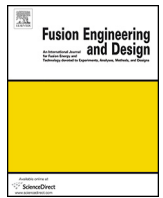




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Deuterium release from Li-D films exposed to atmospheric gases

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HIGHLIGHTS

- The major part of deuterium desorbs from Li-D films in a very sharp peak at 670–710 K.
- Exposure on air leads to intensive deuterium release from the Li-D film at room temperature.
- Interaction with water vapor plays a major role in deuterium release from lithium films in the air.

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ABSTRACT

Deuterium release from Li-D films co-deposited on a Mo substrate at room temperature in magnetron discharge was investigated by means of thermal desorption spectroscopy. The deuterium concentration in the films was estimated to be $D/Li = (14 \pm 4)\%$. TDS from Li-D films just after co-deposition had a sharp peak at 670–710 K. Exposure of deposited Li-D films in the air at room temperature led to deuterium release. Comparison of release in air, water vapor, nitrogen, and oxygen demonstrated that water plays a major role in deuterium release in the air at low temperatures.

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1. Introduction

Lithium is a promising material for plasma-facing components (PFC) in future steady-state fusion devices. It has a low atomic number, good getter properties, and can be used in a liquid state that gives an opportunity to create renewable PFC surface. Tokamak experiments have already demonstrated positive effects of lithium on plasma operation (T-11 M, NSTX, EAST, FTU, TJ-II, etc) [1]. Use of lithium increases the plasma confinement time and decreases both Z_{eff} and hydrogen recycling. Lithium was used in those experiments both for wall conditioning [2] and as the main PFC material [3,4]. Lithium PFC on the base of capillary porous systems (CPS) demonstrated a very good resistance to high heat loads: only Li atoms were sputtered and the CPS matrix was not damaged [5,6]. The gross erosion rate of Li at the high incident fluxes can be rather low due to

both the vapor shielding effect [7] and a high D concentration near the surface [8].

One of the most critical safety issues in fusion devices is tritium inventory. Hydrogen isotopes can be accumulated in liquid lithium to high concentrations [9,10]. Additionally, a large amount of hydrogen can be accumulated in co-deposits in remote areas, as it was observed for other plasma facing materials [11,12]. Therefore, behavior of H in Li-H deposits is important.

This work is devoted to investigation of D release from co-deposited Li-D films during their storage in the air, water vapors, nitrogen gas, and oxygen gas.

2. Experimental

Lithium is very active chemically. Therefore, deposition and further analysis of lithium films must be done without contact of the film with air [13]. The experimental installation MD-2 was used for this purpose. It consists of two separately pumped vacuum chambers: a deposition chamber and a TDS chamber. The lithium film was deposited on the molybdenum substrate ($10 \times 10 \times 0.2$ mm) inside the deposition chamber shown in Fig. 1. The substrate

Abbreviations: PFC, plasma facing components; TDS, thermal desorption spectroscopy; CPS, capillary porous system; QMB, quartz microbalance; QMS, quadrupole mass-spectrometer.

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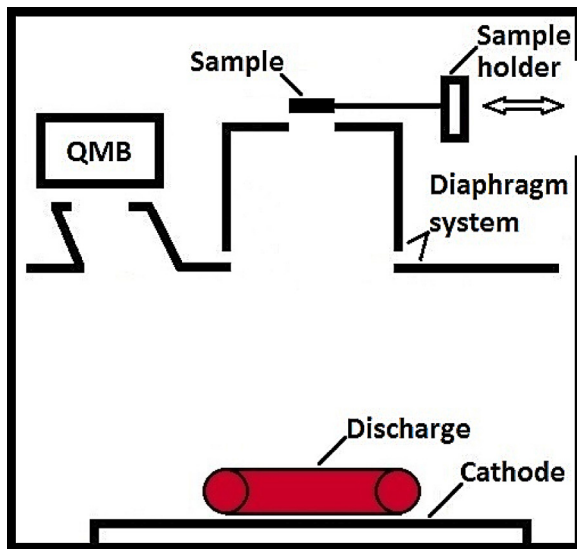


Fig. 1. The scheme of the deposition chamber.

mounted on the sample holder can be moved between the chambers by a feed-through (see details in [13]). The mass of the film during deposition and during exposure in gas was monitored by a quartz microbalance (QMB). Fig. 1 shows also a shielding system that makes possible deposition of Li only on the sample and the QMB crystal. The temperature of the sample was monitored during the deposition, and it increased steadily from room temperature to about 70 °C due to radiative heat from the discharge.

Since the QMB crystal and the sample were placed not in the same place, the correction factor has been measured in a separate series of experiments using the Rutherford backscattering spectrometry. The amount of lithium on the sample was two times higher than that on the QMB. This was then taken into account when calculating the deuterium concentration in the films.

