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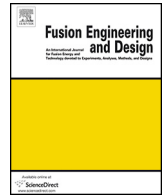
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Behavior of liquid Li-Sn alloy as plasma facing material on ISTTOK



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ABSTRACT

The high power loads impinging on the first wall and particularly the divertor of fusion reactors is a decisive factor to the success of nuclear fusion. An alternative to solid plasma facing components is the use of liquid metals such as lithium or tin due to the regenerative properties of the liquid surface. Another suitable candidate is the eutectic lithium tin alloy (30 at.% Li) which is suggested to display beneficial properties of both its constituent elements. The application of these materials as liquid metal plasma facing components depends on several factors such as their affinity to retain hydrogenic isotopes and the discharge performance degradation induced by the enhanced impurity contamination, among others. An experimental setup has been developed to produce and expose samples to ISTTOK plasmas on both liquid and solid states. Samples of Li-Sn alloy were exposed at ISTTOK to deuterium plasmas. Post-mortem analysis of the samples was performed by means of ion beam diagnostics. To quantify the fuel retention on the samples the nuclear reaction analysis (NRA) technique was applied. Complementary, Rutherford backscattering spectrometry (RBS) was used for determination material composition, particularly of impurities, on the samples. Regardless of the high sensitivity of these techniques no deuterium was detected in the samples. Emission of the Li-I 670.7 nm line indicates that there was interaction of the plasma with the samples. Alternative reasons for the low retention of this material are discussed. Lithium segregation to the surface of the sample was observed.

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1. Introduction

One of the unsolved issues in nuclear fusion is related to the high power loads impinging on the first wall particularly on the divertor region of the reactor. Using liquid metals, such as lithium, gallium or tin as plasma-facing material has been pointed out as possible alternative to the solid walls option due to the regenerative properties of the liquid surface. However, the use of these materials in fusion reactors depends on the discharge performance degradation induced by the enhanced impurity contamination and on their affinity to retain hydrogenic isotopes, among other factors.

Extensive work on lithium exists in the literature [1–3] and it has been claimed that the maximum wall temperature is limited by its high evaporation rate. Less is known for gallium and tin however some studies on gallium's behavior under tokamak conditions were made previously at ISTTOK [4,5] and work is currently being

performed on tin. Nonetheless, the high atomic number of these two materials, particularly for tin, is seen as a disadvantage.

Alternatively, the combination of tin and lithium in an alloy (Sn with 20–30 at.% Li) displays beneficial properties, which also qualify it as a suitable candidate. The evaporation rate of this material is at least three orders of magnitude lower than that of pure lithium while keeping an effective charge similar to lithium's [6].

The aim of this work was to evaluate the deuterium content in the alloy samples (both solid and liquid) for different total integrated exposure times. For this the samples were irradiated under several half second AC discharges within ISTTOK edge plasma. To that purpose an experimental setup was developed to monitor the samples production. Similar plasma conditions and fixed radial positions were used for each individual irradiations. Deuterium retention measurements were achieved by means of ion beam analysis.

2. Experimental setup

For the purpose of exposing the liquid metal samples two different setups were developed: (i) a positioning and conditioning

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system and (ii) a controlled preparation chamber for the samples. These two are built upon the previous systems described in earlier work with gallium [5].

2.1. Sample preparation

The Li-Sn alloy used in this work 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 at.% of lithium and 70 at.% tin ($\text{Li}_{30}\text{Sn}_{70}$).

The preparation chamber, is used to monitor each sample's first fusion under UHV conditions similar to those in the tokamak vessel. A method was developed to prepare the samples in which the samples are loaded inside this chamber using the same holding cup as the one that will be later introduced in the tokamak. Then the current on the heater is raised until the surface temperature of the sample is above its melting point at which point it is kept constant until the sample is uniformly melted and the pressure becomes stable. During this stage it is common to observe a migration of most impurities (namely oxides) to the liquid metal surface. In these situations the chamber's horizontal manipulator is used to wipe out the surface. Since there is no capillary porous system keeping the liquid metal in the holder it is important to assure a good wetting on the holder's cup. Since tin is commonly used as a welding material its wettability is naturally assured. However it is still necessary to have the stainless steel substrate cleaned in hydrochloric acid followed by an ultrasonic bath. After this preparation the samples and the bottom part of the manipulator are quickly transferred to the tokamak.

