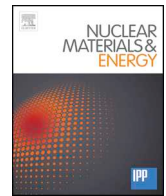




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journal homepage: www.elsevier.com/locate/nmeDeuterium retention and erosion in liquid Sn samples exposed to D₂ and Ar plasmas in GyM device

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ABSTRACT

The use of tin (Sn) as a liquid metal for plasma facing components has been recently proposed as a solution to the high heat load issue on the divertor target plates in nuclear fusion reactors. Due to its low vapor pressure, low reactivity with hydrogen and good resilience to neutron impact, tin is a good candidate as plasma facing component. However its high atomic number poses concerns about plasma contamination.

In this paper two fundamental aspects have been investigated: deuterium retention and erosion fluxes from the Sn surface towards the plasma. The samples were exposed to plasma inside the linear machine GyM in magnetic cusp configuration. This setup permits to expose free liquid specimens without the need for the Capillary Porous System. Moreover it permits to lower the magnetic field in order to increase Sn Larmor radius and consequently limit Sn re-deposition in erosion experiments.

Ex-situ analyses by ion beam diagnostics on solid samples exposed to deuterium plasma have proved that the amount of retained atomic deuterium is very low, approximately 0.18 at% estimated by Nuclear Reaction Analysis and 0.25 at% estimated by Elastic Recoil Detection Analysis.

In the framework of erosion studies, the spectroscopic parameter S/XB was evaluated in Ar plasma for the SnI line at 380.1 nm by Optical Emission Spectroscopy and mass loss measurements in the 5–11 eV T_e range, at a density n_e ~ 1.5 × 10¹¹ cm⁻³. An average value of 150 ± 23 was obtained.

1. Introduction

Heat removal from Plasma Facing Components (PFCs) in magnetic confinement fusion reactors is a crucial issue for the energy generation and structural integrity of the reactors. Liquid Metals (LMs) have been proposed as a possible alternative to solid PFCs (W and Be) because of their high heat dissipation capabilities by convection, evaporative cooling and vapor shielding, their regenerative properties and resilience to erosion and neutron damage [1,2]. So far the research was focused on liquid lithium (Li) because of its low melting point, low atomic number (Z) and reduced recycling compared to most other materials [3]. However the high hydrogen affinity of lithium leading it to form tritium containing co-deposits and its high reactivity with oxygen and copper still pose major problems for its wider acceptance.

Therefore other metals and alloys are being researched, including tin (Sn), whose low vapor pressure and low reactivity with hydrogen and other metals make it compatible with most plasma facing materials. However Sn is a high Z material and, therefore, it represents a potential concern for plasma contamination. Because impurity concentration in the plasma core is linked to erosion flux and transport mechanism, a reliable prediction of the material influx into the plasma is fundamental to guarantee safety operation. Spectroscopic methods are very promising tools for the quantification of the erosion of a PFC. However these methods often suffer from an incomplete database for the interpretation of optical signals in terms of particle fluxes. Hence the need for the empirical evaluation of the S/XB spectroscopic parameter for Sn, converting an emission line intensity into an influx of Sn impurity atoms from limiting surfaces [4]. Compared to non-spectroscopic

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methods, this technique permits to measure the time evolution of the gross erosion from a PFC during tokamak operation. In fact post-mortem analyses based on ion beam techniques provide a measurement of net erosion (or gross erosion if the specimen is small enough that the re-deposition is unlikely) integrated in time over an experimental campaign with thousands of plasma discharges under various operation scenarios, in such a way that the sputtered particle flux into plasma in a specific discharge cannot be assessed [5]. Other methods for the measurement of gross erosion are based on Langmuir probe measurements, but they require the estimate of the fractional abundance of the ion species (impurity and background fuel ions) in the plasma [6]. No theoretical data of SX/B for Sn are available right now.

The present work provides an overview of the recent activities carried out at the Institute for Plasma Physics (IFP) on the linear plasma device GyM [7] on erosion and retention in liquid tin.

The GyM device, the samples preparation and the materials analysis diagnostics are described in Section 2.

