Investigation of the interaction of deuterium with Sn₇₃Li₂₇ tin-lithium alloy

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ABSTRACT

The SnLi alloy has good prospects to be used as a material of intra-chamber elements of fusion facilities, as it has a number of advantages over pure lithium. The results of a series of experiments on deuterium adsorption/desorption by a Sn₇₃Li₂₇ alloy sample are presented. To prepare an alloy sample with the required tin and lithium content, a technique was developed and a special experimental device was constructed. An ampoule device was manufactured to conduct a series of experiments to study sample saturation with deuterium and desorption of deuterium and deuterium-containing molecules from it. Saturation was carried out at alloy temperatures of 650, 600, 550, 500 and 450 °C. TDS experiments were carried out at 20 °C/min. The possible mechanism of deuterium dissolution and release from the tin-lithium alloy was considered and temperature dependences of the effective deuterium solubility constant K_S in the tin-lithium alloy were calculated within the framework of the proposed mechanism. The temperature dependence of the Sieverts' constant for the test sample in the temperature range of 500–650 °C was determined as $K_S(T) = 3.6 \cdot 10^6 \cdot \exp\left(-\frac{32400(J)}{RT}\right) \cdot Pa^{\frac{1}{2}}$.

1. Introduction

Currently, many scientific organizations worldwide are conducting experimental work in the field of fusion energy, focusing on the use of liquid low-melting metals as functional and structural materials in fusion reactors [1–10]. A new concept for a fusion reactor divertor is being developed, proposing the replacement of traditional solid materials with liquid low-melting metals [11–19] due to the limitations of the use of conventional structural materials. Thus, lithium, gallium, and tin are considered as a potential liquid metal components for fusion facilities. Among these, lithium is the most studied, especially regarding its properties relevant to plasma-facing materials and compatibility with structural materials. Although lithium has a number of advantages over other low-melting metals: low nuclear charge $Z = 3$; high specific heat of evaporation; ability to absorb hydrogen isotopes; re-emission of high energies on neutral lithium atoms.

It should be mentioned that there are other, not fully resolved problems associated with the possibility of tritium accumulation in lithium, as well as increased vapor pressure above the liquid lithium.

Recent studies indicate that above 500 °C, lithium undergoes significant evaporation, and subsequently would condense on internal surfaces of the vacuum chamber of plasma-physical facilities [20–23]. Preventing the contamination of the vacuum chamber and all intra-chamber elements, sealed leads and elements of gas-vacuum systems, dusting of diagnostic windows, capture and accumulation of tritium are crucial for ensuring the safety of the reactor and purity of fusion plasma. Therefore, attention has turned to lithium alloys with low-melting metals, characterized by low vaporization parameters and high heat capacity, for example tin-lithium alloy. Liquid tin has a low saturated vapor pressure, making it suitable for protecting the inner elements of fusion reactors from high-density energy and particle fluxes in the Sn-Li system with lithium content ranging from 20 to 30 at.% [24,25]. Figs. 1 and 2 show the temperature dependences of the vapor pressure and thermal conductivity of Li, Sn and Sn-Li alloy [26–31]. The low vapor pressure of Sn-Li raises the upper temperature limit of the alloy's use higher than 500 °C, compared to pure lithium [32–34]. The positive thing is that in the long term, a higher surface temperature of the intra-chamber components may lead to an increase in the coolant temperature, and to

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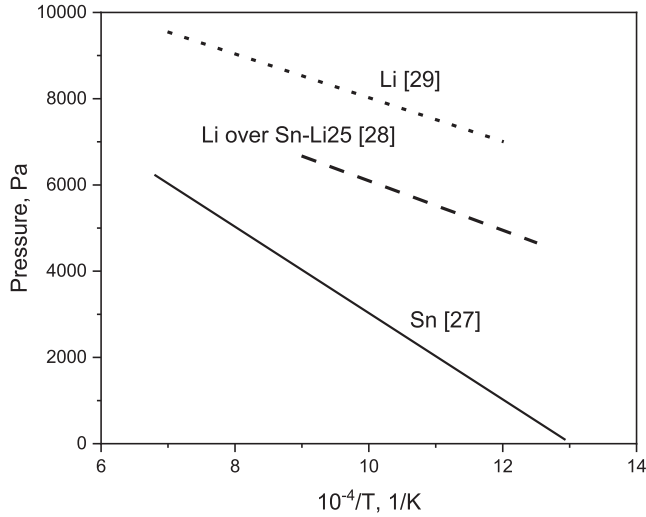


Fig. 1. Temperature dependence of the vapor pressure of Li, Sn and Li-Sn.

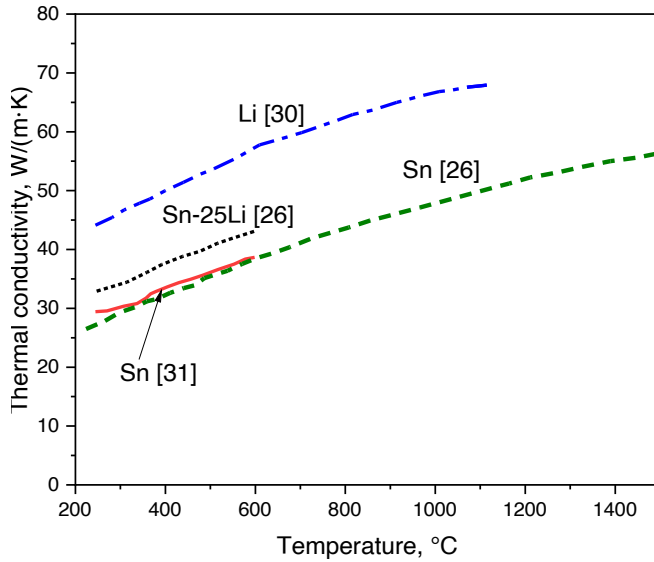


Fig. 2. Temperature dependence of thermal conductivity of Li, Sn and Sn-Li.

improve the energy efficiency parameters of the reactor as a whole. It is also found that in the temperature range of 500–800 °C, the vapor above the Sn-Li alloy mainly contains Li, and the vapor pressure of Sn-Li is at least three orders of magnitude lower than that of pure Li [32].

However, the use of tin (with a higher atomic number Z than lithium) also can cause plasma contamination and its maximum allowable concentration in plasma should not exceed 0.1 % [35]. Corrosion activity of tin and its alloys is very high [36–38] therefore compatibility of SnLi with structural materials should be taken into account. Other important issues are the generation of tritium on lithium nuclei and its accumulation in the alloy during reactor operation, changes in the alloy properties during long-term operation, as well as issues of working gas recirculation. Accordingly, the study of the interaction processes of the Sn-Li alloy with hydrogen isotopes in the range of alloy operating temperatures in fusion installations (about 450–600 °C) is an urgent task of modern research. This paper presents the results of a series of experiments on deuterium adsorption/desorption by a $\text{Sn}_{73}\text{Li}_{27}$ alloy sample.

