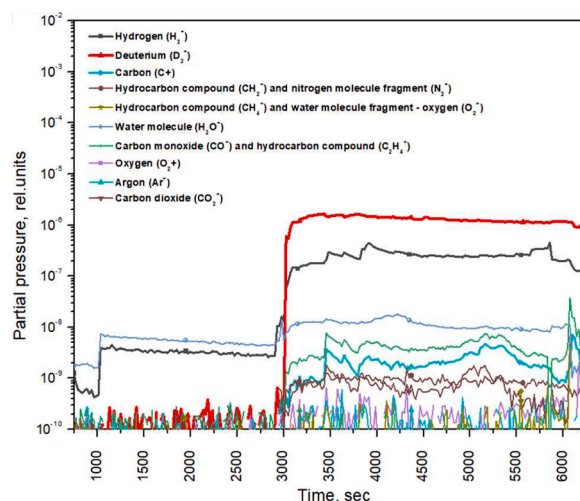


Fig. 6. Time dependence of the gas phase composition in the PBI chamber during irradiation of Sn-Li CPS with deuterium plasma.

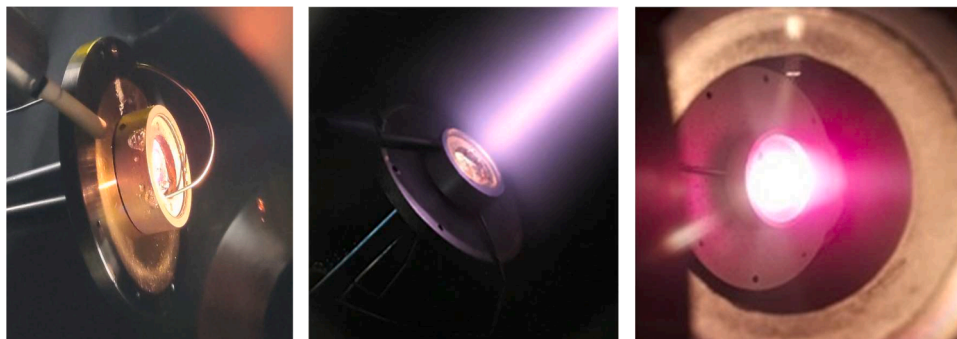


Fig. 5. Irradiation of Sn-Li CPS by deuterium plasma at PBI.

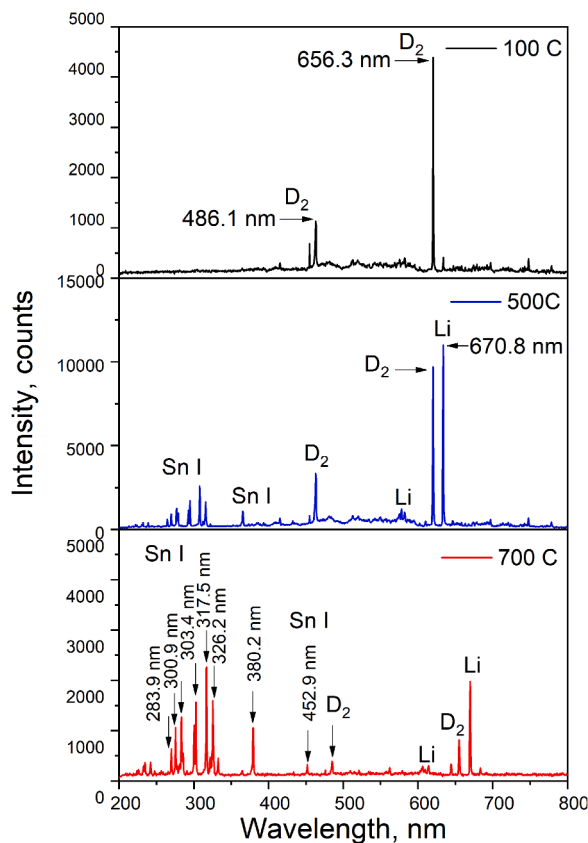


Fig. 7. Optical spectra of the Sn-Li CPS sample under irradiation with deuterium plasma.

low temperatures (100°C), but as the Sn-Li CPS sample's temperature rises, resonant lines for both tin and lithium appears. This fact suggests that the plasma energy deposited on the receiving surface of the target at temperatures above 500°C begins to re-emitted on neutral lithium and tin atoms into optical radiation energy. The obtained data of the optical spectra during plasma irradiation experiment are in good agreement with the data, presented in [39–41].

