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Interaction of Li-D films with water vapor

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Abstract

In this work we investigated co-deposition of lithium-deuterium (Li-D) films in magnetron discharge and interaction of deposited films with water vapor. It was demonstrated that even at room temperature interaction with water vapor led to release of a large portion of deuterium from the deposited layers. After 30 minutes exposure at the pressure about 10^{-1} Pa the main part of deuterium released from the sample and a characteristic peak for deuterium desorption from lithium films completely disappeared.

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1. Introduction

A choice of plasma-facing materials is one of the key issues in fusion reactor design. Lithium as an element with the low atomic number and a good compatibility with fusion plasma is a promising material for plasma-facing components (PFC) in fusion installations. A number of experiments already demonstrated many positive effects of lithium on plasma operation, such as increase in plasma confinement time [Zuo et al. (2013)] and decrease in hydrogen recycling [Tafalla et al. (2011)]. Lithium was used for wall conditioning [Sugai et al. (1996)] or for lithium based PFC [Mirnov (2009), Vertkov et al. (2012)]. In any way, one can expect lithium co-deposition with hydrogen

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isotopes at the surface of PFC and at remote areas. Lithium as a good getter can accumulate a high amount of hydrogen isotopes (deuterium, tritium) which can be a problem from the safety point of view. Deuterium retention and thermal desorption from lithium films formed in plasma discharge conditions were investigated in this work.

Deuterium accumulation in lithium films has been investigated in our previous work [Krat et al. (2014)]. The films were deposited in magnetron discharge in deuterium with lithium cathode. The deuterium content in the films was up to 20 atomic percents. Thermal desorption spectroscopy (TDS) spectra of Li-D films showed two sharp peaks of D_2 (4th mass) and HD (3rd mass) desorption with maxima at 710 K and 850 K, and the first peak was always dominant. TDS spectra containing two peaks at similar temperatures were observed also by Martín-Rojo et al. (2014), Furuyama et al. (2003) and Capece et al. (2015). Additionally, our previous experiments demonstrated deuterium release from lithium films during air exposure. There was no deuterium detected in the films after three days of air exposure. This work describes further investigations of interaction between Li-D films and water vapor in order to clarify mechanisms of low-temperature deuterium desorption.

2. Experimental setup

All experiments have been done at the multifunctional setup MD-2. This setup consists of two vacuum chambers with separate pumping systems. Deposition of Li-D films in magnetron discharge is performed in the first one (deposition chamber). A piece of lithium is placed on the top of a molybdenum target plate for lithium sputtering in deuterium plasma. Thermal desorption spectroscopy of Li-D films is performed in the second chamber (TDS chamber). The sample can be moved from one chamber to another using a linear feedthrough without air exposure of the samples. Gas composition in both chambers can be monitored by the quadrupole mass-spectrometer Extorr 100M (QMS). Further details of the setup can be found in the work of Krat et al. (2014).

For experiments presented in this paper, a new fully-metal gas inlet system was constructed. This system allows to inlet up to five different gases. The gas inlet system is connected to the TDS chamber through a high precision leak valve and to the deposition chamber through two mass flow controllers installed in parallel. A quadrupole mass spectrometer calibration system similar to the one shown in the work of Rusinov et al (2009) was integrated into the gas inlet system.

To inlet water vapor a thermochemical water vapour source (TWVS) based on calcium hydroxide decomposition [Kurnaev et al. (2013) and Voronov et al. (2012)] was used. The principle of TWVS operation is based on the absorption of water molecules by calcium oxide crystals and their desorption by calcium hydroxide crystals $Ca(OH)_2 \rightleftharpoons CaO + H_2O$. As the temperature increases, the pressure of water vapor in the presence of calcium oxide and hydroxide crystals grows rapidly. Pressures in the range of 1–10 Pa can be obtained by a moderate heating of calcium hydroxide crystals to a temperature of about 450 K. The TWVS consists of a small capsule containing fine $Ca(OH)_2$ powder, an electrical heater, and a thermocouple for the temperature measurements

The system of diaphragms in the deposition chamber was also modified to improve protection of the sample holder against particle flux from plasma. The cathode of the magnetron was covered by a molybdenum plate with a freshly cleaned piece of lithium placed on the top (see [Krat et al. (2014)] for details).

3. Equations

3.1. Interaction with atmosphere

Lithium-deuterium films were exposed to atmosphere in the deposition chamber for three days. Afterwards, the deposition chamber was pumped down, the samples transferred to TDS chamber, and TDS was conducted. No deuterium was detected. The film was not evaporated completely, like in the case of pure lithium experiments. Because it was possible that deuterium remained in the film in the form of a chemical compound with high decomposition temperature, nuclear reaction analysis of the film was conducted. Precise measurements were not possible in this experiment [Krat et al. (2014)], however, it was concluded that there was only a little number of deuterium ($D/Li < 0.03$), if any, in the film.