2. Materials and Methods

2.1. Investigated material

A tin-lithium alloy containing 73 at. % tin and 27 at. % lithium was fabricated for experiments. The criterion for choosing a composition with such a ratio was that this alloy (with a melting point of about 326 °C according to [39]) may be optimal in terms of improving its thermophysical properties, reducing the saturated vapor pressure in the liquid phase and the parameters of lithium depletion in the alloy during its operation at temperatures above 500 °C.

In order to obtain a tin-lithium alloy with a chosen percentage, a preliminary calculation of the amount of starting material was required. The calculation of the mass of the necessary components was carried out using the formula for determining mass fractions:

$$\omega(\text{Sn}) = \frac{n_{\text{Sn}} \bullet A_r(\text{Sn})}{n_{\text{Sn}} \bullet A_r(\text{Sn}) + n_{\text{Li}} \bullet A_r(\text{Li})} \quad (1)$$

$$\omega(\text{Li}) = \frac{n_{\text{Li}} \bullet A_r(\text{Li})}{n_{\text{Sn}} \bullet A_r(\text{Sn}) + n_{\text{Li}} \bullet A_r(\text{Li})} \quad (2)$$

where ω is the mass fraction of the element in the alloy;

A_r is the relative atomic mass of the element,

n is the number of atoms.

For the alloy fabrication, lithium of LE-1 and tin of OVCh grades were used. The composition of metals is presented in Tables 1 and 2.

2.2. Test sample preparation

The $\text{Sn}_{73}\text{Li}_{27}$ alloy was prepared using a special experimental device shown in Fig. 3.

The device consisted of a capsule (1) made of 12Cr18Ni10T stainless steel, with a crucible (2) for lithium loading at the bottom of the capsule. To load tin into the device, a second crucible (3) of smaller diameter was provided, lowered to the bottom of the device using the vacuum feed-through of linear displacement. Both crucibles were made of MCH grade molybdenum, the small crucible had a perforated bottom. The melting of metals was achieved by means of an ohmic heater (4) mounted on the bottom of the device. In order to reduce heat losses of the heated part of the device there was a cover filled with mineral insulating material (5). The melt temperature was controlled by a Ch-Al-type thermocouple (6), tightly mounted on the bottom of the device. Vacuum pumping and subsequent supply of noble gas to the device was provided through a connector with a valve (7) located in the upper part of the device. The fabrication of tin-lithium alloy $\text{Sn}_{73}\text{Li}_{27}$ was performed at the experimental facility [40]. The alloy preparation scheme is shown in Fig. 4.

The manufacturing procedures were as follows. Initially, the volume of the device was pumped out by means of oil-free forevacuum pumping to a pressure of no more than 10 Pa. Then, using an ohmic heater, a large crucible with purified lithium was heated to a temperature of 350 °C and maintained for 30 min under continuous pumping conditions. Next, the pumping system was switched to a high-vacuum turbomolecular pump and the device was pumped out to a pressure of no more than 10^{-3} Pa. In this mode, lithium was annealed for another 1 h. After this procedure, high-purity helium was introduced into the volume of the device to a pressure of about $5 \cdot 10^3$ Pa, and, using a sealed lead-in of motion, a small crucible with a perforated bottom and pure tin loaded into it, was immersed in liquid lithium and maintained in this position for 0.5 h.

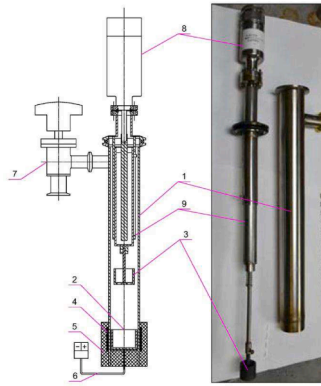
After heating the mixture of liquid metals to 700 °C, the small crucible was brought to the upper position and lowered to the bottom of the device about 7–8 times to mix the alloy. In this mode (in an inert environment, the small crucible in the upper position), to achieve the best homogeneity of the alloy, the system was maintained for 24 h. After the heating stopped, the manufactured alloy cooled naturally. A fragment of the tin-lithium alloy sample is shown in Fig. 5. The total mass of the test sample of $\text{Sn}_{73}\text{Li}_{27}$ alloy was about 6 g.

Table 1
Composition of Lithium (LE-1 Grade) (in mass fraction).

Fe	Mn	N	Al	Ca	Mg	Na	Li
up to 0,005	up to 0,001	up to 0,050	up to 0,003	up to 0,030	up to 0,020	up to 0,040	up to 99,90

Table 2
Composition of Tin (OVCh Grade) (in mass fraction).

Sn	As	Fe	Cu	Pb	Bi	Sb	Zn	Al	Ga	Ag	Au	Co	Ni	In
99,999	$1 \cdot 10^{-4}$	$1 \cdot 10^{-4}$	$1 \cdot 10^{-5}$	$1 \cdot 10^{-5}$	$5 \cdot 10^{-6}$	$5 \cdot 10^{-5}$	$3 \cdot 10^{-5}$	$1 \cdot 10^{-4}$	$5 \cdot 10^{-5}$	$5 \cdot 10^{-6}$	$1 \cdot 10^{-5}$	$1 \cdot 10^{-5}$	$1 \cdot 10^{-6}$	$1 \cdot 10^{-5}$



1 – housing; 2 – large crucible; 3 – small crucible with holes on the bottom; 4 – heater; 5 – mineral insulation; 6 – thermocouple; 7 – vacuum valve; 8 – feedthrough of linear displacement; 9 – vacuum part of feedthrough

Fig. 3. Device for $\text{Sn}_{73}\text{Li}_{27}$ alloy fabrication.

2.3. Characterization of the test sample of $\text{Sn}_{73}\text{Li}_{27}$

After the alloy was manufactured, it was characterized using electron microscopy, EDS analysis, X-ray phase analysis, and differential scanning calorimetry. The results of the characterization are shown in the Figs. 6-9 [37].