Once the sample holder is installed in the tokamak vessel and properly degassed, it is raised to its final exposure position and reheated. The surface temperature of the samples was monitored using a pyrometer (optris CTlaser 3ML) focused on the sample from the top connector of the port. Restricted due to the limited access to the bottom of the tokamak the same two stage system implemented previously in [5] was used. The positioning system consists of a set of bellows whose compression is imposed via the manual fastening of a threaded shaft thus allowing for vertical motion. The upper bellow is used for coarse positioning of the sample beyond the vessel limits while the bottom allows a fine adjustment. A cross-section of this system is depicted in Fig. 1 attached to the tokamak vessel. A gate valve is placed between the tokamak chamber and positioning system to allow vacuum separation of the regions. Along this system is a structural rod where the sample holder is fixated and to which additional wires can be attached to carry current to the sample holder. The sample holder is made of a bulk piece of boron nitride with a threaded nichrome filament that serves as heating element. Around this there is an electrically isolated stainless steel shield which protects the filament and on top is a substrate cup which holds the sample material.

2.2. Sample exposure in ISTTOK

The tokamak ISTTOK is a high aspect ratio device with a circular cross-section whose main parameters are: $R = 46$ cm, $a = 8.5$ cm, $B_T = 0.5$ T, $I_p = 5$ kA. In the recent years ISTTOK has been operated in AC-mode [7] which consists of several alternated pulses per discharge. This operation extended the discharge time to the range of seconds due to improvements in the real-time control system [8,9] which in turn allows for a quicker production of samples. The typical parameters for ISTTOK's edge plasmas are: electron and ion, $T_e \sim T_i = 30\text{--}40$ eV, electron density, $n_e = 0.5\text{--}3 \times 10^{18} \text{ m}^{-3}$, particle flux $\Gamma^{D+} = 1\text{--}7 \times 10^{22} / \text{m}^2 \text{ s}$ and discharge duration of 250 ms (each shot consists of ten alternating pulses with a duration of ~ 25 ms).

All the liquid metal samples were exposed at a normalized radial position of $r/a = 0.8$, heated above their respective melting points

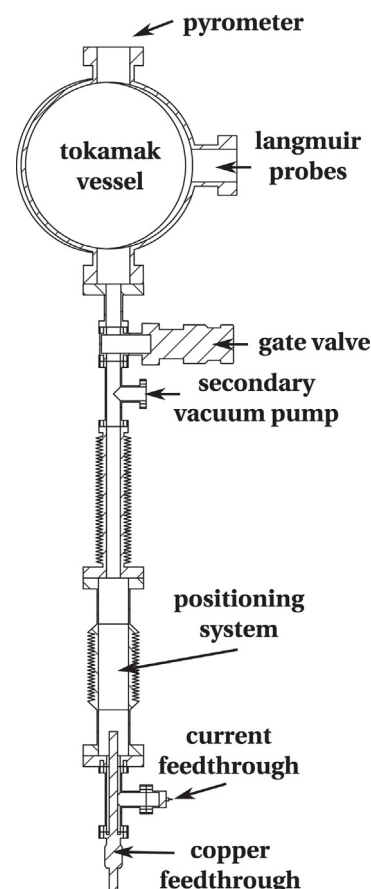


Fig. 1. Sample positioning and conditioning system coupled to the ISTTOK vessel.



Fig. 2. Tin sample in its holder after exposure to ISTTOK plasmas.

and irradiated in the liquid state under deuterium plasmas. Another sample was positioned at the same equivalent position in the solid state allowing for a 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.

