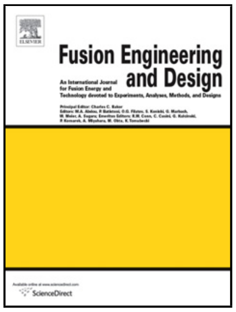

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Electrochemical extraction of hydrogen isotopes from Li/LiT mixtures

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Draft Research Highlights:

1. Described potential methods to simplify tritium removal from Li blankets in fusion machines through direct electrolysis of lithium tritide (LiT).
2. Proposed an electrochemical cell using a solid Li-ion conductor immersed in molten lithium metal containing LiT to perform direct electrolysis of LiT.
3. Synthesized and tested the conductivity of lithium lanthanum zirconate (LLZO) pellets for direct LiT electrolysis.
4. Constructed a cell for electrolysis of LiD from a Li/LiD mixture for demonstration of the feasibility of the direct electrolysis of LiT.
5. Demonstrated the ability to electrolyze LiD through the detection of hydrogen and deuterium gas by mass spectrometry.

Abstract

In this study, we propose a new methodology for lithium hydride electrolysis which can be extended for the extraction of hydrogen isotopes from molten lithium. This new process utilizes a ceramic lithium ion conductor that can be directly immersed in molten lithium to facilitate the electrochemical extraction of hydrogen isotopes in the molten state. The extraction of hydrogen isotopes from lithium is necessary for the safe and reliable operation of fusion reactors that utilize molten metals as a coolant and/or as tritium breeder blanket in the system. A number of technologies for hydrogen isotope extraction from molten lithium have been proposed that can facilitate the extraction of hydrogen isotopes. However, they require a series of complicated mechanical steps (expensive mechanical parts with limited lifetimes) in order to carry out the separation and prevent impurities build up in the extraction process. Our proposed electrochemical process can potentially simplify the isotope extraction process by eliminating many of the mechanical parts and being able to integrate this technology within the fusion reactor to perform the extraction in-line. To determine the feasibility of this process, a proof of concept was demonstrated experimentally utilizing a solid state lithium ion conductor to facilitate the electrochemical extraction of hydrogen and deuterium from lithium in the molten state. The extraction of hydrogen and deuterium from molten lithium was verified during this experiment and indicates this new process is a potential alternative for other processes used for the extraction of hydrogen isotopes. Process modeling was also performed to understand the energy requirements and electrode areas needed for decomposing different concentrations of tritium.

Keywords: hydrogen isotope extraction, electrolysis, solid state electrolyte, LLZO, molten lithium, fusion energy

Introduction

The successful demonstration of a fusion device that can demonstrate net energy production for consumer use would be a significant step toward eliminating the use of fossil fuels for power generation. In this technology, the biggest emphasis has been placed on understanding and fine-tuning the fusion process to generate the heat need to produce power. However, there are many other auxiliary components in the fusion reactor that need improvement in order to achieve safe and reliable operation of a fusion based power plant. One of the auxiliary components is the breeder blanket which can also serve as the cooling blanket surrounding the reactor. Combining both functions into one component simplifies the reactor design. In the deuterium-tritium (DT) fusion reaction, high energy neutrons are released along with helium atoms. The neutrons escape the magnetic fields that confine the plasma and are absorbed by the liquid metal blanket covering the surrounding walls. If the liquid metal cooling the reactor has a high neutron cross section, it can attenuate the neutron effectively. For example, if the blanket module contains lithium, a reaction occurs: the incoming neutron is absorbed by the lithium atom, which generates an atom of tritium and an atom of helium. Because this process produces tritium external to the magnetic containment system, the tritium must be removed in order to keep the inventory of tritium low (~ 1 appm) to ensure safe operation of the reactor in case of a breach of the molten blanket system.[1] If the molten blanket is operated at a temperature of 500°C, the vapor pressure of tritium above the molten lithium is only 3.4×10^{-9} Pa.[2] This could result in a tritium inventory well above the desired 1 appm in the blanket. Due to the extremely low vapor pressure of tritium above lithium at this temperature, it cannot simply be recovered by applying a vacuum to collect it from the blanket head space. This problem has resulted in the investigation of other methodologies that can effectively limit the tritium inventory in the blanket/breeder system in a molten lithium blanket. Some of the proposed processes are molten salt extraction of LiT followed by electrolysis (Maroni Process),[3] "gettering",[4] permeation window,[5] fractional distillation,[6] cold trapping,[7] and a combination of all. While many of these technologies have demonstrated the viable extraction of hydrogen isotopes from a molten lithium blanket, they typically require multiple processing steps to achieve the extraction. The extraction also includes the use of specialized mechanical equipment that would be in contact with highly corrosive materials, at high temperatures, in a high radiation environment for many years during the reactor operation.