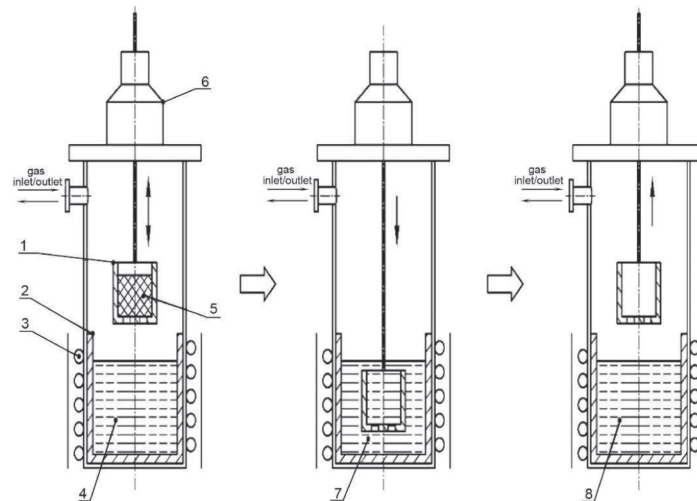
The XRD analysis was performed using an Panalytical “Empyrean” diffractometer with a PIXcel1D detector, whose sensitivity range starts at 12 a.m.u. (carbon), and therefore it was not possible to detect lithium

in its pure form. The diffraction patterns clearly showed peaks of the β -Sn phase. The precise identification of these peaks is challenging due to their low intensity (approximately 30 times weaker than the β -tin peaks). However, the candidate phases include those containing (Sn, Li), specifically Li_2Sn_5 (tetragonal symmetry, space group $P4/\text{mbm}$) and Li_5Sn_2 (rhombohedral symmetry, space group $R\text{-}3\text{m}$). While lithium is observed in the alloys, the precise determination of the lithium-to-tin ratio is challenging due to the highly structured surface.

Data analysis revealed that the quantitative content of the primary elements in the alloy is tin and oxygen, 72.47 % and 19.64 % by mass, respectively (Fig. 7). Carbon was also detected at 7.69 % by mass, along



Fig. 5. $\text{Sn}_{73}\text{Li}_{27}$ alloy fragment.



1 – small crucible; 2 – large crucible; 3 – heater; 4 – liquid lithium; 5 – tin; 6 – sealed lead-in of motion; 7 – liquid Li + Sn; 8 – $\text{Sn}_{73}\text{Li}_{27}$ alloy

Fig. 4. $\text{Sn}_{73}\text{Li}_{27}$ alloy preparation scheme [41].

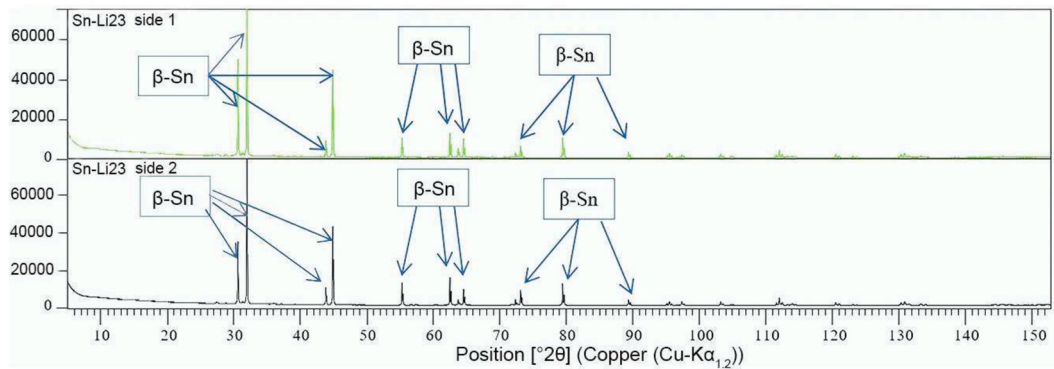
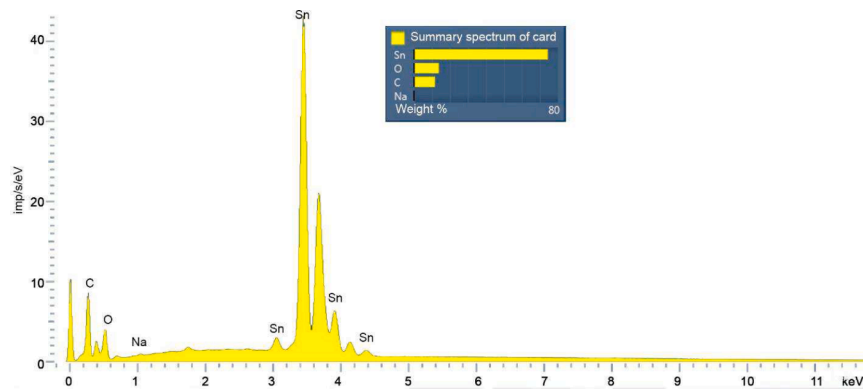


Fig. 6. Alloy diffraction pattern.



Spectrum title	C	O	Na	Sn	Sum
Total spectrum, weight %	7.69	19.64	0.20	72.47	100.00

Fig. 7. Summary spectrum of the EDS map.

with a small amount of sodium (0.20 %), which is likely related to impurities in lithium. It should be noted that elemental composition analysis starts from the element beryllium (Be), which is why lithium does not appear in the results.

The microstructure of the $\text{Sn}_{73}\text{Li}_{23}$ alloy sample surface was carried out using a Tescan Vega 3 scanning electron microscope in secondary electron (SE) and back-scattered electron (BSE) modes. Elemental mapping of the sample was performed with an Oxford Inca X-Act energy-dispersive spectrometer. From the microstructural image and elemental EDS mapping of the $\text{Sn}_{73}\text{Li}_{23}$ sample (Fig. 8), it is clear that the alloy is characterized by the predominance of pure tin and the presence of a needle-like structure, clearly distinguishable in both topographic contrast (SE) and compositional contrast (BSE) modes. This structure is presumed to correspond to the Li_2Sn_5 phase, which actively interacts with atmospheric gases, particularly oxygen and carbon dioxide. Such reactions result in the formation of lithium oxide (Li_2O) and lithium carbonate (Li_2CO_3), leading to increased carbon and oxygen content in this region.

Differential scanning calorimetry (DSC) studies were conducted to determine the melting temperature and enthalpy of the $\text{Sn}_{73}\text{Li}_{23}$ alloy. The sample was heated from 100 °C to 600 °C at a linear rate of 10 °C/min. Analysis of the heat flux curve (Fig. 9) obtained from the $\text{Sn}_{73}\text{Li}_{23}$ sample revealed a melting point of 228.87 °C and an enthalpy of 34.65 J/g, suggesting the presence of the Li_2Sn_5 phase in the alloy. This DSC curve aligns well with previous data [37] and corresponds to the binary Sn-Li phase diagram [25].

2.4. Experimental setup and Methodology

The experimental setup [42] consisted of a vacuum post, quadrupole mass spectrometer RGA-100, pressure sensors, and vacuum fittings, as shown in Fig. 10.

Before conducting a series of experiments, a tin-lithium alloy was loaded into the experimental device shown in Fig. 11. Then the device was evacuated to a pressure of no more than 10^{-3} Pa, and, using a heater, the sample was annealed at a temperature of 700 °C for 1 h under continuous vacuum evacuation.

During the development of the experimental technique, a sample heating-cooling procedure was carried out, as a result of which the error between the thermocouple readings and the actual temperature of the alloy was determined by the crystallization temperature of the alloy, which was no more than 3–5 °C.