Similar work to ours has been performed and presented in [42]. In this work argon plasma interaction with liquid lithium, embedded with 304 stainless-steel multichannel capillary porous systems were studied, using linear plasma device. The emission spectrum of the lithium vapor cloud was analyzed in the experiment on multichannel array structures. The authors reported that SEM images of irradiated 304 stainless steel-based EM-CPS samples show corrosion problems due to the interaction between liquid lithium and argon plasma, but still exhibit good plasma-forming characteristics.

Plasma compatibility studies with Sn-Li alloy were carried out on the TJ-II stellarator to investigate tritium retention, hydrogen recycling and plasma compatibility, although they did not provide definitive clarity on these issues [26,43]. Sputtering of Sn-Li alloys has been widely studied during the APEX project [44]. An interesting insight was obtained from laboratory observations. In these experiments alloys with atomic ratios of Li-Sn components between 20/80 and 30/70 were tested. It should be noted here that re-deposition of sputtered metals can cause the changes in the surface composition of the alloy during plasma exposure [45]. Selective deposition of lithium, for example, can produce crystallites with high melting points. Or vice versa, depletion of the lithium surface will eventually lead to a pure tin scenario with a higher plasma contamination potential than lithium.

At the final stage of our experiment there was an uncontrolled growth of the sample temperature above 1500°C, which is most likely this happened due to intense evaporation of the alloy from the target

which resulted in the direct interaction of the plasma and the mesh, and subsequent overheating of this mesh, which led to the dusting of the viewing windows on the PBI and the inability to control the parameters of the experiment. After the experiment was completed, the sample of Sn-Li CPS was removed from the vacuum chamber of the PBI. The appearance of the partially destroyed Sn-Li CPS sample is shown in Fig. 8.

Further post-material science studies with a sample were performed by using microstructure, energy dispersion, thermal and X-ray phase analysis methods.

3.2. Results of post-experimental material science studies

3.2.1. SEM results

The microstructure and surface of Sn-Li CPS were studied using the Tescan Vega 3 scanning electron microscope with X-Act energy-dispersive spectral analysis technique. The surface images of the Sn-Li CPS sample at various zoom levels are shown below (see Figs. 9 and 10).

Further, the Sn-Li CPS sample fragment was investigated by energy dispersive analysis (see Figs. 11 and 12).

A close examination of the surface of the Sn-Li CPS sample an inclusion of bubbles was noticed (Fig. 13), presumably, this is deuterium, embedded as a result of interaction with plasma.

At the next stage of the research, a thermal analysis of a fragment of a Sn-Li CPS sample was conducted.

3.2.2. TGA results

Thermal analysis of a fragment of a Sn-Li CPS sample was performed using the TiGrA experimental complex [46] in which thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) and mass spectrometry (MS) methods are simultaneously combined. The results of the TGA analysis are shown below.

As can be seen from the thermogram in Fig. 14, the melting temperature of the fragment sample was about 221°C, which indicates a decrease in the percentage of lithium in the alloy. This circumstance is most likely due to the fact that in the process of high-temperature interaction of the alloy with plasma lithium is washed out due to its release to the surface and evaporation from the surface of the Sn-Li CPS. Obtained data for melting point of SnLi CPS agrees well with the data obtained in [47,48].

3.2.3. XRD results

At the final stage, the phase analysis of the Sn-Li CPS sample fragment was performed using the Emyrean X-ray diffractometer. The operating mode of the PIXcel1D detector was a scanning line detector. Radiation: Cu K α ; voltage and current: 45 kV, 40 mA. A fixed divergence slit with an angular divergence of 1°, an anti-scattering slit of 2°, and an incident beam mask marked 10, providing an incident beam width of 9.9 mm, were used. The exposure time for the surveys was 91.8 s, the scanning step size for the XRD patterns was 0.026 °2 θ , and the investigated angular range was 5 - 153°2 θ . The surface phase composition of Sn-Li CPS sample was identified using the Crystallography Open Database and the PDF-2 ICDD Release 2004 database [49]. The results of the analysis are shown in Figs. 15 and 16.