Deuterium retention was investigated by ex-situ analysis on solid Sn samples exposed to GyM plasma in liquid state. In particular the quantification of deuterium retention was performed by Elastic Recoil Detection Analysis (ERDA) in the near-surface layer (100 nm) and Nuclear Reaction Analysis (NRA) in a thicker layer (1000 nm) while the Sn depth profile was evaluated by Rutherford Backscattering Spectrometry (RBS). Preliminary measurements by Thermal Desorption Spectroscopy (TDS) were carried out to study deuterium retention in bulk Sn.

Sn erosion was estimated by mass loss measurements and Optical Emission Spectroscopy (OES) was used to determine the S/XB spectroscopic parameter for the Sn I line at 380.1 nm, according to the methodology described in Section 3.

All the experimental results are presented and discussed in Section 4.

2. Experimental details

2.1. GyM device

Solid and liquid Sn samples were exposed in GyM to Argon (Ar) or Deuterium (D₂) plasma for erosion and retention experiments respectively, at operating pressures ranging from 7×10^{-5} to 1×10^{-4} mbar. A schematic of GyM is presented in Fig. 1. GyM [7] consists of a stainless steel vacuum chamber ($\varnothing = 0.25$ m, length

2.11 m) mounted in a ten coil solenoid with a magnetic field of 0.08 T on the machine axis. Plasmas are generated and continuously sustained by means of microwave power (3 kW CW) at the electron cyclotron frequency (2.45 GHz). Power is injected perpendicularly to the magnetic field lines in O-mode polarization. The resonance at 0.0870 T is located at a position close to one end of the vessel, just to the left of the power launcher. The diameter of the plasma column is ~ 20 cm.

In order to expose liquid Sn samples without the use of the Capillary Porous System (CPS) [1,2], a double-cusp magnetic configuration, obtained by current inversion in two coils, was used (Fig. 2). This configuration allowed to suitably divert the magnetic field lines driving the plasma ions on the horizontal target.

2.2. Preparation of the samples and plasma exposure

Each sample consists of ~ 3 g of solid Sn (purity 99.9%) press fitted into a steel tray holder of overall size 20.4×18.4 mm². The samples were kept in vacuum (0.1 mbar) for 1 h at a temperature of 400 °C to remove any impurity and then were re-solidified in vacuum at room temperature (20 h). Finally they were placed in GyM at 6 cm below the machine axis, with the long side perpendicular to the machine axis (Fig. 3).

In retention experiments the samples were heated up to 300 °C (Sn melting temperature = 231.9 °C) by a lamp placed under the rear side of the sample holder. After plasma exposure, the samples were re-solidified in vacuum and stored in dry air at room temperature.

In retention experiments the specimens were kept solid and a negative bias $V = -150$ V was applied to achieve an incident ion energy of ~ 180 –200 eV, taking into account the plasma potential ($V_p \sim 30$ –50 V).

2.3. Plasma diagnostics

A high resolution (0.06 nm) spectrometer (Horiba Jobin Yvon) for Optical Emission Spectroscopy has been used to analyze the radiation emitted by plasma in the 300–900 nm wavelength range. The spectrometer consists of a Czerny-Turner monochromator (iH550) coupled to a CCD camera (Synapse), thermoelectrically cooled to -70 °C. The entrance optics consists of a focusing lens ($f = 7.5$ cm) and an optical fiber (600 μ m core diameter). The Line of Sight (LOS) intercepts the sample and is perpendicular to the machine axis. The optical system, including the optical window, was absolutely calibrated on an optical bench by a