The adsorption method and the method of Thermal Desorption Spectrometry (TDS) were used for experiments with $\text{Sn}_{73}\text{Li}_{27}$ sample. The essence of the adsorption method was as follows. After pumping out the ampoule with the test sample to a pressure of no more than 10^{-3} Pa, a known amount of deuterium was introduced and the sample was saturated from the gas phase at the chosen temperature. Throughout the experiment, a continuous recording of the gas pressure change in the ampoule was maintained. The adsorption method was used to study the patterns of deuterium absorption by the liquid tin-lithium alloy at various temperatures using an absolute pressure sensor.

The TDS method involved measuring the gas flows released from the tin-lithium alloy into the evacuated chamber under conditions of linear

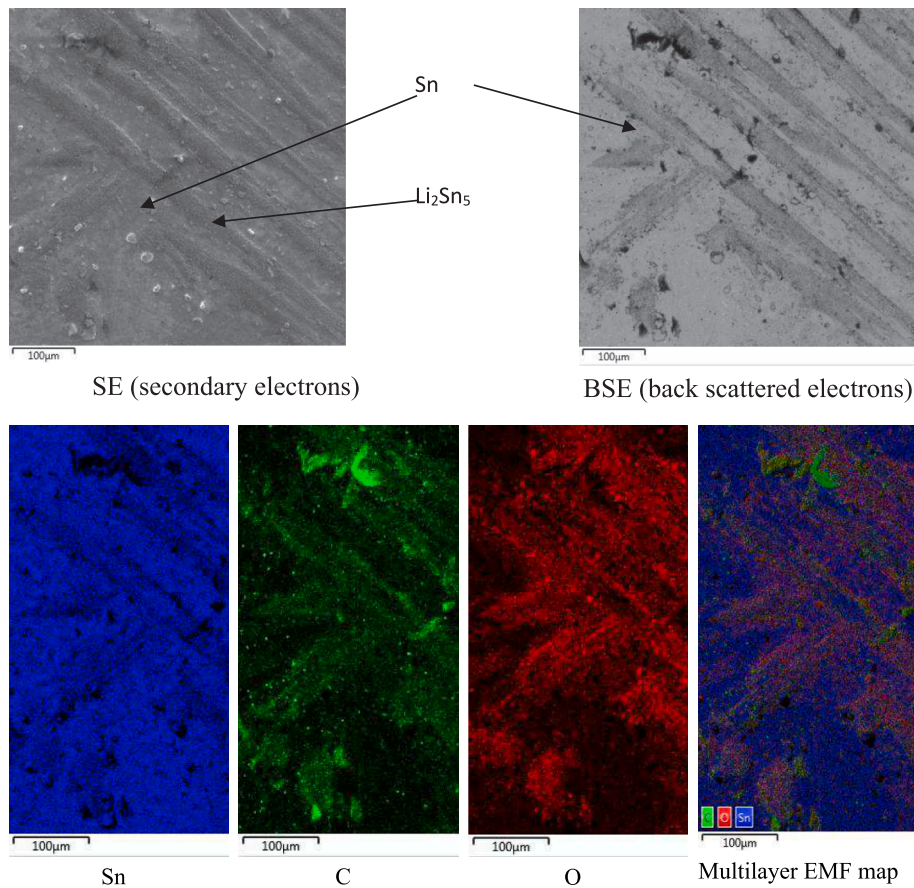


Fig. 8. Micrographs and elemental EMF mapping of the $\text{Sn}_{73}\text{Li}_{27}$ sample.

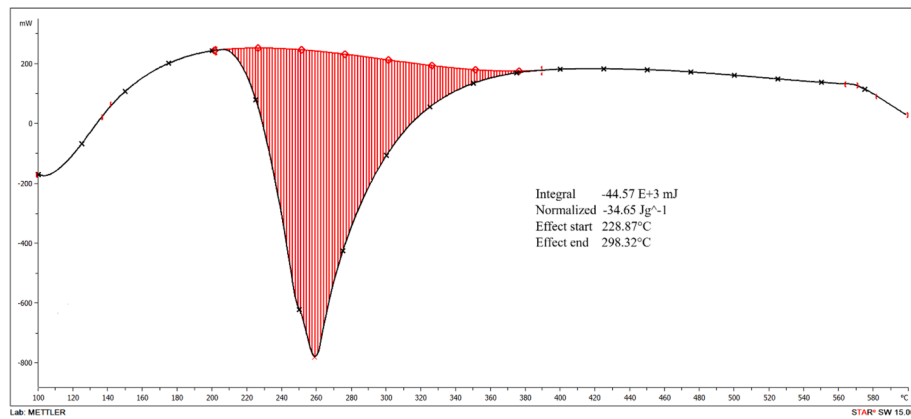


Fig. 9. Typical thermogram of the $\text{Sn}_{73}\text{Li}_{27}$ alloy.

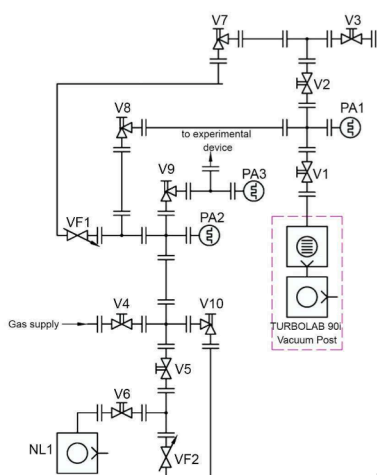
heating. This method was applied to obtain time dependencies of the release of deuterium and deuterium-containing molecules from the $\text{Sn}_{73}\text{Li}_{27}$ alloy as a function of the sample temperature using the RGA-100 QMS.

Experiments on deuterium sorption/desorption by $\text{Sn}_{73}\text{Li}_{27}$ alloy were conducted on the same sample. The device shown in Fig. 11 was designed in such a way that the sample was located directly on its bottom and sample replacement was impossible. In order to extract the sample for post-experimental studies to determine the degree of lithium depletion in the alloy, it would be necessary to completely destroy the experimental device. However, as the authors believe, such experimental conditions are close to the operating modes of $\text{Sn}_{73}\text{Li}_{27}$ in a fusion reactor, where the alloy operates without replacement for many

heating-cooling cycles and without opening to the atmosphere. The authors would like to note that in these experiments, under conditions of indirect heating and a relatively small number of cycles, changes in the sample structure were negligible and will insignificantly affect the change in its sorption properties.

3. Results of series of sorption/desorption experiments

The experiments with the test sample were carried out in series. The series was carried out in the saturation temperature (T_{sat}) range of 450–650 °C, which is close to the operating modes of the alloy in the fusion reactor, without critical loads arising during plasma disruptions and ELMS; a similar conditions of TDS experiments were performed in



NL1 – fore vacuum pump; VP1...VP3 – manual valve KF 40; VP4...VP6 – manual valve KF 16; VP7 – manual angle valve KF 25; VP8...VP10 – manual angle valve KF 16; PA1...PA3 – absolute pressure sensor; VF1...VF2 – leak valve; RGA-100 – residual gas analyzer (QMS)

Fig. 10. The diagram of the experimental facility based on a portable vacuum post.

