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Abstract

The use of lithium (Li) or tin (Sn) as a liquid metal plasma facing component has purposed as a solution to the high power load issue on the divertor region of nuclear fusion reactors. The possibility to use these materials depends on their compatibility with hydrogen plasmas. With the purpose of realizing deuterium retention studies, specimens of pure Sn (99.999 % Sn) and Li-Sn eutectic alloy (30 at.% Li) were exposed in the ISTTOK edge plasma ($r/a \approx 0.8$). At this position the typical plasma parameters are $T_e \approx 30$ eV and $n \approx 1 \times 10^{18}$ m^{-3}. *Ex situ* analysis of the samples was performed by means of ion beam diagnostics. Nuclear reaction analysis (NRA) technique was applied using the D(^{3}He,p)^{4}He reaction to quantify the fuel retention on the samples.

In this work the deuterium retention is compared between pure Sn and Li-Sn alloy samples in both states. All the samples were found to have retention smaller than 0.1 at.%. This low retention is expected for pure tin given its high mass and the instability of tin hydrides. However the retention was unexpectedly low for the case of Li-Sn which was thought to be proportional to the lithium fraction in the alloy. These results suggest that tin has a dominant role in the retention mechanism in this material.

Keywords: Liquid metals, Plasma-surface interaction, Lithium, Tin, Deuterium retention, Tokamak ISTTOK

1. Introduction

The use of liquid metals (LM) as plasma-facing material has been pointed out as a possible alternative to the solid walls option in order to cope with the high power loads impinging on the first wall and particularly the divertor region of fusion reactors. The regenerative properties of the liquid surface and "vapour shielding" effect are part of the advantages that come from using a liquid wall. Metals such as lithium (Li), gallium (Ga) or tin (Sn) have been suggested as well as the eutectic lithium-tin (Li-Sn) alloy (30 at.% Li) which is expected to display the beneficial properties of both its constituent elements [1]. The application of these ma-

terials in fusion reactors depends on their compatibility with the plasma. The discharge degradation induced by the enhanced impurity contamination and their affinity to retain hydrogenic isotopes are two important factors.

In the framework of liquid metal plasma facing components (PFC) the most commonly used material in the literature is lithium [2, 3, 4] where it has been used in several different configurations and different magnetic confinement fusion devices due to its low atomic mass. However lithium's evaporation rate quickly becomes a problem as temperatures raises thus limiting its utilization. The other two elements, Galium and Tin, are not so commonly used. Previously, studies were performed in ISTTOK with gallium to understand its behaviour in tokamak conditions [5, 6]. Therfore working with tin was deemed relevant and the results regarding deuterium retention will be discussed here. The

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work with Li-Sn alloy is also discussed here as its hybrid behaviour of both low and high nuclear mass materials revealed unpredicted results.

In this work we aim at an evaluation of the deuterium retention in two materials pure Sn and Li-Sn alloy each exposed in two different states liquid and solid. The total integrated exposure time was kept similar for all samples to that their retention is comparable. With this intent the samples were exposed to the edge plasmas of the tokamak ISTTOK for several of its AC discharges. Similar plasma conditions and a fixed radial positions were used for each individual irradiations. Deuterium retention measurements were achieved by means of ion beam analysis.

2. Experimental setup

Recently, an experimental setup has been developed to produce and expose LM samples in both liquid and solid states to ISTTOK plasmas which has been described in greater detail in [7]. It consists of two different assemblies, a manipulator (i) for the purpose of positioning and conditioning the samples during exposure and (ii) an auxiliary controlled preparation chamber for the samples. These two are built upon the previous systems described in earlier work with gallium [6].

2.1. Sample preparation

The Sn used in this work is a 99.999% pure rod sourced from MaTeck [8] (Lot. No. 14011517) and the Li-Sn alloy was produced by alloying pure Li and Sn metals under vacuum at the Nuclear Radiations Laboratory at the U. Illinois Urbana. Its composition was chosen to be 30 atomic percent of lithium and 70 atomic percent tin ($Li_{30}Sn_{70}$).