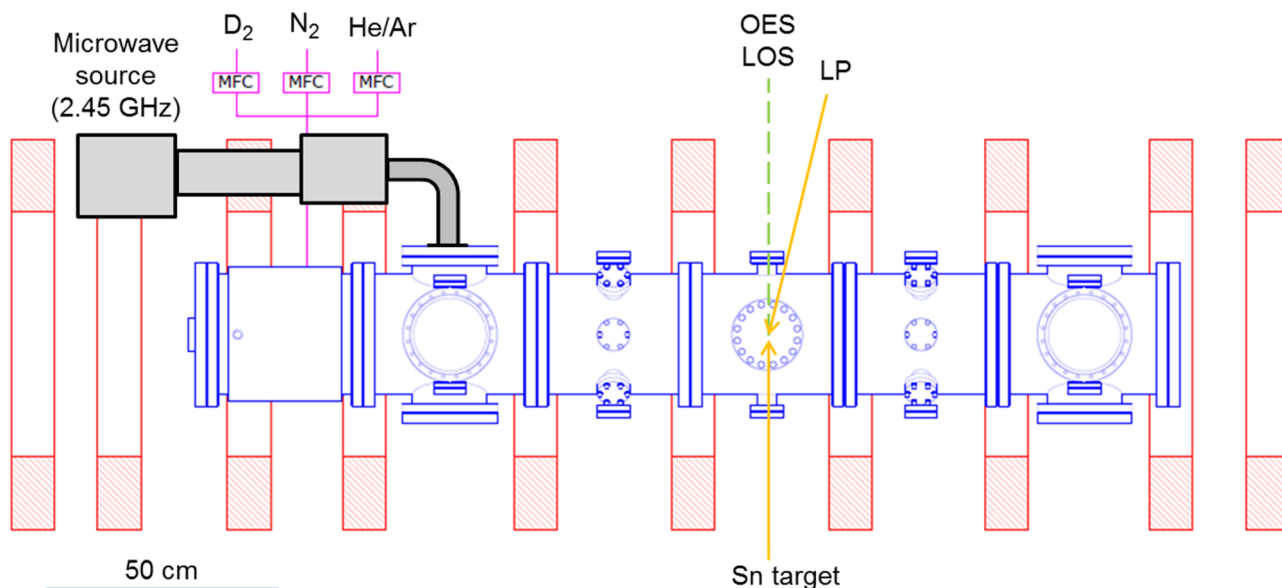


Fig. 1. Schematic of GyM plasma device.

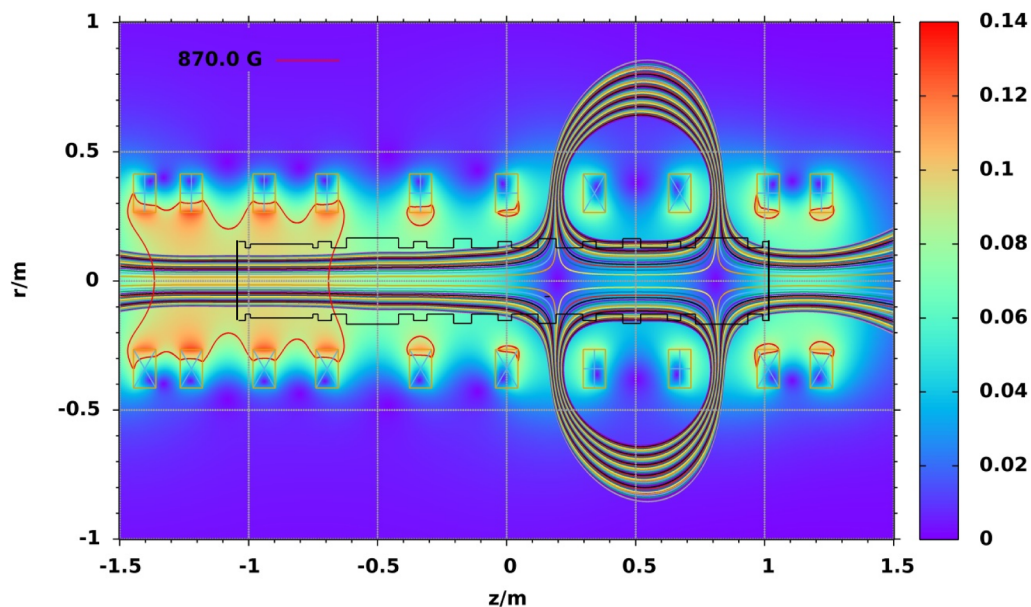


Fig. 2. Double-cusp magnetic configuration in GyM device. The red line indicates the resonance position, the black line the silhouette of the chamber. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

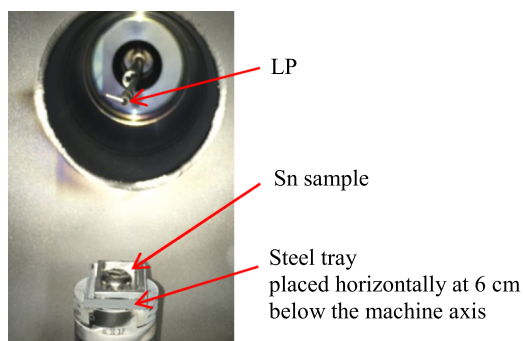


Fig. 3. Photograph of a Sn sample in GyM device.

tungsten (W) halogen lamp and a plane Lambertian diffuser in the 380–780 nm wavelength range. A Langmuir Probe (LP) is mounted on the machine in the same radial position of the Sn samples.