The analysis of XRD pattern showed that the basis of the phase composition is tin and lithium phases. The tin phase of tetragonal syngony, spatial group I41/amd, the redistribution of intensities of the tin phase is observed, which indicates the presence of texture on the surface of the sample. Lithium phases were of cubic syngony (spatial group Im-3 m and Fm-3 m). Steel-bearing phases with tin (e.g., Fe_{2.5}O₄Sn_{0.5}) of cubic syngony, spatial group Fd-3 m, are identified as vase of low intensity. Presumably, these are traces of dissolution of the steel shell of the thermocouple. No traces of molybdenum on the surface were found and this indicates that even at high temperatures there is no corrosion interaction between the Sn-Li alloy and molybdenum.

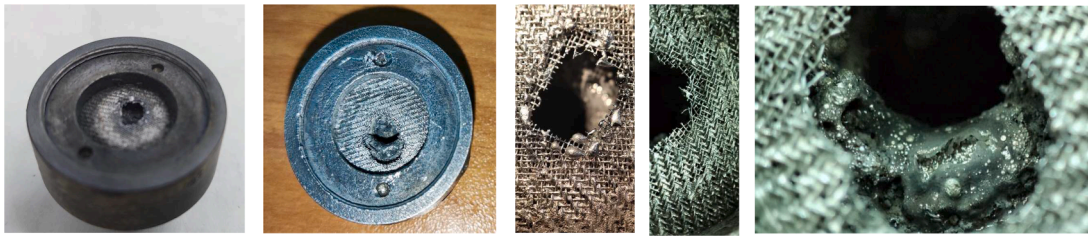


Fig. 8. Appearance of the target (left) and Sn-Li CPS sample after plasma irradiation (right).