In our proposed process, we intend to leverage the recent advances in solid ionic conductors for lithium ion batteries to facilitate the extraction of hydrogen isotopes from a molten lithium blanket through direct lithium hydride electrolysis. Lithium ion ceramic conductors have been studied for more than 40 years for use in all solid-state batteries. While most of the research has focused in temperature ranges below 300 ºC for civilian applications, military applications that make use of high temperature batteries has pushed for the development of lithium ion ceramic conductors at high temperatures. Solid electrolyte candidates must be stable at high temperatures (~600 ºC) in contact with molten Li and Li alloys, and have negligible electrical conductivity but high ionic conductivity (> 0.1 S/cm at 400 ºC).[8] While there are several lithium ion conductors that possess most of the required qualities, such as LISICONs, NASICONs analogs, perovskites, garnets, and LIPONs; only garnet-type of electrolytes seems to be able to maintain its electrochemical stability and performance at high temperatures and in the presence of molten Li.[9] Garnets are capable of accommodating an excess of lithium cations beyond that normally found in this prototypical structure. This excess lithium is found in a mixture of coordination environments with considerable positional and occupational disorder and leads to ionic conductivity of up to 4×10^{-4} S/cm at room temperature.[10] This high value for total conductivity, combined with excellent thermal and electrochemical resistance makes these candidate materials for operation in all solid-state batteries. New research on a garnet-related structure using $Li_7La_3Zr_2O_{12}$ (LLZO) shows that this material is able to maintain high ionic conductivities (>1 S/cm) in the presence of molten Li and at temperatures of up to 900 ºC.[11]

Because the ceramic conductors are not electrical conductors but ionic conductors, the proposed process will reduce the number of steps for the extraction of the hydrogen isotopes and minimize the use of expensive mechanical parts in contact with corrosive substances at elevated temperatures in a high radiation environment. This simplification may reduce the system downtime and cost associated with replacing the mechanical equipment that would be used in the previously explored options. This approach would also reduce the footprint of the extraction system and potentially allow it to be integrated directly into the blanket and limit the possibility of tritium release. A comparison of the molten salt extraction process with the proposed process is outlined in Figure 1.

Experimental

Chemicals. Lithium oxide (Li_2O_3), lanthanum oxide (La_2O_3), zirconium oxide (ZrO_2), lithium hydride (LiH), lithium deuteride (LiD), lithium sulfide (Li_2S), phosphorous pentasulfide (P_4S_{10}), tetrahydrofuran (THF), and lithium metal were all purchased from Sigma-Aldrich and used as received.

Instrumental Techniques. The synthesis of lithium phosphorous sulfide (Li_3PS_4) was performed inside of an inert atmosphere glovebox with O_2 and H_2O concentrations < 1 ppm. The LLZO was synthesized outside of the glovebox, but was stored inside of the glovebox afterwards to ensure that it did not absorb moisture that will react with lithium during the electrolysis experiments. The electrolysis experiments were performed inside of the glovebox in a specially designed stainless steel electrochemical cell that can handle temperatures up to 550°C. A two electrode setup was used to carry out the electrochemical experiments. A residual gas analyzer (RGA) (external to the glovebox) was also attached via a capillary line to the electrochemical cell during operation to monitor the formation of H_2, D_2, and HD during electrolysis. The electrolysis experiments were controlled and monitored by a Princeton Applied Research potentiostat. The lithium deuteride-acetylene black (LiD/AC) composite used for the electrolysis experiments performed with the β-Li_3PS_4 electrolyte was prepared by milling for 10 minutes in a SPEX 8000M high energy mill with stainless steel balls. Lithium metal infused with hydrogen for the LLZO electrolysis experiments was prepared by mixing LiH with a lithium foil and heating the sample to 600°C in a sealed vessel. The sample was held at 600°C for 1 hour before allowing the sample to cool to room temperature naturally.