This secondary chamber offers more control during the preparation of the samples while mimicking the vacuum conditions of the tokamak vessel. The main purpose of this chamber is to monitor the first fusion of the sample material under UHV. This is done in the same holding cup (and same manipulator) as that which will be loaded in the appropriate port of the tokamak for exposure. The current of the heater is then ramped until the surface temperature of the sample is above melting point. Then these conditions are kept until the until the sample has uniformly molten and the pressure becomes stable. It is common to observe a migration of most impurities (namely oxides) to the liquid surface during this period. When this occurs the horizontal

Figure 1: Tin sample in its holder after exposure to ISTTOK plasmas.

manipulator is used to wipe out the surface. This is of particular importance to the Li-Sn alloy since minimal exposure to the atmosphere can lead to oxidation and this would alter the properties of the surface exposed to the plasma. Furthermore it is important to assure a good wetting on the holder's cup since there isn't any porous system holding the sample. To achieve good wetability it was necessary to have the stainless steel substrate cleaned in chloridric acid followed by a ultrasonic bath. After this preparation in the auxiliary chamber(which now contains the sample) is quickly transferred to the tokamak.

Finally the sample is degassed, raised to the selected hight for exposure position and reheated. When installed in the tokamak the surface temperature of the samples is monitored using a pyrometer (optris CTlaser 3ML) focused on the sample from the top connector of the port. Figure 1 depicts a Sn sample still in its holder after extraction from the tokamak.

2.2. Sample exposure in ISTTOK

The samples were exposed in ISTTOK, a high aspect ratio tokamak with a circular cross-section. Its main parameters are: $R = 46$ cm,$a = 8.5$ cm, $B_T = 0.5$ T, $I_p = 5$ kA. One of the main advantages of this device is the fact that it can be operated in AC-mode [9], meaning that each discharge consists of several alternated pulses which was made possible due to improvements in the real-time control system of ISTTOK [10, 11]. This operation mode extends the length of discharge to the range of seconds allows for a quicker production of samples. The typical parameters in the edge

2

plasma, where the samples are exposed, are: electron and ion, $T_e \sim T_i = 30 - 40$ eV, electron density, $n_e = 0.5 - 3 \times 10^{18}$ m^{-3}, particle flux $\Gamma^{D+} = 1 - 7 \times 10^{22}$/m^{2}s and discharge duration of 250 ms (with each shot consisting of ten alternating pulses with a duration of ~ 25 ms).

The samples were all exposed at a normalized radial position of $r/a = 0.8$, heated above their respective melting points and allowed to completely degas. For the purpose of melting pure Sn samples where kept at 250°C while the Li-Sn alloy were heated to 385°C. Afterwards the samples were irradiated under deuterium plasmas either in the liquid state or solid state. This allowed the comparison between induced effects caused by solid and liquid exposures. After being exposed to the plasma the samples were allowed to solidify and then removed from the tokamak to open air conditions and quickly transferred ion beam facilities. The reason why deuterium plasmas where used to perform this retention study has to do with its rare occurrence in the atmosphere and consequently any detected quantity can be related directly to this experiment and not contamination.

Samples were analysed less than 48 hours after the last irradiation discharge at the Laboratory of Accelerators and Radiation Technologies using two different ion beam techniques. Carbon (C), oxygen (O) and tin (Sn) depth profiles were evaluated by Rutherford Backscattering (RBS) by using 2.2 MeV incident H$^+$ ion beams, being the RBS detector located at a scattering angle of 165°, while the quantification of deuterium (D) and lithium (Li) was performed via Nuclear Reaction Analysis (NRA) making use of 1.2 MeV ^{3}He$^+$ beams at a scattering angle of 140°. At this time, a mylar foil with a thickness of 700 μm was placed in front of the NRA detector in order to decelerate the protons emitted from the D(^{3}He,p)^{4}He, ^{7}Li(^{3}He,p)^{9}Be, ^{6}Li(^{3}He,p)^{8}Be nuclear reactions. Data analysis was carried out with the IBA Data-Furnace (NDF) code [12, 13]. As it can be seen in figure 1 the surface of the sample is rough and therefore it was deemed relevant to analyse each sample on different points in order to assure homogeneity of the exposure.

3. Results and discussion

The NRA spectra and the corresponding best fit lines for all the samples are shown in figures 2 to 5.