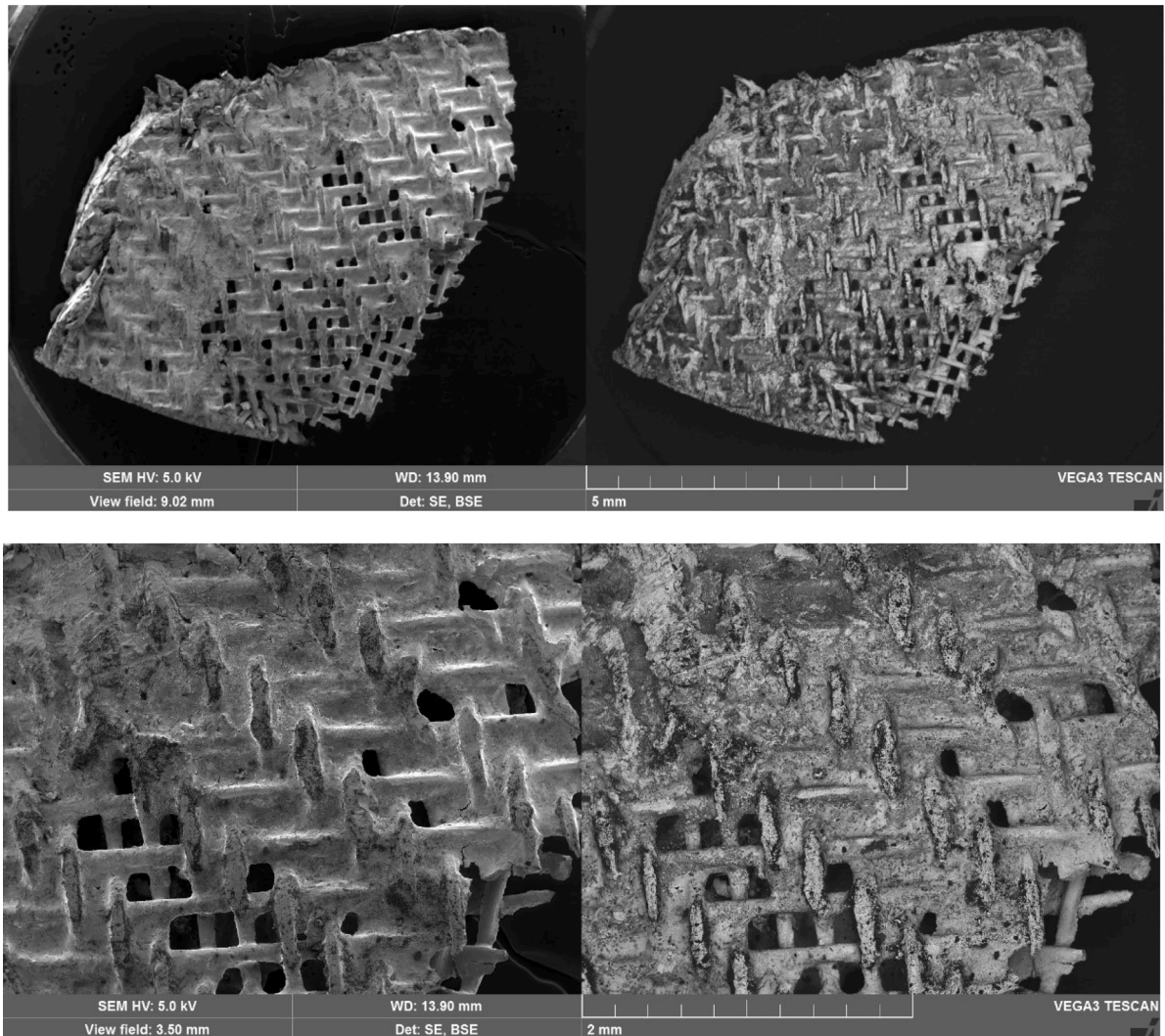


Fig. 9. Surface image of Sn-Li CPS sample at various zoom scales.

4. Conclusion

The paper presents the results of high-temperature tests of tin-lithium CPSs under conditions of deuterium plasma irradiation, carried out at a linear accelerator with a plasma-beam discharge. As shown in our earlier tests utilizing CPS without liquid Sn-Li alloy, the solid metal matrix is destructed under the influence of plasma loads and as a result of temperature rise. According to the findings of this study, we can conclude that Sn-Li CPS effectively removes heat from the CPS matrix and protects it from destruction even at critical increases of deuterium plasma current, which is explained by the heat of melting and vaporizing liquid Sn-Li alloy, and subsequently when the temperature increases due to the re-emission of plasma energy on neutral lithium atoms located on

the surface. Analysis of the obtained mass spectra revealed that, in comparison to pure lithium, the sorption-desorption parameters of deuterium (recycling) by Sn-Li alloy have very low values. Analysis of the optical spectra revealed that whereas the resonant lines of deuterium only dominate during plasma irradiation at low temperatures, tin and lithium's resonant lines starts to predominate as the sample's temperature rises. This observation shows that at temperatures over 500C, the plasma energy deposited on the target's receiving surface starts to re-emit on neutral lithium and tin atoms into optical radiation energy. This findings supports the concept of using liquid-metal lithium alloys as PFM.

However, there is an assumption that, during the high-temperature interaction of the alloy with the plasma, lithium is washed out due to