A high temperature electrochemical cell was fabricated for evaluating the ability of the solid-state electrolytes to extract hydrogen isotopes from a lithium melt (Figure 2). This cell was designed to operate inside of a glovebox with an RGA interfaced with the cell via a capillary line. The RGA was located externally to the glovebox as shown in the supporting information.

Model Description. A model has been developed to allow prediction of the cell area to get an approximation for how much electrode area would be needed to carry out the reaction as improvements are made in the electrolyte to increase conductivity and reduce the electrolyte thickness. The model utilizes a mass and energy balance on the direct LiT electrolysis cell. The equation for the reaction rate of LiT from a mass balance on the electrolyzer,

$$r_{LiT} = \frac{W_{blanket}\left(c_{LiT,in} - c_{LiT,out}\right)}{1000 \cdot 3600 \cdot M_{LiT}} \tag{1}$$

where, r_{LiT} is the molar reaction rate of LiT in mol/s, $W_{blanket}$ is the mass flow rate of the lithium blanket in kg/hr, $c_{LiT,in}$ and $c_{LiT,out}$ are the inlet and outlet concentration of LiT in ppmw. As an approximation, it will be assumed that all of the LiT in the Li blanket is reacted and that $c_{LiT,out}$ is zero. This simplifies (1) to:

$$r_{LiT} = \frac{c_{LiT,in} W_{blanket}}{1000 \cdot 3600 \cdot M_{LiT}} \tag{2}$$

The rate of electrolysis for LiT can be expressed as the current going to the LiT reaction as,

$$r_{LiT} = \frac{I_{LiT}}{nF} \tag{3}$$

where, I_{LiT} is the current going to the LiT reaction, n is the number of electrons per mole of LiT reacted, and F is Faraday's constant.

The current going to the LiT reaction can also be expressed as a fraction of the overall cell current by

$$\varepsilon_{LiT} = \frac{I_{LiT}}{I_{cell}} \tag{4}$$

where ε_{LiT} is the current efficiency to the LiT electrolysis reaction and I_{cell} is the total cell current. Substituting (3) and (4) into (2) gives:

$$I_{cell} = \frac{nFW_{blanket} c_{LiT,in}}{1000 \cdot 3600 \cdot M_{LiT} \varepsilon_{LiT}} \tag{4}$$

The cell voltage, V_{cell}, equation for LiT electrolysis is a function of the equilibrium reduction potentials for the LiT oxidation and Li reduction, U_{LiT} and U_{Li}, at the temperature of interest along with the overpotentials for both reactions, η_{LiT} and η_{Li} and the ohmic losses in the electrolyte as expressed in equation 5.

$$V_{cell} = \left(U_{LiT,T} + \eta_{LiT}\right) - \left(U_{Li,T} + \eta_{Li}\right) + I_{cell} \frac{l}{\kappa A_{electrolyte}} \tag{5}$$

where l is the electrolyte thickness, κ is the electrolyte conductivity, and $A_{electrolyte}$ is the electrolyte area in contact with molten lithium. Substituting (4) into (5) and solving for the electrolyte area gives:

$$A_{electrolyte} = \frac{nFW_{blanket}c_{LiT,in}}{1000\cdot 3600\cdot M_{LiT}\varepsilon_{LiT}} \frac{l}{\left(V_{cell}-\left(U_{LiT,T}+\eta_{LiT}\right)+\left(U_{Li,T}+\eta_{Li}\right)\right)\kappa} \tag{6}$$