Each figure shows two different NRA spectra gathered in two different locations to assure homo-

Sample	Pos.	D retained ($at./cm^2$)	D retained ($at.\%$)
Sn solid	1	11.70×10^{15}	0.30
	2	12.92×10^{15}	0.34
Sn liquid	1	3.42×10^{15}	0.09
	2	3.20×10^{15}	0.08
Li-Sn solid	1	9.92×10^{15}	0.062
	2	9.59×10^{15}	0.060
Li-Sn liquid	1	2.70×10^{15}	0.018
	2	2.85×10^{15}	0.019

Table 1: Deuterium content in the samples exposed in IST-TOK determined by NRA. The at.% are evaluated for a pure Sn matrix.

geneity of the exposition and of the sample (in the case of the Li-Sn samples) followed by the fit of these spectrum from which the deuterium intake is inferred. The value of the retention measured in absolute by the NRA technique and consequent fit by the NDF software is shown for all samples in table 1. In previous works the observation of deuterium in the Li-Sn samples was very difficult however taking advantage of longer beam time it was possible to quantify the quantity of deuterium in all present samples. Using the cross-sectional data for the interaction of a ^{3}He beam with 1.2 MeV energy with a deuterium target it is possible to recover the absolute quantity of deuterium retained in the sample without the need to use a reference sample.

The ion flux at the edge of the tokamak is taken to be $\Gamma = 4 \times 10^{22}$ ion/m^{2}s at equivalent radial position $r/a = 0.8$ where all the samples were exposed. This quantity was monitored regularly using Langmuir probe data during the discharges, where a set of probes was kept at the same radial position as the samples.

The Sn samples we exposed to 3s of plasma while the Li-Sn samples were exposed to 4s. With this we can have the total amount of deuterium impinging

$\frac{D_{retained}}{D_{incident}}$	Sn	Li-Sn
Liquid	2.76×10^{-4}	1.73×10^{-4}
Solid	1.03×10^{-3}	6.10×10^{-4}

Table 2: Deuterium content in the samples expressed as retained fraction. The total ion flux is calculated with the total exposition time and retained particles are taken from the average of the two points in table 1.

3

on a sample, $D_{incident}$, and with the results presented in table 1 we can define retained fraction as $D_{retained}/D_{incident}$. These fractions, using the average for the two positions, are shown in table 2. In the balance of deuterium flux to the sample only the ion fraction is accounted and the neutral particle flux is neglected, particularly during the switching of the ISTTOK plasmas. This is a known underestimation however this would further reduce the presented ratios.

Comparing the results for both materials reveals that the retention is lower by almost a factor of 4 in the case where samples are kept in liquid phase. This is expected since liquid metals have a natu-ral tendency to expel impurities to their surface. Once there the probability that a deuterium atom is returns back to the plasma is much higher. This mechanism becomes specially relevant for tin because of its high mass and, together with the insta-bility of tin hydrides greatly reduces the retention. This can be inferred by the low NRA yield in figures 2 and 3.

From the fits to the NRA yield of the Li-Sn sam-ples, shown in figures 4 and 5, its possible to identify a thin layer of roughly 3 micrometers where the rel-ative lithium content is enriched from its 30 at.% to 50 at.%. Moreover this is also the region where most of the retained deuterium was found. This
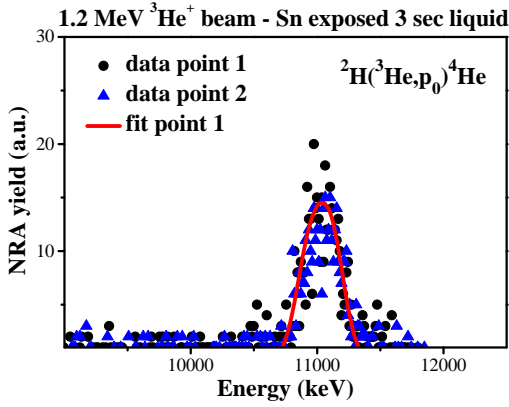