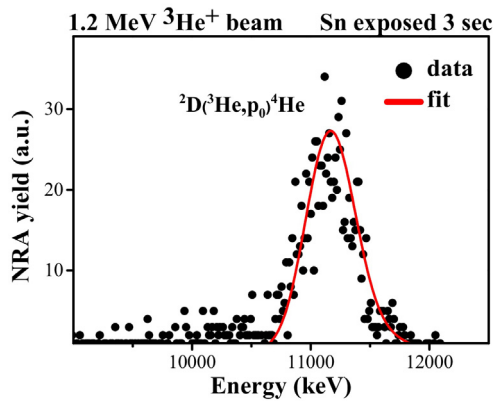


Fig. 3. Nuclear reaction analysis spectrum of the pure tin sample.

Samples were analyzed less than 48 h after the last irradiation discharge at the Laboratory of Accelerators and Radiation Technologies (LATR) 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 2.2 MeV $^3He^+$ beams and of a scattering angle of 140° . At this time, a Mylar foil with a thickness of $700 \mu m$ was placed in front of the NRA detector in order to decelerate the protons emitted from the $D(^3He,p)^4He$, $^7Li(^3He,p)^9Be$, $^6Li(^3He,p)^8Be$ nuclear reactions. Data analysis was carried out with the IBA Data-Furnace (NDF) code [10,11].

3. Results and discussion

The NRA spectra and the corresponding best fit lines for all the samples are shown in Figs. 3 and 4. One of the main advantages having exposed the samples to deuterium plasmas is that its occurrence in the atmosphere is small and therefore any detected quantity can be related directly to this exposure. Deuterium is clearly visible in the pure tin sample however the same cannot be seen in the Li-Sn alloy samples. Using the cross-sectional data for the interaction of a 3He 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 is taken to be 4×10^{22} ion/ m^2s at equivalent radial position $r/a=0.8$ where all the samples were tested. This flux is determined by averaging the appropriate Langmuir probe signals

during this discharges, which were kept at the same radial position as the samples. Thus the retained fraction can be defined as $D_{retained}/D_{incident}$. Since ISTTOK plasmas are switched there is also an additional flux of neutral atoms which is not accounted for in our balance. This would further reduce the presented values.

A liquid tin sample was exposed for a total integrated exposure time of 3 s. A total amount of deuterium of 8.2×10^{19} at/ m^2 was found retained in the sample, which corresponds to a retained fraction of 0.068 at.%, as seen in Fig. 3. This sample is shown here for comparison purpose with the following samples. The liquid Li-Sn alloy samples were exposed under the same conditions for integrated exposure times of 3, 4 and 5 s. Most of the obtained spectra have a small NRA yield in the deuterium reaction meaning that only an insignificant amount is retained in the samples. In Fig. 4 the region where this peak should be is denoted.

One of the speculated behaviors for this alloy is that lithium should play a preferential role in the interaction with the plasma. Both the NRA and RBS measurements performed on these samples support this hypothesis in complementary ways by exhibiting the lithium segregation to the surface of the sample. Both diagnostics are susceptible to a very thin layer of the sample with roughly 3 micrometers. A comparison of the spectrum from a virgin alloy sample and one that was exposed to the plasmas show that the intensity of the NRA yield increases by a factor of roughly 1.3 in the region corresponding to the lithium peaks ($^7Li(^3He,p)^9Be$, $^6Li(^3He,p)^8Be$). This difference is due to absence of stirring in the samples (particularly during solidification) and not due to the irradiation itself. Interestingly the comparison of this two spectra, as shown in Fig. 5, clearly denotes a presence of additional residual counts in the deuterium region for the irradiated sample. Complementary, the RBS spectra in the thin film approximation exhibits a layer mostly composed of lithium with some common contaminants present, such as carbon and oxygen, while the tin barrier is much shifted to the lower energies as shown in Fig. 6. Again this suggests an enrichment of the outer layers with lithium.