2.4. Ion beam diagnostics for measurements of deuterium retention

Liquid Sn samples exposed to D_2 plasma were allowed to solidify and then removed from the machine for deuterium retention studies. A sample was analyzed 6 days after plasma exposure at the Legnaro National Laboratory of the Italian Institute of Nuclear Physics (INFN) by ERDA. With this diagnostics D forward recoiled atoms, ejected from the Sn target under irradiation with a 2 MeV ^4He ion beam ($\theta_{\text{IN}} = 75^\circ$, $I_{\text{beam}} \sim 15$ nA), were detected at an angle $\theta_{\text{OUT}} = 30^\circ$. A 10 μm Mylar foil was placed in front of the solid state detector to prevent the scattered higher energy helium projectiles and heavier nuclei from striking the detector and causing degradation. Deuterium loss from the first 100 nm was estimated from the comparison between the collected spectra and the spectra simulated using SIMNRA 7 code [8].

The quantification of deuterium retained was also performed 6 months after plasma exposure at the Institute for Plasmas and Nuclear Fusion (IPFN) by NRA [9–10]. A Mylar foil with a thickness of 70 μm assured that only the protons emitted from the $^2\text{H}(^3\text{He},p)^4\text{He}$ were collected in the NRA spectra. The measure was performed at a scattering angle of 140° using a 1.0 MeV $^3\text{He}^+$ incident beam, nearby the broad-resonance energy for the reaction (~ 700 keV) [10]. The

scattering of the incident $^3\text{He}^+$ ions by the Sn nuclei (Rutherford scattering) was collected simultaneously in additional RBS [9] spectra in order to normalize the available proton yields in the NRA spectra and compute the deuterium content. Four different points with superficial diameters of 1 mm were analysed.

Ion beam analysis is also helpful to check the presence of carbon (C) in the irradiated surfaces. This is commonly performed by using proton beams with incident energies close to the resonant energy for the elastic scattering of protons by ^{12}C , $^{12}\text{C}(p,p)^{12}\text{C}$ (~ 1735 keV for the present scattering geometry of 165°) and therefore the present RBS experiment was performed with an incident proton beam of 1.75 MeV. Corresponding cross sections for the $^{12}\text{C}(p,p)^{12}\text{C}$ scattering were calculated from the SigmaCalc code [11]. All the RBS and NRA quantifications were performed using the NDF code [12].

2.5. Thermal desorption spectroscopy for measurements of deuterium retention

Preliminary TDS measurements were carried out 2 months after plasma exposure at ENEA-Frascati laboratory on a sample exposed to the same deuterium fluence than the ones analyzed at the other laboratories and on a “blank”, not exposed, sample, taken as reference sample. The vacuum chamber connected to the Quadrupole Mass Spectrometer (Balzers QMG 421) is provided with inductive heating in such a way to heat only the sample and to minimize the background effect of the chamber walls. The samples were heated up to 700 $^\circ\text{C}$ with a temperature ramp of about 6 $^\circ\text{C}/\text{minute}$. Masses 2 (H_2), 3 (HD) and 4 (D_2) were monitored.

3. Method for evaluating the S/XB spectroscopic parameter

Erosion of Sn targets due to physical sputtering by Ar plasma was quantified by mass loss measurements aimed at the empirical determination of the S/XB atomic factor [10] on solid targets. In the present experimental configuration Ar was preferred to D_2 or He due to its higher sputtering efficiency with the same projectiles energy and the same plasma parameters [13].

The double-cusp magnetic configuration was adopted in this case to reduce the intensity of the magnetic field from 0.08 T to 0.02 T in proximity of the target with the aim of increasing the Larmor radius of

Sn ions up to ~12 cm. In this way Sn⁺ ions will most likely hit the chamber wall before reaching the Sn target, thus making re-deposition negligible.