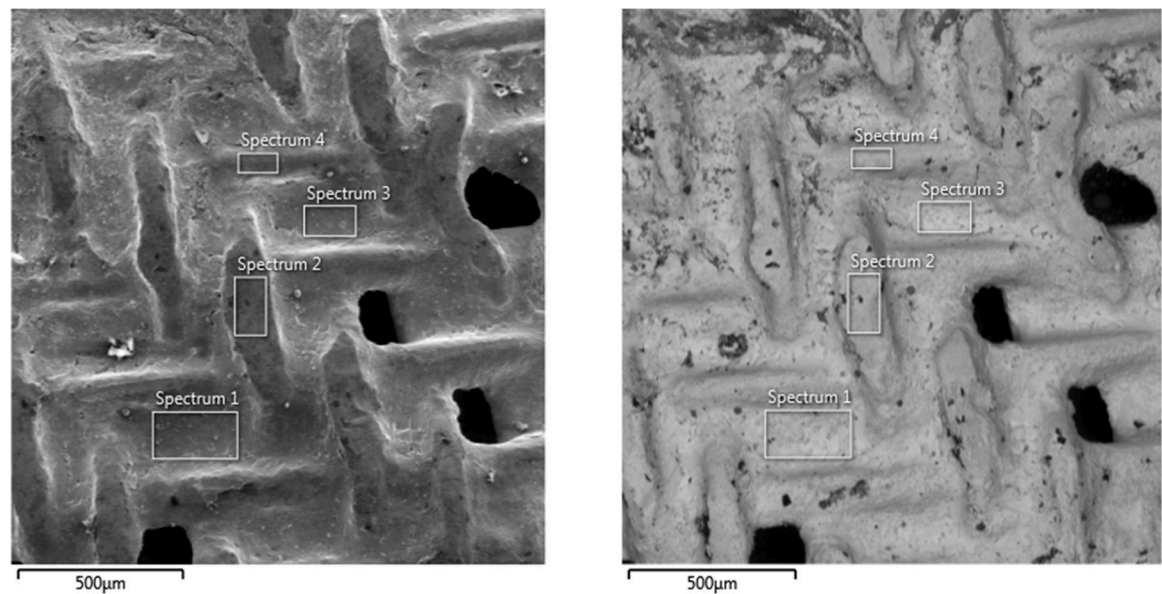


Fig. 10. Enlarged Sn-Li CPS sample (in secondary electron mode (SE) – left, and back-scattering electron (BSE) mode – right) section for further EDS analysis.

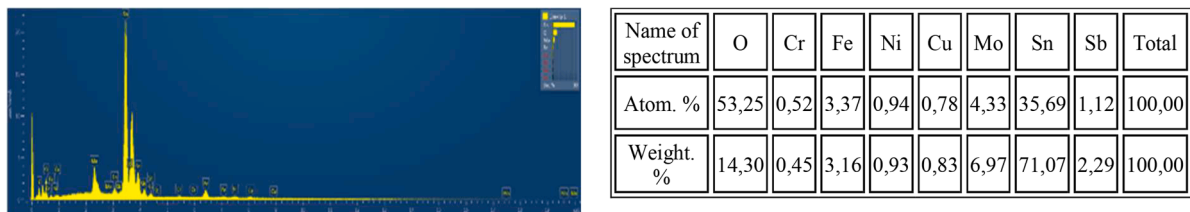


Fig. 11. EDS analysis of section (Spectrum 1).



Fig. 12. EDS analysis of section (Spectrum 2).