At room temperature lithium is supposed to react with all main components of atmospheric air: oxygen, nitrogen, water vapor. Water is supposed to be the most reactive of those, also serving as an accelerant for the lithium-nitrogen reaction. Therefore, lithium film interaction with water vapor was studied in more details.

3.2. Interaction with water vapor

The deposited in magnetron discharge Li-D films (about 2 μm thick) were exposed in the deposition chamber, where a gas (water vapor) was admitted. In this case gases interacted not only with the sample, but also with lithium films deposited on the surrounding walls, which increased overall sensitivity of the dynamic measurements.

Interaction of the Li-D film with water vapor inside the deposition chamber at the pressure about 10^{-1} Pa is illustrated by Fig.1. Immediately after water vapor admission one can see an intensive release of hydrogen isotopes. The water pressure grew with a significant delay. This is a clear evidence of active water sorption by lithium and intensive reaction of lithium with water molecules resulting in hydrogen release. Deuterium desorption started also immediately after water vapor admission. Deuterium desorption started mainly in the form of HD and D_2 molecules, however a significant amount of heavy water molecules was observed later. The temperature of the substrate (200 μm molybdenum plate) was monitored by the thermocouple welded to the back side, and the temperature increase was not observed during exposure.

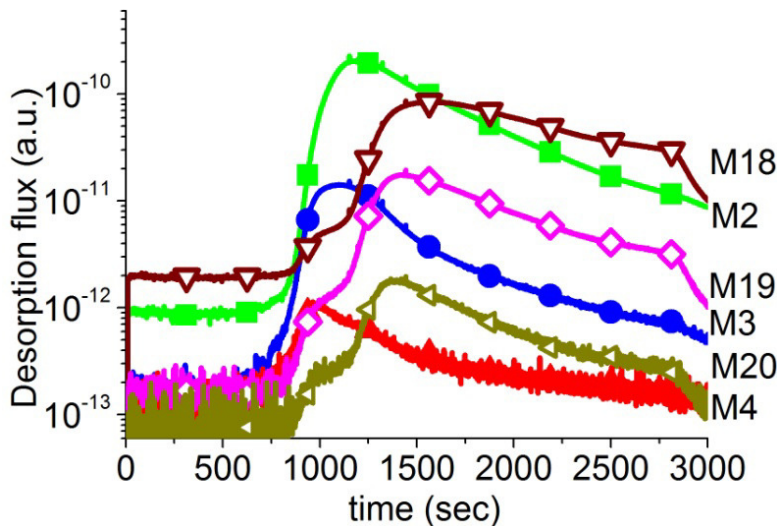


Fig. 1. Fluxes of hydrogen isotopes containing compounds released from Li-D film at the deposition chamber walls during water vapor admission versus time.

After exposure in water vapor the sample was moved to the TDS chamber to examine the deuterium amount in the films. Fig. 2 presents TDS spectra of 4th mass for experiments with immediate TDS, TDS after 3 day long atmospheric exposure and TDS after 30 minutes water vapor exposure. One can see that the short time exposure to water vapor led to release of the main part of deuterium. The typical for lithium films peak at 700 K completely disappeared. However, there was still small amount of deuterium in the sample desorbed mostly at low temperatures, below 600 K. Therefore, one can conclude that interaction of Li-D with water vapor leads to release of the main part of deuterium accumulated in the film during deposition even at the room temperature.

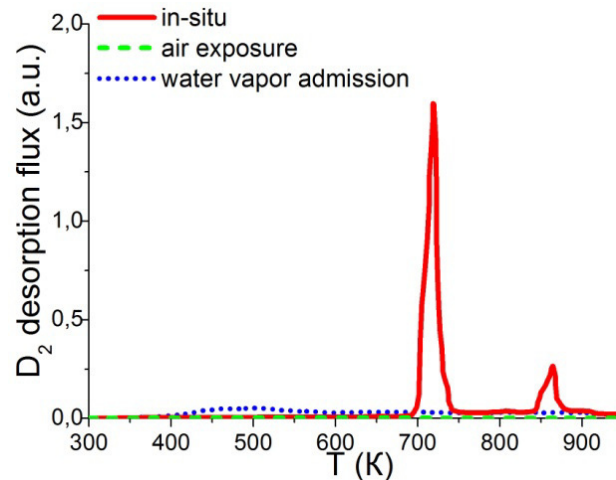


Fig. 2. TDS spectra of 4th mass: red line - immediate TDS, green dashes - after atmospheric exposure for 3 days, blue dots- after interaction with water vapor (30 minutes).

4. Summary

Interaction of Li-D films deposited in magnetron discharge with water vapor has been investigated in this work. It was shown that already after 30 minutes exposure at the pressure of 10^{-1} Pa at room temperature the main part of deuterium released from the lithium film. The remained part of deuterium released mainly below 600 K and the characteristic peak around 700K completely disappeared.

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