Because in GyM only a fraction of Sn sputtered atoms is ionized in the plasma, the flux of sputtered atoms can be represented as

$$\Gamma_{Sn}^{Sputt} = \Gamma_{Sn \rightarrow Sn^+} + \Gamma_{Sn}^{GL} \quad (1)$$

where $\Gamma_{Sn \rightarrow Sn^+}$ is the ionization flux and Γ_{Sn}^{GL} is the geometrical loss flux.

The ionization flux is written in terms of the S/XB factor as:

$$\Gamma_{Sn \rightarrow Sn^+} = 4\pi \frac{S}{XB} I_{SnI} \quad (2)$$

where I_{SnI} is the absolute line-integrated intensity of sputtered Sn atoms.

The sputtered flux, written as

$$\Gamma_{Sn}^{Sputt} = Y_{Sn} \Gamma_i \quad (3)$$

where Y_{Sn} is the sputtering yield and Γ_i the incident ion flux, was determined by mass loss measurements (balance precision 10⁻⁵ g).

Therefore the S/XB parameter was calculated using the following expression:

$$\frac{S}{XB} = \frac{1}{4\pi I_{Sn}} \Gamma_{Sn}^{Sputt} \left(1 - \frac{\Gamma_{Sn}^{GL}}{\Gamma_{Sn}^{Sputt}} \right) \quad (4)$$

where the geometrical correction factor was approximated by [13]

$$1 - \frac{\Gamma_{Sn}^{GL}}{\Gamma_{Sn}^{Sputt}} \approx 1 - e^{-\frac{L}{\lambda_{mfp}}} \quad (5)$$

where L is the characteristic length of the system, i.e., minimum escape length of Sn sputtered atoms and λ_{mfp} is the ionization mean free path of Sn atoms:

$$\lambda_{mfp} = \frac{v_{Sn}}{n_e \sigma v_e} \quad (6)$$

(v_{Sn} = velocity of Sn sputtered atoms, n_e = electron density,

σv_e = ionization rate coefficient). Here the energy of the sputtered atoms was assumed to be 1.8 eV, half of the surface binding energy of Sn, according to the Sigmund–Thompson transport theory [13], and n_e , T_e were estimated from spectroscopic LOS-integrated measurements. In particular n_e was obtained from the ratio of the Photon Emission Coefficients (PECs) of the Ar II lines at 480.6 nm and 488.0 nm [14]:

$$\frac{I_{480.6}}{I_{488.0}} = \frac{PEC_{480.6}}{PEC_{488.0}} \quad (7)$$

and the electron temperature T_e from the absolute intensity of Ar I line at 750.4 nm [15]:

$$I_{750.4} = n_0 n_e PEC(T_e) \quad (8)$$

with the PECs taken from ADAS database [16] and derived using the electron excitation cross-sections calculated by Griffin et al. [17] for the Ar II lines and Dasgupta et al. [18] for the Ar I line. A dedicated experimental campaign was conducted for the benchmark of the spectroscopic method with the Langmuir probe. A good agreement between the two diagnostics was obtained for T_e within 1.5 eV, while for n_e the two diagnostics differ by a factor up to 2, which is under investigation. This discrepancy has been taken into account in the calculated error bars in Fig. 6.

4. Results and discussion

4.1. Deuterium retention experiments

Sn samples were exposed to a D₂ plasma characterized by $n_e \sim 1.6 \times 10^{10} \text{ cm}^{-3}$, $T_e \sim 7 \text{ eV}$. Deuterium uptake measured by ERDA at a depth of $3 - 4 \times 10^{17} \text{ at/cm}^2$ of Sn (corresponding to 90–100 nm) is ~0.25 at%. The quantity of deuterium retained in the liquid Sn simulated layer, at a temperature of 300 °C, following the exposure to a deuterium ion fluence of $\sim 10^{24} \text{ m}^{-2}$ (2 h 15'), was $\sim 9 \times 10^{14} \text{ at/cm}^2$. Therefore the retained fraction of deuterium estimated is $\sim 10^{-5}$. An ERDA spectrum showing the presence of hydrogen in the deuterated Sn layer is presented in Fig. 4.