[16]. The sample was saturated at a chosen temperature, then followed by a thermal desorption experiment. T_{sat} changed from higher (650 °C) to lower (450 °C), with an interval of 50 °C. The temperature reduction mode was chosen due to the features of the system of temperature registration and control, which had some response time delay.

The conditions, modes and summarized data of the adsorption and desorption experiments are presented in Table 3 and Figs. 12 – 17. Fig. 12 shows the time dependences of the change in deuterium pressure during saturation of the $\text{Sn}_{73}\text{Li}_{27}$ sample. Figs. 13–17 show the temperature dependences of the change in partial pressures of deuterium and deuterium-containing molecules recorded in the chamber during linear heating of the saturated $\text{Sn}_{73}\text{Li}_{27}$ sample. Recording was carried out in real time using an RGA-100 quadrupole mass spectrometer (Stanford Research Systems) pre-calibrated using pure gases (H_2 , D_2 and He).

It is worth noting some features of the experiments:

- in the 2nd experiment the TDS peak of D_2 was observed after heating on a plateau of 875 °C for 500 s;
- in the 3rd experiment the TDS peak of D_2 was observed on the plateau $\sim$ 890 °C.
- in the 4th experiment saturation ceased after about 2000 s.
- in the 5th experiment saturation ceased after approximately 1200 s. The TDS peak of D_2 was observed after heating on a plateau of 855 °C for 250 s.

4. Discussion

4.1. Deuterium saturation kinetics of the $\text{Sn}_{73}\text{Li}_{27}$ sample

As can be seen from the Fig. 11 the kinetics of deuterium sorption had a similar form for saturation at temperatures of 650, 600 and 550 °C. At temperatures of 500 and 450 °C the saturation rate reached a quasi-equilibrium level of stable pressure reduction faster than at higher

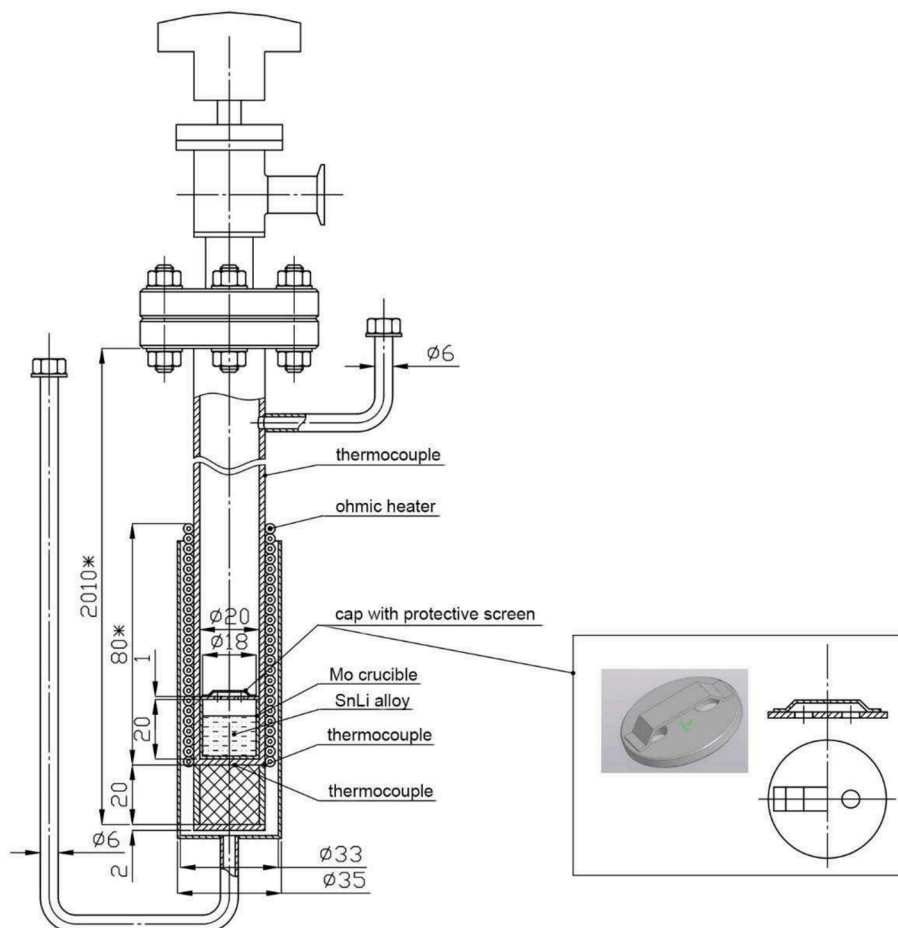


Fig. 11. Experimental ampoule device for study deuterium adsorption/sorption processes of $\text{Sn}_{73}\text{Li}_{27}$ alloy [41].

Table 3
Parameters of adsorption/desorption experiments.

Exp. No	Adsorption			Desorption		
	Saturation temperature, °C	D ₂ pressure, Pa	Saturation time, s	Heating rate, °C/min	T of D ₂ release start, °C	Temperature of the maximum peak of D ₂ release, °C
1	650	$136.7 \cdot 10^2$	3300	20	300–350	845
2	600	$133 \cdot 10^2$	5200		350	875
3	550	$137 \cdot 10^2$	8600		500–550	890
4	500	$136.9 \cdot 10^2$	5300		500	600–875
5	450	$28.9 \cdot 10^2$	2900		500	855

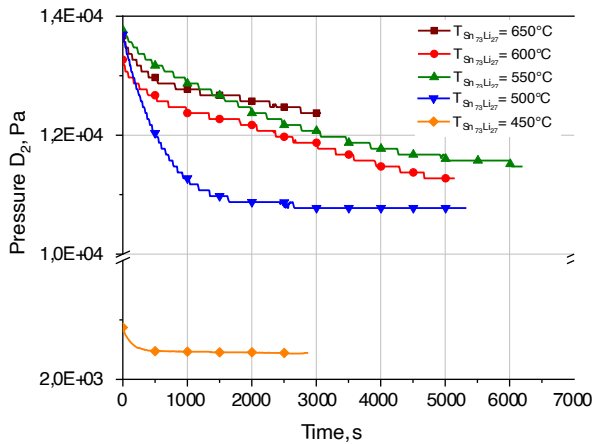


Fig. 12. Results of adsorption experiments with Sn₇₃Li₂₇ sample.