The absence of deuterium in the Li-Sn samples was an unexpected result particularly when compared with the result obtained for pure Sn presented here and pure Li [12]. The emission line of Li-I at 670.7 nm was compared with the intensity of the D_α . This observation was made using a tangential port of the tokamak that points toward the location of the sample. The ratio between the total integrated intensity of the two lines (I_{Li}/I_{D_α}) was observed to be consistent from shot to shot basis and mostly a function of the depth of the sample. Those ratios are approximately 10% for $r/a=0.88$ and 25% for $r/a=0.76$ whereas no Li emission was observed with the sample removed. Furthermore an additional increase on the surface temperature of the sample (as recorded by

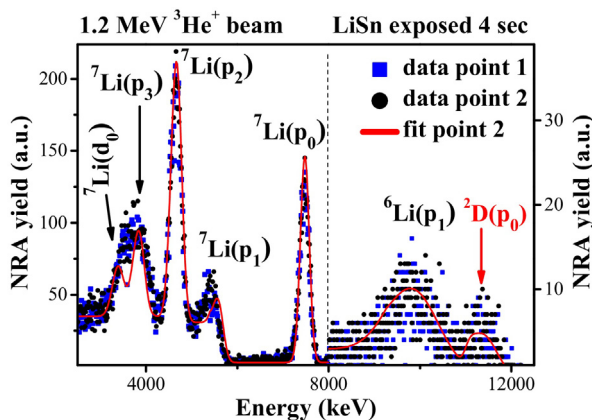


Fig. 4. Nuclear reaction analysis spectrum of the Li-Sn alloy sample. The NRA spectrum is shown for two different positions and the fit for one of them is shown.

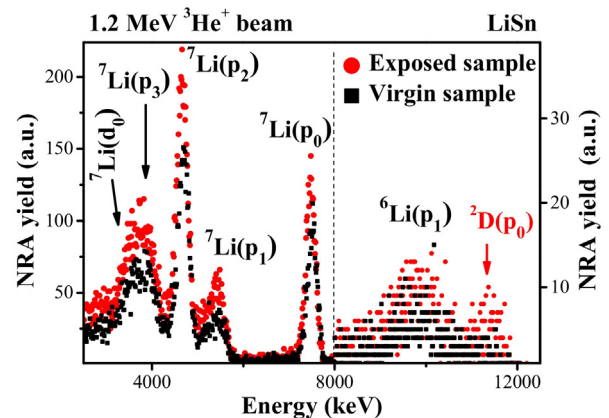


Fig. 5. Nuclear reaction analysis spectra comparison of a virgin Li-Sn alloy sample and an irradiated one.

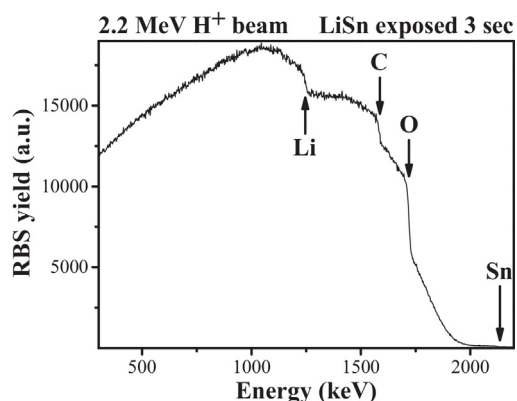


Fig. 6. Rutherford backscattering spectrum of the Li-Sn alloy sample.

the pyrometer) during the shot was observed in the range of some tens of degrees. These two evidences lead to the conclusion that there was an interaction between the plasmas and the samples and that they were irradiated. However the absence of deuterium retention for the Li-Sn alloy might be explained by other reasons. The heating capabilities of the sample holder were limited and the occurrence of several phases in the alloy, created an irregular surface on top of the meniscus. It is therefore possible that this rugosity lead to a preferential plasma interaction region and consequently a non-uniform deposition of the deuterium. Alternatively it is possible that the sample was contaminated during transport in atmospheric conditions. In pure lithium samples hydrogen is often retained in the form of lithium hydride [13] which reacts violently with air leading to quick loss of all retained material. Further investigation in the retention mechanism in the alloy must be made and complementary *in situ* measurements of retained material must be made to access the impact of the sample exposure during transport.