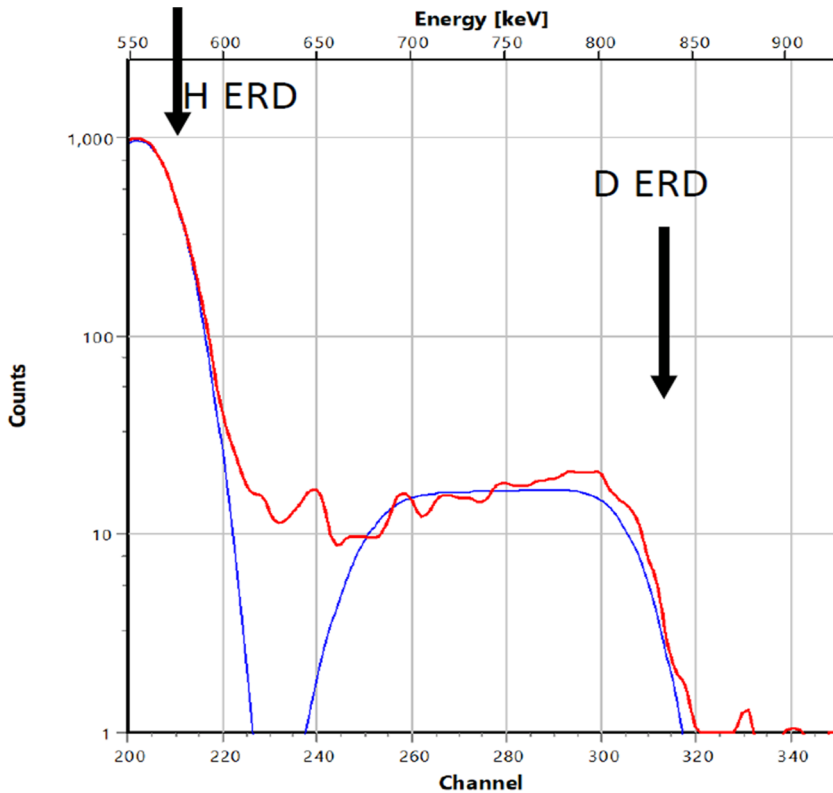


Fig. 4. ERDA experimental spectrum (only deuterium part) taken at $\theta_{IN} = 75^\circ$, $\theta_{OUT} = 30^\circ$, $E_\alpha = 2000 \text{ keV}$ using a 10 μm Mylar absorber foil. $I_{Beam} \sim 15 \text{ nA}$. Deuterium uptake calculated up to a depth of 3 to $4 \times 10^{17} \text{ at/cm}^2$ of Sn (corresponding to a maximum of about 90 – 100 nm - see simulation) is about 0.25 at%. Deuterium is clearly seen to penetrate at larger depths.