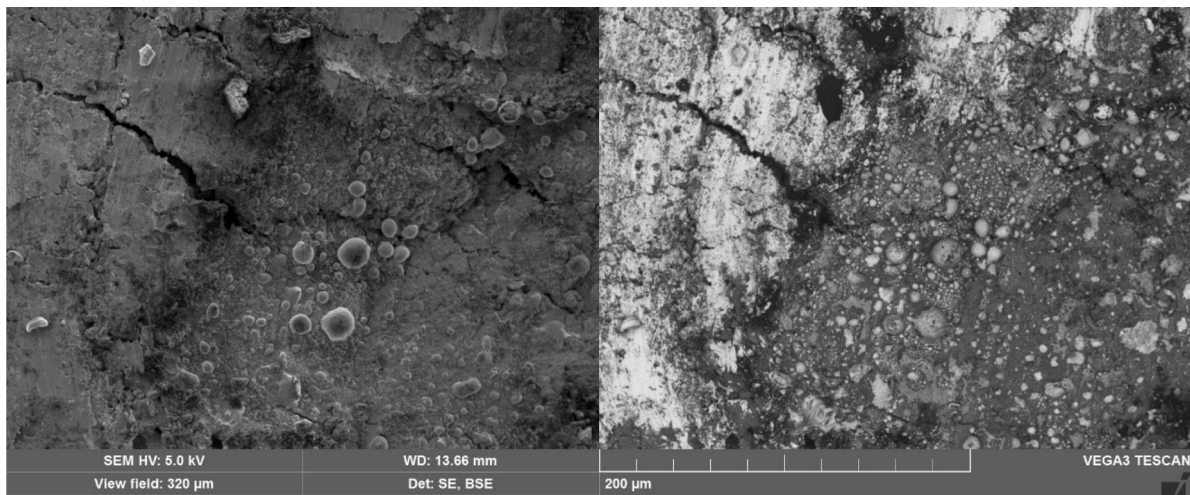


Fig. 13. Magnified section of a fragment microphotograph of a Sn-Li CPS sample.

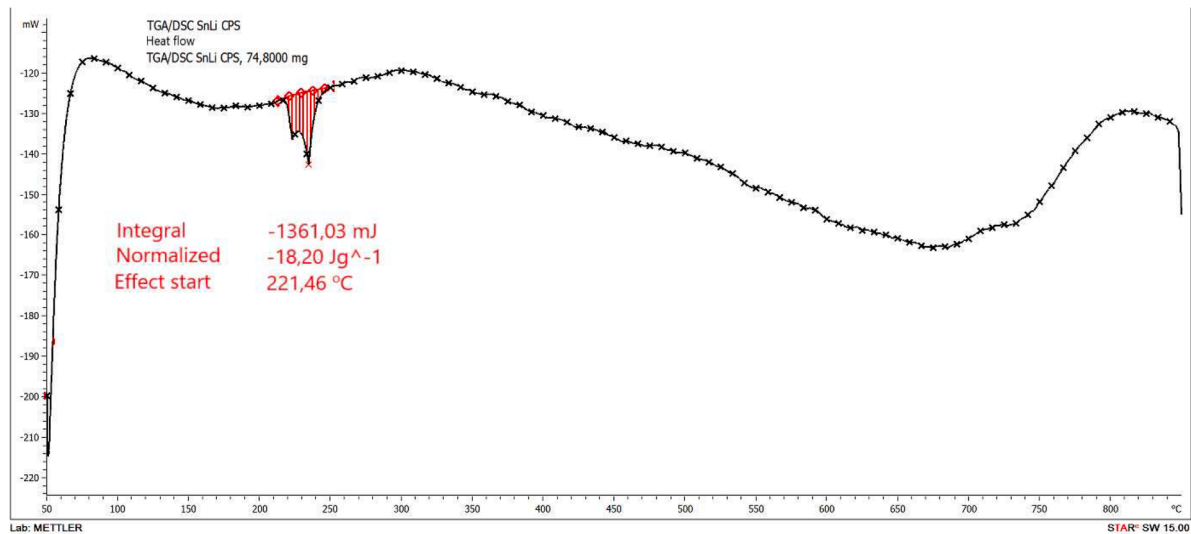


Fig. 14. Thermogram of Sn-Li CPS sample heating at a rate of 10°C /min.

