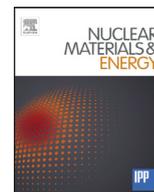




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## Nuclear Materials and Energy

journal homepage: [www.elsevier.com/locate/nme](http://www.elsevier.com/locate/nme)

# Dissolution behavior of lithium compounds in ethanol

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## ARTICLE INFO

## Article history:

Received 29 October 2015

Revised 20 April 2016

Accepted 17 May 2016

Available online xxx

## Keywords:

IFMIF

Lithium target

Components cleaning

Components replacement

## ABSTRACT

In order to exchange the components which received irradiation damage during the operation at the International Fusion Materials Irradiation Facility, the adhered lithium, which is partially converted to lithium compounds such as lithium oxide and lithium hydroxide, should be removed from the components. In this study, the dissolution experiments of lithium compounds (lithium nitride, lithium hydroxide, and lithium oxide) were performed in a candidate solvent, allowing the clarification of time and temperature dependence. Based on the results, a cleaning procedure for adhered lithium on the inner surface of the components was proposed.

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## 1. Introduction

A key step toward the realization of a demonstration fusion reactor is to develop structural materials that can withstand the 14 MeV neutrons generated as a result of the deuterium–tritium fusion reaction [1]. In order to establish an irradiation facility for candidate materials, the International Fusion Materials Irradiation Facility (IFMIF) project is in progress under the Broader Approach Agreement [2,3] to support the materials development programs by providing a fusion-relevant neutron source in which candidate material irradiation performance can be fully assessed. Because the IFMIF is an accelerator-based neutron source that utilizes the deuterium–lithium stripping reaction, the target assembly (T/A), which is the main component, undergoes significant irradiation damage. Therefore, the exchange of the T/A must occur during the in-service inspection [4].

Here, the structure of the T/A is described briefly. The T/A of the IFMIF will be made from ferritic-martensitic steel such as EUROFER and F82H steel. In the T/A, liquid lithium screen flowing at a speed of 15 m/s and a temperature of 250 °C will be formed for the stripping reaction. Since liquid lithium is used as the target material, it exists in the inner surface after operation as the contamination.

Cleaning of the adhered lithium on the inner surface of the removed T/A is required prior to long-term storage. In a previous study [5], we performed dissolution experiments of pure lithium in candidate solvents such as ethanol, pure water, and an ethanol–

water mixture, allowing the clarification of temperature and time dependence.

However, lithium adhering to the T/A is able to react with the atmosphere during the exchange and long-term storage. Therefore, we investigated the chemical reaction of pure lithium at room temperature in air, oxygen, nitrogen, and argon atmospheres under varying humidity to estimate the chemical reaction that occurs during the exchange [6].

Based on these results, we herein describe the dissolution behavior in a candidate solvent of lithium compounds formed upon reaction with the atmosphere during the exchange.

## 2. Experimental procedure

### 2.1. Reagents

For the experiments, lithium nitride ( $\text{Li}_3\text{N}$ ), lithium hydroxide ( $\text{LiOH}$ ), and lithium oxide ( $\text{Li}_2\text{O}$ ) were prepared as the reagents. These shape before the experiments are shown in Fig. 1.

$\text{Li}_3\text{N}$  and  $\text{LiOH}$  were synthesized from the pure lithium of the sphere with a diameter of 10 mm through reaction with moisture-containing nitrogen or argon at 80 °C for 7 days and 80 °C for 12 days, respectively. Each shape after the synthesis was upholding the original sphere shape. Although we tried the same synthesis technique also to  $\text{Li}_2\text{O}$ , it was brittle and the cracks were generated during synthesis. Then, we used  $\text{Li}_2\text{O}$  powder of the commercial reagents produced by Soekawa Chemical Co. Ltd. and the reagent for the experiments was manufactured by compression molding using a hydraulic pressure system, resulting in a cylindrical shape with a diameter of 12 mm and a height of 10 mm.

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<http://dx.doi.org/10.1016/j.nme.2016.05.005>

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Fig. 1. Shape of the synthesized reagents (before immersion into ethanol).

Table 1  
Results of X-ray analysis of the synthesized reagents.