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 these liquid metals were exposed at ISTTOK to deuterium plasmas for several different total integrated exposure times ($\Gamma^{D^+} = 1-7 \times 10^{22}/\text{m}^2\text{s}$; $T_i = 30-40$ eV). The pure tin sample and three lithium-tin samples were irradiated in liquid state (at 250 °C for the pure tin and 385 °C for the alloy). The observation of deuterium retention on the samples was tested with nuclear reaction analysis (NRA) technique. The only case where retention could be quantified was in the pure tin sample. There was no occurrence of deuterium in the lithium-tin samples, however the absence of deuterium might be explained by reasons other than the low retention of the material, such as: the uniformity of the sample surface leading to preferential retention zones or the contamination of the samples during transport to be analyzed. Another subject that was investigated was the segregation of Li under liquid phase of the alloy samples. To such end a comparison was made using an irradiated sample and a virgin sample. These two samples were compared using both nuclear reaction analysis and Rutherford backscattering which provides complementary information. The intensity of the Li peaks in the NRA spectrum doubles from the virgin to the exposed sample while the RBS spectrum shows a tin barrier that is much deeper than the surface of the sample.

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References

- [1] F.L. Tabarés, Present status of liquid metal research for a fusion reactor, *Plasma Phys. Control. Fusion* 58 (1) (2016) 014014 <http://stacks.iop.org/0741-3335/58/i=1/a=014014>.
- [2] G. Mazzitelli, M. Apicella, G. Apruzzese, F. Crescenzi, F. Iannone, G. Maddaluno, V. Pericoli-Ridolfini, S. Rocella, M. Reale, B. Viola, I. Lyublinski, A. Vertkov, Experiments on {FTU} with an actively water cooled liquid lithium limiter, *J. Nucl. Mater.* 463 (2015) (2014) 1152–1155, <http://dx.doi.org/10.1016/j.jnucmat.2014.12.050>, pLASMA-SURFACE {INTERACTIONS} 21Proceedings of the 21st International Conference on Plasma-Surface Interactions in Controlled Fusion Devices Kanazawa, Japan, May 26–30, <http://www.sciencedirect.com/science/article/pii/S0022311514009921>.
- [3] R. Majeski, R. Kaita, M. Boaz, P. Eftimion, T. Gray, B. Jones, D. Hoffman, H. Kugel, J. Menard, T. Munsat, A. Post-Zwicker, J. Spaleta, G. Taylor, J. Timberlake, R. Woolley, L. Zakharov, M. Finkenthal, D. Stutman, G. Antar, R. Doerner, S. Luckhardt, R. Seraydarian, R. Maingi, M. Maiorano, S. Smith, D. Rodgers, V. Soukhanovskii, Testing of liquid lithium limiters in cdx-u, *Fusion Eng. Des.* 72 (1–3) (2004) 121–132, <http://dx.doi.org/10.1016/j.fusengdes.2004.07.002>, special Issue on Innovative High-Power Density Concepts for Fusion Plasma Chambers, <http://www.sciencedirect.com/science/article/pii/S0920379604001127>.
- [4] R. Gomes, H. Fernandes, C. Silva, A. Sarakovskis, T. Pereira, J. Figueiredo, B. Carvalho, A. Soares, C. Varandas, O. Lielausis, A. Klyukin, E. Platacis, I. Tale, Interaction of a liquid gallium jet with the tokamak {ISTTOK} edge plasma, *Fusion Eng. Des.* 83 (1) (2008) 102–111, <http://dx.doi.org/10.1016/j.fusengdes.2007.08.019> <http://www.sciencedirect.com/science/article/pii/S0920379607004243>.