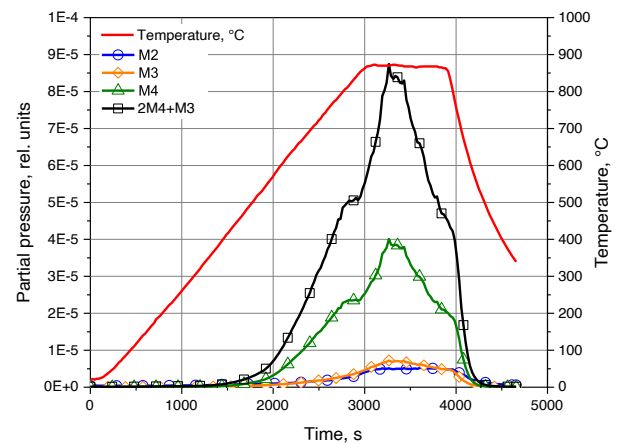


Fig. 14. Results of TDS experiment No 2.

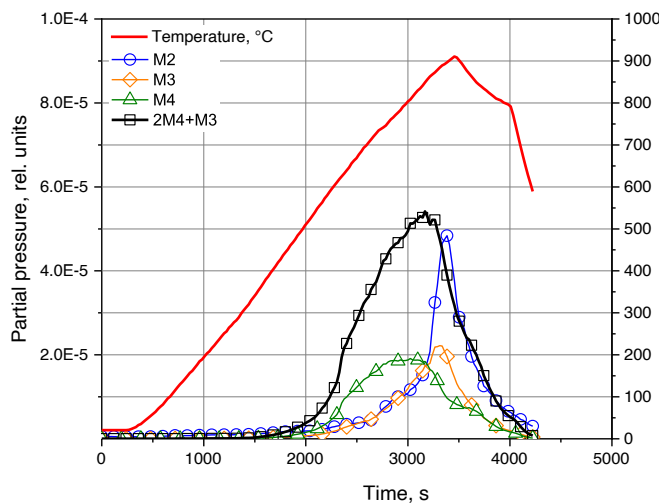


Fig. 13. Results of TDS experiment No 1.

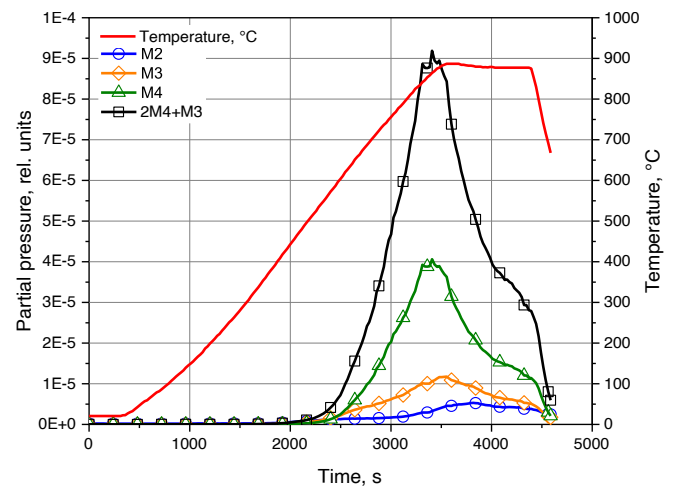


Fig. 15. Results of TDS experiment No 3.

temperatures. This may be due to a significant change in the parameters of the deuterium dissolution rate in the alloy, as well as the rates of formation/decomposition of deuteride phases.

The saturation experiment at 500 °C differed significantly from the first three: the equilibrium state was reached quickly. This was possibly due to the complex relationship between the saturation rate and the dissolution rate of mobile deuterium atoms (which in turn depends on the processes of interaction of these atoms with traps in the alloy).

The last cycle of experiments at the lowest temperature of 450 °C was carried out at a significantly lower pressure and a short saturation time. The lower saturation pressure of deuterium was due to the fact that deuterium for this experiment underwent additional purification (through a palladium-silver filter), which greatly affected the time it

took for deuterium to enter the buffer tank and did not allow the pressure values to be reached as in previous experiments.

4.2. Deuterium release in TDS experiments

Unlike the experiments on deuterium sorption by the sample, that share a similar general trend, a significant difference was noted in the series of desorption experiments.

Let us consider the TDS experiment No 1 (saturation at 650 °C (Figs. 13 and 18)). Unlike other experiments, here a significant release of hydrogen (M2 on Figs. 13–17) was observed, which occurred mainly when heated above 700 °C. Such hydrogen release is most likely due to the fact that some hydrogen was present in the sample before the sorption and was not released as a result of preliminary vacuum

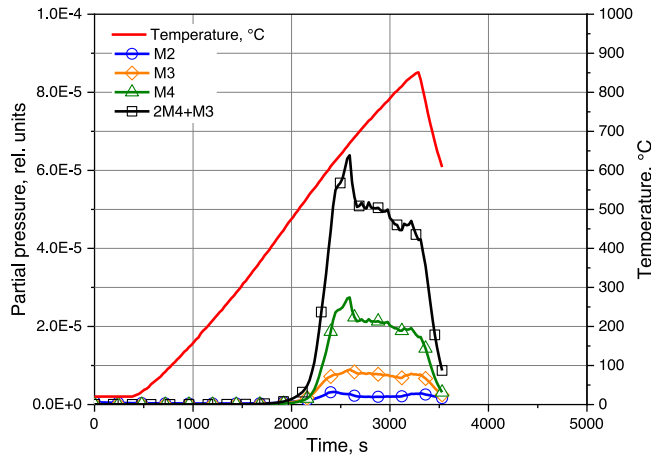


Fig. 16. Results of TDS experiment No 4.

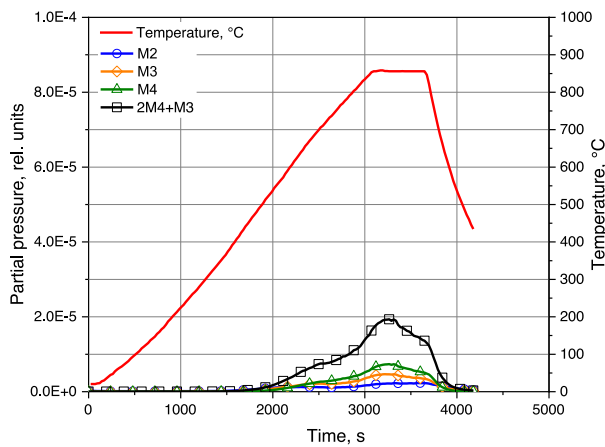
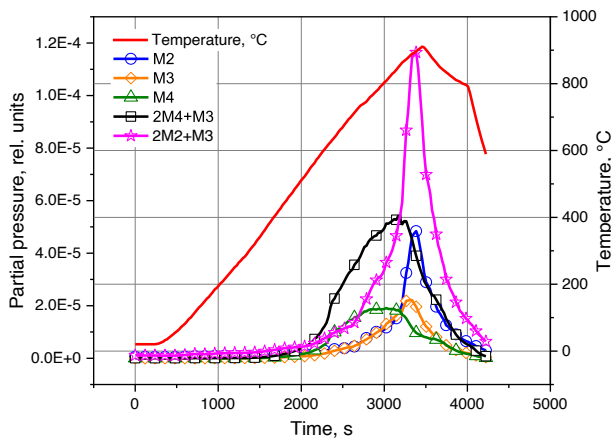


Fig. 17. Results of TDS experiment No 5.

