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Time-resolved studies of deuterium release from lithium films exposed to water vapor

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HIGHLIGHTS

- Li-D films rapidly react with water vapor at room temperature, increasing in mass 3 times and fully transforming into LiOH.
- The dynamics of the film's mass can be well described using a diffusion driven model for the first 70% of the reaction and combined nucleation-diffusion based model for the whole reaction.
- D remaining in the film after the exposure to water vapor is released in the form of hydrogen and water molecules at low temperatures up to about 540 K.

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ABSTRACT

In continuation of a previous study of Li-D films' interactions with atmospheric gases, dynamics of Li-D codeposit interaction with water vapor was studied. Li-D codeposits very actively react with water vapor, leading to formation of LiOH and release of major part of deuterium. The dynamics of the film mass (thickness of the reacted layer) was well described using a diffusion-based model at the early stage of the reaction and using a combined nucleation-diffusion based model in whole period of time. The most of D remaining in the film after exposure to water vapor is released during a heatup below 550 K, that is attributed to LiOH decomposition and occurs at lower temperatures than in the case of initial Li-D film.

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1. Introduction

Lithium is considered in fusion devices as a part of liquid first wall technological approach [1]. Use of plasma facing components based on liquid lithium [2–4] demonstrated positive effects on plasma operation, such as an increase in confinement time, and a decrease in Z_{eff} . Using lithium also decreases the hydrogen recycling coefficient, that is promising for achievement of special regimes of plasma operation [1]. However, this may also raise the problem with hydrogen isotope accumulation and removal.

Liquid lithium is chemically very active material and can accumulate a large amount of hydrogen isotopes up to H:Li = 1:1. Li-H codeposits will be also formed during operation with liquid lithium components in remote areas and amount of hydrogen in codeposits can also be high. This can be a problem, especially, in the case of steady state operation. Additionally, a controlled redeposition of

lithium from the wall on the cryogenic lithium collectors is also considered as a part of lithium circulation cycle [5]. As such, quick and energy efficient methods of hydrogen removal from lithium codeposits are an important topic of investigation.

On the other hand, uncontrolled release of hydrogen isotopes from Li-D codeposits can be undesirable. It can represent radiological danger during maintenance, and negatively affect hydrogen recycling during operation.

There are several probable methods for hydrogen isotope removal from Li-H codeposited layers in fusion devices: heating, isotope exchange and chemical reactions with gas. Previous research has shown that the main part of D is released from Li-D codeposits at around 700 K [6,7]. It has also been shown that D is quickly released from the codeposits at room temperature when they are exposed to atmospheric air [8], which can lead to dangerous release of radioactive tritium when Li-containing installations are opened up for maintenance purposes, and could affect such installation parameters as hydrogen recycling in the case of atmospheric leaks. It has also been shown that the reaction with water vapor is most intensive and plays the major role in deu-

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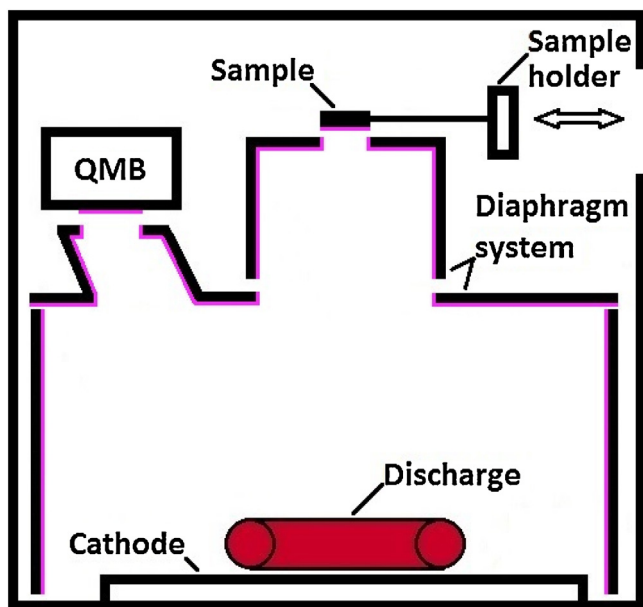


Fig. 1. A scheme of the MD2 deposition chamber. Pink areas indicate locations where Li film can be deposited.

terium release at air conditions [9]. Recently, it was also shown by [10]. Products of such interaction release deuterium at lower temperatures than normal Li-D codeposits do [8,9]. However, the mechanisms of this reaction, and its kinematics have not been fully understood yet. The Li-D codeposits interaction with water vapor is, thus, a relevant topic.

As a continuation of the research described in contribution [8], this paper presents a more detailed time-resolved investigation of D release from Li-D codeposits exposed to water vapor, as well as analysis of Li-D – water vapor reaction kinetics and mechanisms, using time resolved mass spectroscopy measurements and mass measurements with the quartz microbalance (QMB) system, which were not shown before. The properties of the Li-D – water vapor reaction solid product are studied with thermal desorption spectroscopy (TDS).