Figure 2: Nuclear reaction analysis spectrum of a pure Sn sample exposed in liquid state. The NRA spectrum is shown for two different positions (black dots and blue triangles) and the fit (red line) for the first position is shown.
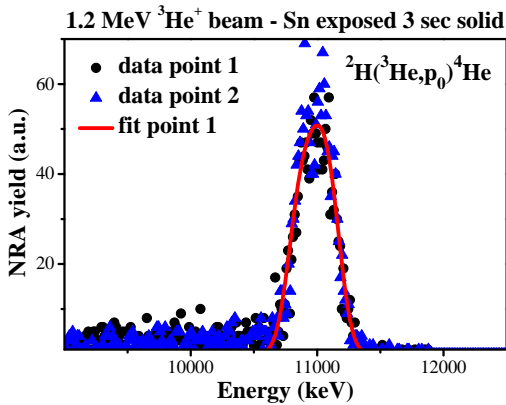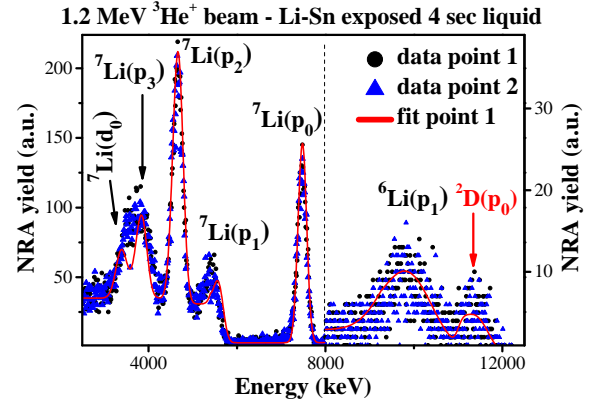

Figure 4: Nuclear reaction analysis spectrum of a Li-Sn alloy sample exposed in liquid state. The NRA spectrum is shown for two different positions (black dots and blue triangles) and the fit (red line) for the first position is shown.

Figure 3: Nuclear reaction analysis spectrum of a pure Sn sample exposed in solid state. The NRA spectrum is shown for two different positions (black dots and blue triangles) and the fit (red line) for the first position is shown.
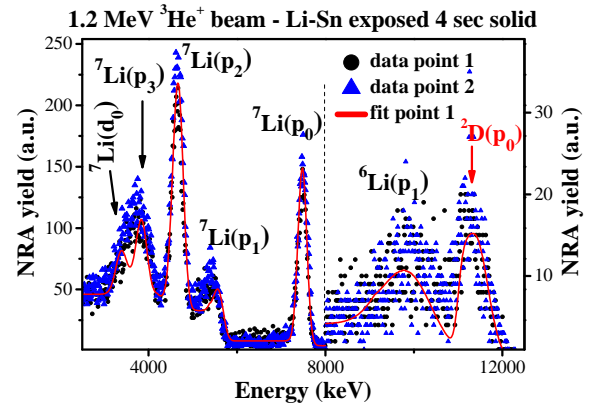

Figure 5: Nuclear reaction analysis spectrum of a Li-Sn alloy sample exposed in solid state. The NRA spectrum is shown for two different positions (black dots and blue triangles) and the fit (red line) for the first position is shown.

corroborates the speculated behaviours for this alloy where lithium plays a preferential role in the interaction with the plasma and in particular in fuel retention. However the retention is much lower than the recorded retention for pure lithium [14] or for similar alloys [15]. Complementary RBS measurements where made on the samples. These show the presence of common airborne contaminants present, such as carbon and oxygen.

All the obtained retention ratios are all bellow 0.1 at.%. This means that for this particular property these materials are similar to that reported for tungsten [16, 17, 18] and outperforms carbon [19]. By itself this suggest that all studied case make good candidates for a PFC. In fact a comparison between pure Sn and Li-Sn shows that for each state the later roughly half the retention ratio of the former. Although this would seem to indicate that the Li-Sn would make a better PFC material the authors must alert that the utilization of this material is accompanied by handling issues. The metal corrosion of Sn is enhanced by the chemical activity of Li which introduces a trade-off for the choice of materials.

4. Summary

The experimental setup and relative experimental procedure for the exposure of tin and lithium-tin alloy samples is described in the present work.

Samples of Sn and Li-Sn where exposed at IST-TOK to deuterium plasmas for comparable total integrated exposure times ($\Gamma^{D+} = 1 - 7 \times 10^{22}/\text{m}^2\text{s}$; $T_i = 30 - 40$ eV). The samples were irradiated in liquid and solid states (at $250°$C for the pure tin and $385°$C for the alloy for the liquid states). The observation of deuterium retention on the samples was tested with nuclear reaction analysis (NRA) technique. Deuterium retention was observed and quantified in all samples.

5. Acknowledgements

6. References

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