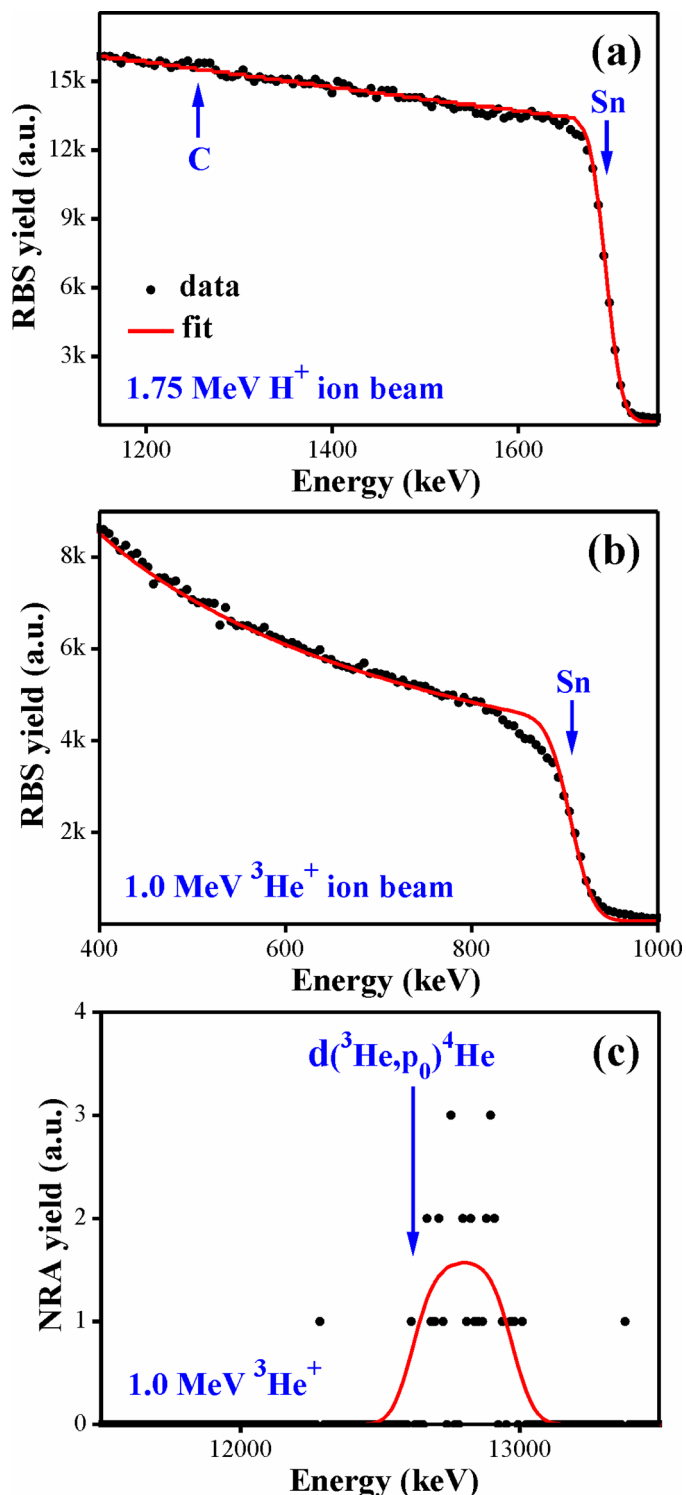


Fig. 5. (a) RBS with incident 1.75 MeV proton beam; (b)(c) RBS and NRA analyses with incident 1.0 MeV ³He⁺ ion beam, respectively.

RBS and NRA spectra are reported in Fig. 5, where the vertical arrows highlight the energies corresponding to the presence of different elements at the superficial layer: Fig. 5(a) signals the energies for superficial C and Sn in the RBS spectra collected with incident proton beams, and Fig. 5(b) and (c) show the corresponding energies for superficial Sn and deuterium in the RBS and NRA spectra collected from incident ³He⁺ beams. The analytical RBS results evidence that, if C impurity exists in the irradiated Sn surface, the corresponding amount is negligible and it should be not higher than 1-2 at%. From the NRA

spectra it is evident that very low deuterium content is present in the surface at the different analysed points. It is possible to resolve the deuterium depth profile with a fit line considering medium retained amounts of ~0.18 at% along a thickness of ~1000 nm. As example of the analytical NRA procedure, Fig. 5(b) presents the Rutherford back-scattered yield of ³He⁺ ions scattered by the Sn nuclei, used to normalize the NRA yield induced by the ²H(³He, p₀)⁴He reaction (see Fig. 5(c)). The corresponding fit lines arising from the chemical analysis are also visualised in the figures.

Because D retention estimated by ERDA (0.25 at%) 6 days after the exposure is very similar to the value obtained by NRA (0.18 at%) 6 months later, it can be argued that the samples are quite stable and the ageing effect on the measurement is negligible.

TDS measurements have shown a large amount of H₂ in both the exposed and unexposed samples, with much less Deuterium mainly detected as mass 3 (HD) probably for recombination of H and D in the vacuum chamber or in the quadrupole chamber. The value of retained deuterium obtained with TDS from the bulk sample is 1.0 × 10¹⁵ at/cm², very similar to that obtained with ERDA from a near-surface layer of 100 nm. Therefore, for the present experimental conditions, it can be deduced that deuterium is adsorbed on the surface of Sn samples and does not penetrate into the bulk.