2. Experimental details

All experiments were done in the multi functional installation MD-2, which consists of two separately pumped vacuum volumes: the deposition chamber and the TDS chamber. The gate valve separates the chambers from each other, and the sample transfer is realized using the linear feedthrough [7]. Therefore, in-vacuo TDS analysis of the deposited films can be performed.

A planar magnetron with the molybdenum cathode plate is installed at the bottom of the deposition chamber (Fig. 1). To sputter lithium in the discharge, a piece of lithium about 0.5 cm^3 large was placed on the cathode (see [7] for details). The residual gas pressure was $3 \times 10^{-5} \text{ Pa}$. During the discharge, deuterium with the purity of 99.98% was admitted to the deposition chamber up to the pressure of about 4 Pa. During first seconds of the discharge, the Li piece was melted, and lithium spread over the sputtering area of the cathode. A molybdenum sample (1 cm^2) was mounted on the sample holder in front of the cathode at the distance of 10 cm. To minimize the risk of any part of the holder except the sample itself being exposed to Li deposition flux, the sample was surrounded by the system of diaphragms (Fig. 1). The color of the deposition area was also visually controlled through the window.

A quartz microbalance (QMB) system is installed in the deposition chamber to control the thickness of deposited layers and to

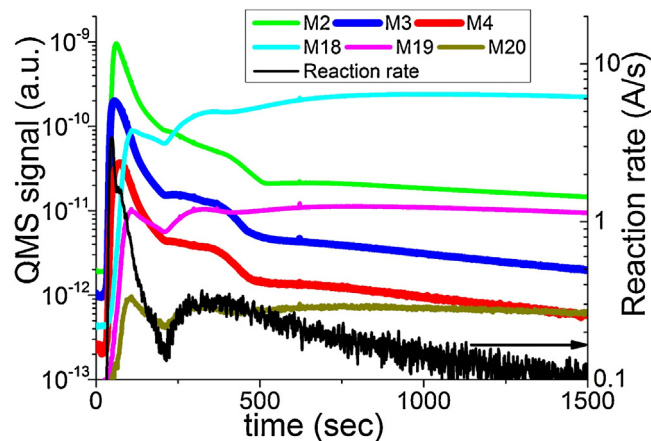


Fig. 2. Partial pressure of various gases during interaction of the 100 nm Li-D film with water vapor at room temperature and at the pressure of 1 Pa. Right Y axis indicates the reaction rate for the film on the QMB calculated in angstroms per second, and shown in a black line.

investigate increase of the film's mass during exposure to various gases (see [8] for details).

The thickness of the deposited film was about 100 nm on the molybdenum sample used for TDS analysis, and about 50 nm on the QMB crystal used for reaction rate monitoring. The deposition rate was about 2 nm/s for the experimental sample and 1 nm/s for the QMB crystal. The difference was due to different locations of the QMB monitor and the experimental sample relative to the magnetron. Due to the high deposition rate, contaminations of the film from residual gases are expected to be low, and exist only in the top surface layer.

The thermochemical water vapor source (TWVS) based on $\text{Ca}(\text{OH})_2$ thermal decomposition in vacuum was used to generate the water vapor flux, similar to that used in [11,12]. It consisted of a quartz tube with about 2 cubic centimetres of $\text{Ca}(\text{OH})_2$ tightly packed in it. When heated using the tungsten wire surrounded the quartz tube, $\text{Ca}(\text{OH})_2 = \text{CaO} + \text{H}_2\text{O}$ decomposition reaction took place, and the water vapor flux released.

When the water vapor is admitted into the experimental chamber, it reacted simultaneously with the films on the experimental sample, QMB crystal, and the diaphragms (all areas are indicated in Fig. 1)

A quadrupole mass spectrometer is installed in the TDS chamber to monitor partial pressures of various masses desorbed from the sample during TDS analysis and to monitor the gas composition in the deposition chamber. The sample was linearly heated during TDS with the rate of 2 K/s by radiation from the U-shape heater made of the tungsten foil. The maximal temperature was 1300 K. The temperature of the sample was monitored using a K-type thermocouple mounted on the back side of the sample. The residual gas pressure in the TDS chamber was below 10^{-6} Pa . An additional vacuum volume with the baratron vacuum gauge and two leak valves is connected to the TDS chamber to calibrate QMS using the procedure similar to [13].

3. Experimental results

Exposure of the lithium film to the water vapor was done at the pressure of about 1 Pa for six hours. During this time, the heater in TWVS was kept at the constant power. This gives a nearly constant flux of the water vapor during several hours, according to previous experiments. During this time, the mass of the film was monitored using QMB. Fig. 2 shows (right Y axis) time resolved measurements of films mass growth rate, calculated in angstrom

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