Fig. 18. Change in the release of hydrogen and deuterium-containing molecules in the first TDS experiment ($T_{sat} = 650$ °C).

annealing of the sample (which was carried out before the saturation). This circumstance significantly affected the proportion of D in the sample: the amount of deuterium released in the form of the HD molecule was about 28 % of its total release and the amount of deuterium released in the form of the D_2 molecule was 72 %, respectively. The peaks corresponding to the desorption of hydrogen (M2) and to the desorption of deuterium (evaluated as $2D_2 + HD$) occurred at different

temperatures: the hydrogen peak was observed at 892 °C and the deuterium peak – at 840 °C. This can be explained by the fact that either hydrogen was in the alloy in a trapping site, presumably in the form of lithium hydride, and deuterium was in a mobile state. Or, if both deuterium and hydrogen were in bound states, then the decomposition of lithium deuteride occurred at a lower temperature than lithium hydride. However, the authors believe that it is more likely that deuterium was mainly in a mobile state in the alloy, since the peak of deuterium release was significantly broader than the hydrogen peak.

It should be noted, that in the other experiments the release of hydrogen was significantly lower, and the contribution to the total release of deuterium due to the HD molecule, according to calculations, was about 7–13 % (for different experiments). In addition, the HD and D_2 release shared similar shapes for experiments 2 to 5 (Figs. 13–17). Further analysis of the TDS dependences obtained in the experiment was carried out on the basis of possible mechanisms of deuterium interaction with a sample of tin-lithium alloy.

The authors assume that there is a single deuterium trapping site in tin-lithium alloy, possibly lithium deuteride [43]. At lower temperatures, hydrogen isotopes tend to be in trapping sites, and they are released at higher temperatures.

At the qualitative level, the obtained series of experiments can be explained as shown in Fig. 19.

The rate of onset of the quasi-equilibrium state at saturation is characterized by stabilization of the pressure in the chamber, and accordingly, the achievement of quasi-equilibrium concentrations of mobile C_m and trapped in the LiD C_t deuterium atoms. It apparently depends significantly on a large number of factors: sample temperature, deuterium pressure above the sample, deuterium solubility parameters in the sample ($K_S(T)$ – Sievert's constant), capture constant ($K_{cap}(T)$), release of deuterium from trapping sites in the sample ($K_{rel}(T)$), which have an Arrhenius dependence. For the equilibrium state inside the alloy, the following expression is valid [44,45]:

$$C_m(T) \cdot K_{cap}(T) = C_t(T) \cdot K_{rel}(T) \quad (3)$$

$$C_m(T) + C_t(T) = \frac{p_2^{\frac{1}{2}}}{K_S(T)} \quad (4)$$

It should be noted here that due to the fact that the process of deuterium dissolution is complex and depends not only on the processes of dissolution and release at the boundary of the alloy and the gas phase, but also on the processes of its capture and release from traps in the volume, the Sieverts constant is estimated taking into account all the deuterium absorbed by the sample (which is in a free and captured state). In this regard, it is more correct to call the calculated constant the effective Sieverts constant.

At the highest saturation temperature of 650 °C, deuterium dissolved in the alloy was mainly in a mobile state. At a lower saturation temperature of 600 °C, deuterium was present in both dissolved form and trapping sites in the sample. Therefore, the desorption peak was a superposition of the peak caused by the diffusion of deuterium atoms and the peak caused by the transfer of deuterium released from trapping sites (due to activation mechanism during heating). The initial stage of deuterium release coincides in shape with the TDS curve at $T_{sat} = 650$ °C. At an even lower saturation temperature of 550 °C, deuterium was mainly in trapping sites in the sample. Accordingly, the release peak during heating is largely determined by the release of deuterium from the trapping sites, having a symmetrical shape and shifted to the right on the temperature scale (compared to the diffusion peak). This was also evidenced by the significant shift of the beginning of deuterium release towards high temperatures.

In the experiment on thermal desorption of a sample saturated at 500 °C, the beginning of deuterium release was also shifted towards higher temperatures. The peak of release differed significantly from the previous ones since the sample contained a significantly smaller amount

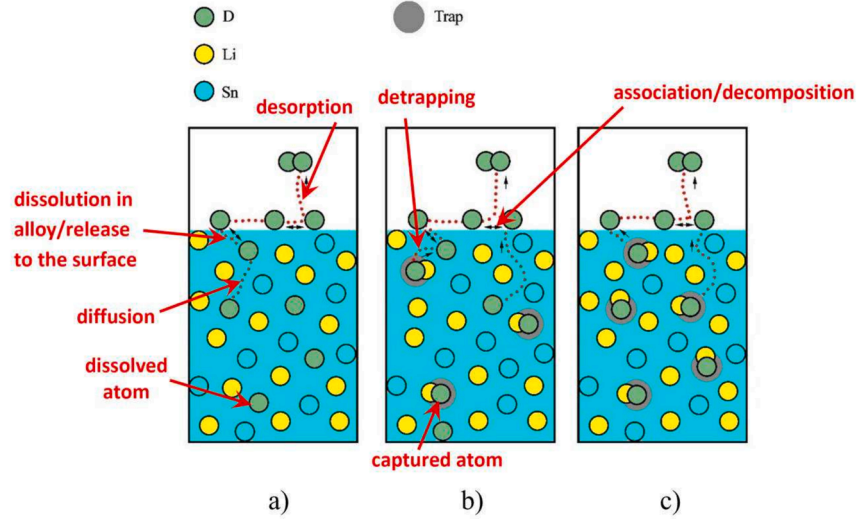


Fig. 19. Diffusion mechanism of deuterium release from the sample in the presence of sources of deuterium detrapping: a) – all D is mobile (dissolved); b) – mix of mobile + trapped D; c) – all D is trapped.

of deuterium after saturation. Deuterium desorption from the sample saturated at 450 °C looked like a superposition of two deuterium release peaks from two different bound states, which may presumably indicate deuterium was in two types of trapping sites.

4.3. Calculation of the Sieverts' constant for deuterium in a tin-lithium alloy

The Sieverts' constant $K_S(T)$ at the saturation temperature $T_{sat}(K)$ was calculated using the formula:

$$K_S(T) = \frac{\sqrt{P}}{X_D}, \quad (5)$$

where P is the pressure of deuterium in the gas phase at saturation, Pa; X_D is the molar fraction of saturated deuterium in the alloy.