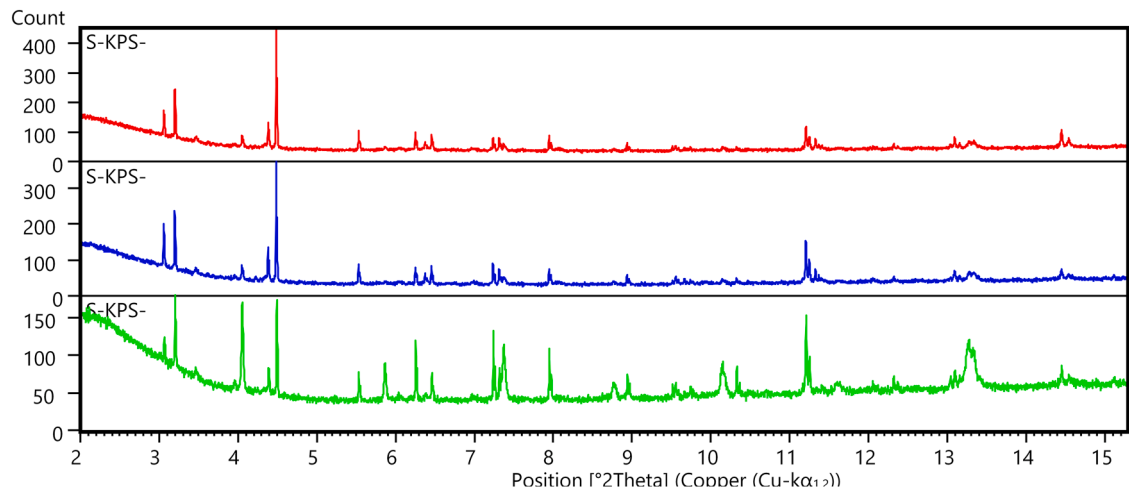


Fig. 15. General view of XRD pattern obtained from the Sn-Li CPS sample.

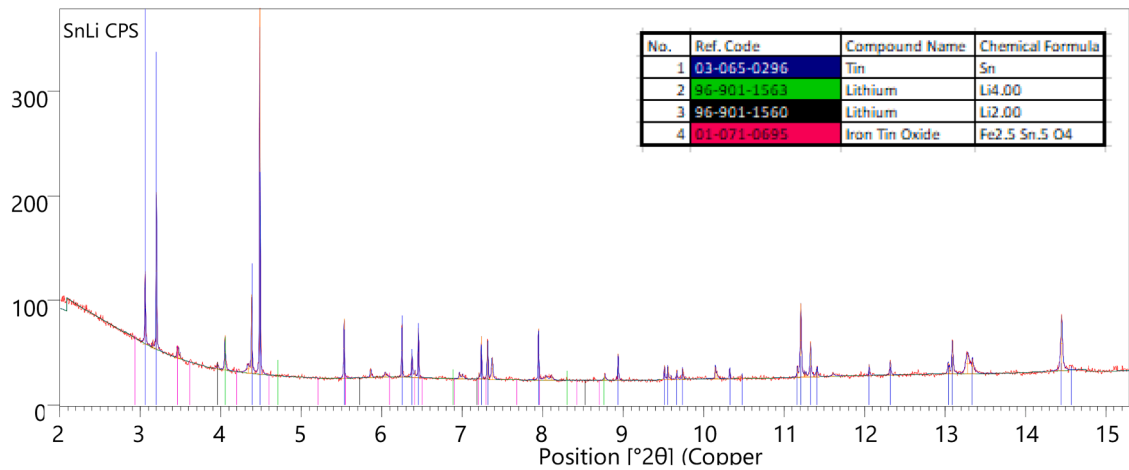


Fig. 16. The result of the analysis of XRD pattern of the Sn-Li CPS sample.

its release to the surface and evaporation from the surface of the Sn-Li CPS. It is assumed that this results in the vaporization of tin, which can lead to the destruction of CPS at the points of contact with the

plasma. In this regard, to use this alloy in a FR, it is necessary to provide a sufficient amount of "fresh" alloy to continuously feed the plasma-contacting surface of the CPS. Accordingly, further research is needed

to qualify the liquid-metal concept for the creation of demonstration models of in-vessel FR elements.

CRedit authorship contribution statement

Yu.V. Ponkratov: Writing – original draft, Methodology, Supervision. **K.K. Samarkhanov:** Investigation, Writing – review & editing, Formal analysis. **V.V. Baklanov:** Project administration. **V.S. Bochkov:** Investigation, Data curation. **I.A. Sokolov:** Investigation, Resources. **A. Zh. Miniyazov:** Investigation, Resources. **T.R. Tulenbergenov:** Investigation, Resources. **I.E. Kenzhina:** Writing – review & editing, Formal analysis. **M.M. Begentayev:** Formal analysis. **Ye.Yu. Tulubayev:** Data curation. **O.S. Bukina:** Visualization, Data curation. **N.A. Orazgaliyev:** Visualization, Data curation. **E. Sapparbek:** Data curation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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