- [5] R. Gomes, R. Mateus, E. Alves, H. Fernandes, C. Silva, P. Duarte, Hydrogen retention in gallium samples exposed to {ISTTOK} plasmas, *Fusion Eng. Des.* 86 (9–11) (2011) 2458–2461, <http://dx.doi.org/10.1016/j.fusengdes.2011.03.055>, Proceedings of the 26th Symposium of Fusion Technology (SOFT-26), <http://www.sciencedirect.com/science/article/pii/S0920379611003280>.
- [6] J. Allain, D. Ruzic, M.D. Hendricks, He and Li sputtering of liquid eutectic Sn-Li, *J. Nucl. Mater.* 290–293 (2001) 33–37, [http://dx.doi.org/10.1016/S0022-3115\(00\)00504-3](http://dx.doi.org/10.1016/S0022-3115(00)00504-3), 14th Int. Conf. on Plasma-Surface Interactions in Controlled Fusion Devices, <http://www.sciencedirect.com/science/article/pii/S0022311500005043>.
- [7] H. Fernandes, C. Varandas, J. Cabral, H. Figueiredo, R. Galvão, Engineering aspects of the {ISTTOK} operation in a multicycle alternating flat-top plasma current regime, *Fusion Eng. Des.* 43 (1) (1998) 101–113, [http://dx.doi.org/10.1016/S0920-3796\(98\)00263-4](http://dx.doi.org/10.1016/S0920-3796(98)00263-4) <http://www.sciencedirect.com/science/article/pii/S0920379698002634>.
- [8] I.S. Carvalho, P. Duarte, H. Fernandes, D.F. Valcárcel, P.J. Carvalho, C. Silva, A.S. Duarte, A. Neto, J. Sousa, A.J. Batista, T. Hekkert, B.B. Carvalho, R.B. Gomes, Real-time control for long ohmic alternate current discharges, *Fusion Eng. Des.* 89 (5) (2014) 576–581, <http://dx.doi.org/10.1016/j.fusengdes.2014.03.029>, proceedings of the 9th {IAEA} Technical Meeting on Control, Data Acquisition, and Remote Participation for Fusion Research, <http://www.sciencedirect.com/science/article/pii/S0920379614002087>.
- [9] I.S. Carvalho, P. Duarte, H. Fernandes, D.F. Valcárcel, P.J. Carvalho, C. Silva, A.S. Duarte, A. Neto, J. Sousa, A.J. Batista, T. Hekkert, B.B. Carvalho, {ISTTOK} real-time architecture, *Fusion Eng. Des.* 89 (3) (2014) 195–203, <http://dx.doi.org/10.1016/j.fusengdes.2013.12.025>, design and implementation of real-time systems for magnetic confined fusion devices, <http://www.sciencedirect.com/science/article/pii/S0920379613007394>.
- [10] N.P. Barradas, C. Jaynes, R.P. Webb, Simulated annealing analysis of Rutherford backscattering data, *Appl. Phys. Lett.* 71 (2) (1997) 291–293, <http://dx.doi.org/10.1063/1.119524>.
- [11] www-nds.iaea.org/exfor/exfor.html (accessed 30.10.15).
- [12] E. Oyarzabal, A. Martín-Rojo, F. Tabarés, Laboratory experiments of uptake and release of hydrogen isotopes in liquid lithium, *J. Nucl. Mater.* 463 (2015) (2014) 1173–1176, <http://dx.doi.org/10.1016/j.jnucmat.2014.10.088>, pLASMA-SURFACE {INTERACTIONS} 21Proceedings of the 21st International Conference on Plasma-Surface Interactions in Controlled Fusion Devices Kanazawa, Japan, May 26–30, <http://www.sciencedirect.com/science/article/pii/S0022311514007727>.
- [13] A. Martín-Rojo, E. Oyarzabal, F. Tabarés, Laboratory studies of h retention and lih formation in liquid lithium, *Fusion Eng. Des.* 89 (12) (2014) 2915–2918, <http://dx.doi.org/10.1016/j.fusengdes.2014.07.015> <http://www.sciencedirect.com/science/article/pii/S0920379614005225>.