The calculation was based on both sorption and desorption experiments. The integral amount of absorbed (released) deuterium was determined for each saturation temperature. The amount of absorbed deuterium $Q_{ads}(T)$ obtained from adsorption experiments was determined by the change in pressure in the saturation chamber according to equation:

$$Q_{ads}(T) = \Delta P \cdot \frac{V_c}{R \cdot T_{room}}, \quad (6)$$

where ΔP is the change in pressure in the saturation chamber, Pa; V_c is the volume of the saturation chamber; $R = 8.31 \text{ J}/(\text{mole} \cdot \text{K})$ – universal gas constant; T_{room} – room temperature ($\sim 293 \text{ K}$). The area of the heated region of the saturation chamber was more than 100 times smaller than the area of the saturation chamber itself, so no corrections were made for the average gas temperature in the saturation chamber.

The amount of deuterium $Q_{des}(T)$ obtained from desorption experiments was determined by integrating the area under the experimental curve of deuterium atom ($2 \cdot D_2 + HD$) release, after that it was multiplied by the calibration coefficients and divided by 2 to bring the total number of deuterium atoms to the amount of D_2 gas. The calculation results summarized in Table 4.

As seen in the Table 4, there is a good match between the solubility values calculated from different experiments. Fig. 20 shows the temperature dependence of the Sieverts' constant for the test sample of $Sn_{73}Li_{27}$ in comparison with a number of literature data for tin-lithium alloys. It is clear that the Sieverts' constant for deuterium in the $Sn_{73}Li_{27}$ alloy agrees well with the calculated values for hydrogen in the $Sn_{70}Li_{30}$. According to the calculated estimates given in [46], the values

Table 4

The calculated amount of deuterium $Q(T)$ obtained from absorption/desorption experiments.

Exp. No	1	2	3	4	5
$T_{sat}, ^\circ\text{C}$	650	600	550	500	450
$Q_{ads}(T), \text{mole}$	$7.38 \cdot 10^{-5}$	$1.13 \cdot 10^{-4}$	$1.28 \cdot 10^{-4}$	$1.66 \cdot 10^{-4}$	$2.49 \cdot 10^{-5}$
$Q_{des}(T), \text{mole}$	$6.99 \cdot 10^{-5}$	$1.20 \cdot 10^{-4}$	$1.15 \cdot 10^{-4}$	$1.46 \cdot 10^{-4}$	$2.41 \cdot 10^{-5}$

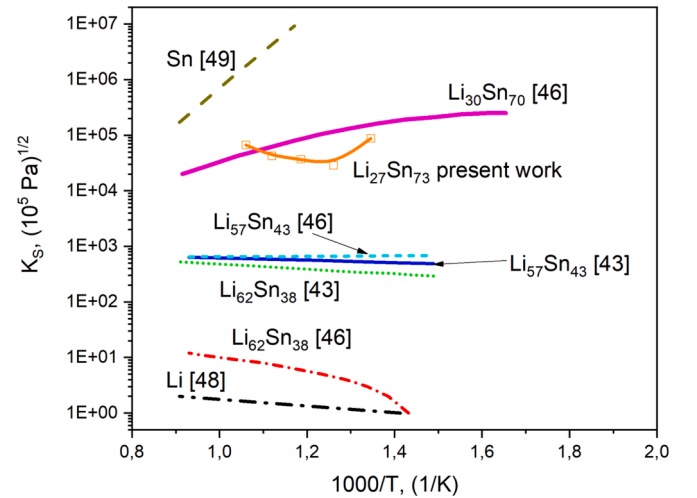


Fig. 20. Temperature dependence of Sieverts' constant [43, 46, 48-49].

of the derivative function $\frac{d \ln(K_S)}{d \left(\frac{1}{T} \right)}$ (where K_S is Sieverts' constant for

hydrogen) in the $SnLi$ undergo changes from negative to positive values depending on the lithium content. It was found that at 800 °C near $X_{Sn} \sim 0.68$, the nature of the hydrogen absorption reaction for the alloys should change from exothermic at $X_{Sn} < 0.68$ to endothermic at $X_{Sn} > 0.68$. In addition, the results given in [47] for thermodynamic data indicate that the value of lithium activity in the $Li-Sn$ system changes significantly with a change in the alloy temperature. Apparently, this is associated with a change in the concentration on boundary separating the regions of exothermic and endothermic reactions. Calculations carried out for temperature of alloy of 500 °C showed that the value of the

concentration on boundary separating the two regions shifted to the region of higher values ($X_{Sn} = 0.88$).

Thus, the obtained Sieverts' constants for deuterium in the $\text{Sn}_{73}\text{Li}_{27}$ alloy lie in the region of the transition boundary between the endothermic and exothermic reactions of deuterium absorption by the alloy, and, apparently, there is an effect of changing this boundary at different sample temperatures.

The temperature dependence of the Sieverts' constant for the test sample in the temperature range of 500–650 °C was determined as:

$$K_S(T) = 3.6 \cdot 10^6 \cdot \exp\left(-\frac{32400(J)}{RT}\right) \cdot Pa^{\frac{1}{2}}. \quad (7)$$

5. Conclusion

This paper presents the results of a series of experiments on deuterium adsorption/desorption by a $\text{Sn}_{73}\text{Li}_{27}$ alloy sample. To prepare an alloy sample with the required tin and lithium content, a technique was developed and a special experimental device was constructed. An ampoule device was manufactured to conduct a series of experiments to study sample saturation with deuterium and desorption of deuterium and deuterium-containing molecules from it. Saturation was carried out at alloy temperatures of 650, 600, 550, 500 and 450 °C. Sample heating during TDS experiments was 20 °C/min.

The possible mechanism of deuterium dissolution and release from the tin-lithium alloy was considered and temperature dependences of the effective deuterium solubility constant K_S in the tin-lithium alloy were calculated within the framework of the proposed mechanism. The temperature dependence of the Sieverts' constant for the test sample in the temperature range of 500–650 °C was determined as $K_S(T) = \exp\left((15.1 \pm 0.9) - \frac{3900 \pm 700}{T}\right) \cdot (10^5 Pa)^{\frac{1}{2}}$. Since the nature of the reaction of deuterium absorption by the sample apparently changes in the studied temperature range, the estimates obtained in the work for expressing the Sieverts constant should be considered as evaluative (effective), and the analysis carried out in this work is not final. In the future, it is planned to conduct research on computer modeling of the results of these experiments, within the framework of hypotheses on the identified mechanisms and processes. A number of additional experiments are also planned to study the interaction of hydrogen isotopes with tin-lithium alloys under conditions of high plasma loads and under conditions of high-density neutron irradiation.

CRedit authorship contribution statement

T. Kulsartov: Writing – original draft, Investigation, Conceptualization. **I. Kenzhina:** Investigation, Funding acquisition, Formal analysis. **Yu Ponkratov:** Investigation. **Yu Gordienko:** Methodology, Investigation. **Zh Zaurbekova:** Validation, Data curation. **K. Samarkhanov:** Investigation. **S. Askerbekov:** Investigation. **Ye A. Kenzhin:** Supervision, Conceptualization. **A.B. Yelishenkov:** Visualization, Software, Methodology.

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.

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Data availability

The authors are unable or have chosen not to specify which data has been used.

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