4.2. Erosion experiments

Experimental results for S/XB of Ar I line at 380.1 nm, corrected for the geometrical loss factor obtained using plasma parameters (n_e and T_e) estimated by OES, are presented in Fig. 6. The ionization mean free path λ_{mfp} of Sn sputtered atoms ranges from 8 to 18 cm while the characteristic length of the system L was assumed to be 18.5 cm (distance of the sample from the upper wall). With these values of λ_{mfp} and L, the fraction of Sn atoms lost at the wall with respect to the total amount of Sn sputtered atoms Γ_{Sn}^{GL}/Γ_{Sn}^{Sputt} increases from 11% to 35% as T_e decreases from 9.9 to 6.6 eV. Mass loss increases from 0.9 × 10⁻⁴ to 5.6 × 10⁻⁴ gcm⁻² as T_e increases from 6.6 to 9.9 eV (samples exposure time to plasma = 12 min). The order of magnitude of the S/XB parameter obtained for Sn is consistent with that obtained for W in PISCES-B [13] and in TEXTOR [19], considering that calculated S/XB values for Sn are not available in the literature. The use of Sn, which has a lower Z than W, as PFC material is beneficial to tokamak operation. Experiments are underway to explore the possibility of measuring S/XB for different Sn lines and in a wider range of n_e, T_e for a more realistic plasma scenario.

However, a more precise estimate of n_e, T_e along the spectroscopy LOS is required to improve the evaluation of the geometrical loss flux. This can be achieved adopting more accurate theoretical calculations for deriving the PECs within ADAS, in particular using the R-matrix

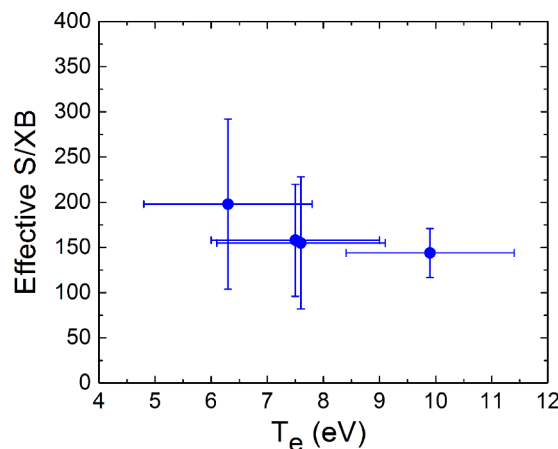


Fig. 6. Effective S/XB for Sn I line at 380.1 nm (n_e = 1.5 × 10¹¹ cm⁻³).

method (Burke and Robe [20] and references therein) to compute the electron excitation cross-sections for neutral and single ionized Argon. Moreover, a more accurate estimate of the loss flux would require the evaluation of the angular distribution of the sputtered Sn atoms. In fact the angular distribution of the eroded particles from a surface generally has a characteristic “butterfly-like” shape in polar coordinates, especially at low incident energies [21]. Therefore the loss factor may be higher than that estimated by means of the mean free path approximation used here.

5. Conclusions

Liquid Sn samples were exposed in GyM to D₂ plasma ($n_e \sim 1.6 \times 10^{10} \text{ cm}^{-3}$, $T_e \sim 7 \text{ eV}$, fluence $\sim 10^{24} \text{ m}^{-2}$ in 2 h 15') for retention measurements without Capillary Porous System. Deuterium retention was quantified by ERDA in a near-surface layer of 100 nm after 6 days from the plasma exposure and by NRA in a thicker layer of 1000 nm after 6 months. Results from the two diagnostics are in agreement, indicating that Deuterium retention in Sn is very low, i.e. 0.25 at% (ERDA) - 0.18 at% (NRA). Deuterium retention estimated by ERDA ($9 \times 10^{14} \text{ at/cm}^2$) in a near-surface layer of 100 nm was confirmed by TDS measurements in Sn bulk. Therefore, for the present experimental conditions, it can be deduced that deuterium is adsorbed on the surface of Sn samples and does not penetrate into the bulk.

Regarding the issue of plasma contamination by Sn impurities due to the erosion of PFCs, S/XB was empirically estimated by OES and mass loss measurements. The average value for the effective S/XB for the 380.1 Sn I line, evaluated in the 5–11 eV T_e range at $n_e \sim 1.5 \times 10^{11} \text{ cm}^{-3}$, was 150 ± 23 , but more effort for an accurate evaluation of Sn geometrical loss flux is required.

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