The residual gas pressure in the chamber was below 10^{-4} Pa. However, the residual gas pressure dropped below 3×10^{-5} Pa after the start of lithium target sputtering due to gas sorption by lithium on diaphragms.

In several experiments, individual lithium pieces placed on the molybdenum magnetron cathode were the target for sputtering (similar to [13]). In other experiments, a capillary porous system (CPS) was used as the target. CPS was made as a stainless steel bath (5 cm in a diameter, 5 mm height) with several stainless steel meshes inside, which were saturated with lithium. For storage and transportation before use, the bath was covered by a protective stainless steel foil (like in [14]), which was removed just before installation the bath in vacuum. This cathode can be used for many deposition cycles without opening the deposition chamber. In both configurations (CPS and Li metal pieces), lithium was melted at the early stage of the discharge.

The residual gas pressure in the TDS chamber was below 10^{-6} Pa, and it was separated from the deposition chamber by a gate valve during TDS analysis and deposition. Gas release during TDS was monitored by a quadrupole mass-spectrometer (QMS). Gas composition in the deposition chamber during the discharge was also measured through a calibrated leak between the chambers (see details in [13]).

The multi channel gas leak system was assembled to inject various gases from the gas bottles through gas flow controllers into the vacuum chambers and to calibrate QMS with respect to various gases. The calibration of all gas flows in the TDS chamber was also done using two additional leak valves and a baratron capacitance manometer (see [15] for details of the calibration procedure).

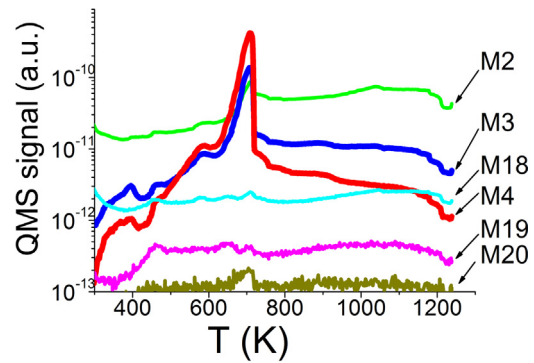


Fig. 2. Typical TDS spectra of various masses from a Li-D film.

Before deposition, the substrate was always outgassed at 1100 K in the TDS chamber to reduce the background during TDS. The temperature of the sample was monitored using a thermocouple spot welded to the sample, and the heating rate during TDS was 2 K/s. Linearity during heating was ensured by using a feedback control system.

3. Deuterium retention in lithium films

Our preliminary experimental estimations of deuterium retention in Li-D films gave the D concentration of about 5–20 at.% [13]. However, the Li amount in the films was measured rather roughly from the film thickness obtained by SEM. In this work, the Li amount was calculated from the QMB data. Additionally, vacuum conditions and reproducibility were improved with respect to previous experiments thanks to the CPS cathode. The CPS system worked very stable; there were no indication of matrix sputtering when lithium was in the bath. Only when all Li was spent, and the bath was empty, upper layers of CPS stainless steel mesh started to melt.

According to QMB measurements, the surface density of the deposited films was in the range of 50–250 ng/cm² (approximately 100–500 nm, assuming the density of 0.5 g/cm³). The deposition rate was about 2 nm/s (~13 monolayers per second), that is much higher than the flux of impurities from the residual gas to the sample. Therefore, one can expect a high purity of the lithium film in the bulk. On the other hand, the top surface could be modified by impurities rather quickly after the end of the deposition due to relatively high residual gas pressure in the deposition chamber. According to [16], the oxidation of the top surface occurs already after exposure of 10 Langmuirs (1 Langmuir = 1×10^{-6} Torr × s).

Typical TDS spectra of various masses from Li-D films are shown in Fig. 2. One can see that the main part of deuterium desorbed in a very sharp peak. The peak position varied slightly from run to run in the range of 670–710 K. Deuterium desorbed mainly in the form of D₂ and HD molecules. This is in good agreement with our previous experiments [13], as well as with other experiments performed in laboratory conditions [17,18] and in the tokamak T-11 M [3,19].

The amount of deuterium released at temperatures above the main peak was small, but not zero in all experiments. In some early experiments, an additional peak at 850 K was observed [13], and in many, but not all, our current experiments, a plateau in the range of about 700–900 K was observed as that in Fig. 2. The integral deuterium release at these high temperatures was up to 10% of total release in some experiments. High temperature D release was also observed in [20] after irradiation of liquid lithium with deuterium plasma and in [21] after deuterium ion implantation in lithium.

The total deuterium retention in the films was calculated from the integral of TDS spectra for D₂ and HD molecules. The amount of Li in films was calculated from QMB data. Finally, the deuterium concentration was calculated to be D/Li = (14 ± 4) %. For example,

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