Reagent	Sampling position	Compounds (ICDD no.)	Traces
Li <sub>3</sub> N	Surface	Li <sub>3</sub> N (76-0821)	–
	Inside	Li <sub>3</sub> N (76-0821)	Li <sub>2</sub> O, LiOH, Li <sub>2</sub> NH
LiOH	Surface	LiOH (85-0736)	–
	Inside	LiOH (85-0736)	–
Li <sub>2</sub> O	–	Li <sub>2</sub> O (12-0254)	LiOH

After the manufacturing process, the samples were removed and the synthesized reagents and crystal structures were analyzed by X-ray diffraction (XRD), as summarized in Table 1.

## 2.2. Apparatus

A schematic representation of the experimental apparatus is shown in Fig. 2.

These devices, which comprise an exposure pot, a micro balance, and a vacuum chamber, were installed in the argon

Table 2  
Experimental conditions.

Reagent	Solvent	Temperature (°C)	Exposure time (h)
Li <sub>3</sub> N	Ethanol	10, 30, 50, 70	~7
LiOH			~17
Li <sub>2</sub> O			~30

glove box. The Pyrex glass exposure pot was connected to a constant temperature water bath system, allowing the temperature of the ethanol to be controlled with the combined use of a stirrer. The reagent was set in a stainless steel mesh container and temporarily removed during weight change measurements using a microbalance. The vacuum chamber was also used for the evaporation of ethanol in the sample prior to the measurement.

## 2.3. Experimental procedure

The experimental conditions and procedures are summarized in Table 2 and Fig. 3, respectively.

Lithium compounds (Li<sub>3</sub>N, LiOH, and Li<sub>2</sub>O) were immersed in ethanol (99.5% purity) at 10 °C, 30 °C, 50 °C, and 70 °C, with maximum exposure times of 7, 17, and 30 h, respectively. As described above, the reagents were extracted from the exposure pot during the experiment and the weight and size of the samples were measured after the evaporation of ethanol. The sampling step for weight measurement was approximately 1 h.

The dissolution progress of the samples in ethanol was measured using the weight and outer diameter.

## 3. Results and discussion

### 3.1. Dissolution behavior of lithium compounds in ethanol

A photograph of the lithium compounds immersed in ethanol at 30 °C is shown in Fig. 4 as an example.

Although Li<sub>3</sub>N was dark brown prior to the examination, the surface became partially white after the immersion. The surfaces of LiOH and Li<sub>2</sub>O did not change from their original colors of white and gray, respectively, upon immersion. All reagents were reduced to similar figures with time at each temperature. No exfoliation was observed for any of the reagents upon ethanol immersion.

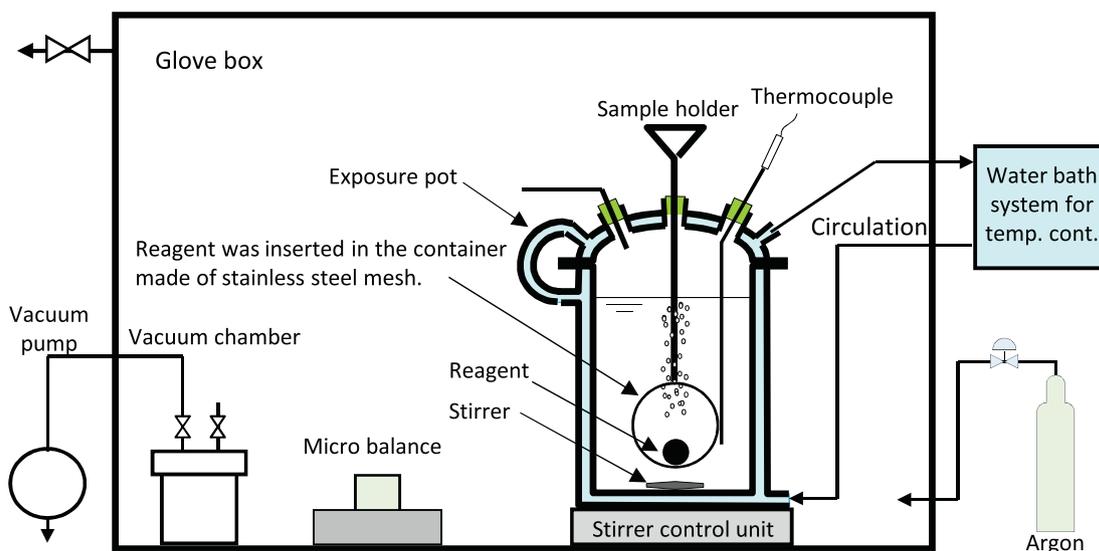


Fig. 2. A schematic representation of the experimental apparatus.

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