The power required for electrolysis can be calculated by the formula:

$$P = V_{cell} \cdot I_{cell} \tag{7}$$

Results and Discussion

For this work two potential solid state electrolytes (β-Li_3PS_4 and c-$Li_7La_3Zr_2O_{12}$)[11, 13]are synthesized and used for the extraction of hydrogen and deuterium from lithium in the solid and molten states. These two materials were selected for this application because of their negligible electrical conductivity and high lithium ionic conductivity at high temperatures. Additionally, β-Li_3PS_4 and LLZO have demonstrated good compatibility with lithium metal in solid and molten states, respectively. The β-Li_3PS_4 was prepared via solvent assisted mixing in THF followed by annealing to crystallize the desired phase as previously described.[13] LLZO was synthesized via a mechanical homogenization of the starting materials (Li_2CO_3 in 10 wt% excess, La_2O_3, and ZrO_2) followed by annealing in air. LLZO can exist in both tetragonal and cubic phase; however the latter is desired due to its higher ionic conductivity (~10^{-3} s/cm).[14] The LLZO phase formation is typically determined by the annealing temperature and time. Therefore, in order to determine the optimal temperature and time needed to prepare the desired c-LLZO phase, a series of in-situ XRD experiments were performed to monitor the transformation of the starting materials into the c-LLZO. The sample (~200 mg) was heated at 2°C/min and soaked at the respective temperatures for 10 minutes before measuring the diffraction pattern. Figure 3 shows that the desired cubic phase begins to appear at 850°C and becomes exclusively present in the sample at 950°C. When the sample is soaked for more than 60 minutes at 950°C, the LLZO begins to decompose and the diffraction peaks corresponding to La_2O_3 begin to reappear. This is caused by the loss of lithium in the sample due to its high vapor pressure at the annealing temperatures. To counteract the lithium loss during sintering and promote the formation of a lithium rich phase, an excess (~ 10 wt. %) of Li_2CO_3 is utilized for the LLZO preparation.

To determine if the electrochemical LiH breakdown followed by the release of hydrogen is feasible, the experiment was first tested using the β-Li_3PS_4 solid electrolyte to extract H and D from solid lithium was evaluated. To test this, a pellet (1 cm diameter and 1 mm thick) was pressed in a die set inside of a glovebox. Then a lithium foil was attached to one side of the pellet to serve as the counter electrode. To prepare the H and D infused lithium, ~25 wt. % LiH and LiD was added to lithium metal (~200 mg total) and sealed in a stainless steel Swagelok reactor using VCR gaskets. The composites were then heated up to 690°C and allowed to soak for 1 hour before allowing it to cool naturally back to room temperature. The H and D infused lithium was then collected and placed in a sample basin in the reactor (Figure 4). Once the electrochemical reactor was assembled, a heating jacket was attached to the cell with a thermocouple to monitor the cell temperature. Refer to the supporting information for more details on the cell design and configuration.

The cell was then heated to 150°C and allowed to soak for 1 hour before the electrochemical experiment and RGA of the released H_2, D_2, and HD was performed. Once the cell was at thermal equilibrium and the RGA signal was stable, a desired voltage (vs. Li/Li^+) was applied to the cell and the release of hydrogen isotopes was observed. Figure 5 shows the experiment results for the electrolysis as well as the RGA analysis for the release of H_2, D_2, and HD from a D infused lithium composite. When a voltage (2.0 V vs. Li/Li^+) is applied to the cell, the release of D_2 and HD is observed in the RGA. When the voltage to the cell is turned off, the release of D_2 and HD from the D infused lithium stops and the corresponding ion current in the RGA response is zero. To ascertain the electrolysis process, the voltage is once again applied and an increase in ion current due to the release of D_2 and HD is observed. This experiment demonstrates the feasibility of using this type of electrochemical process for the extraction of hydrogen isotopes from lithium. No attempt was made to operate the cell at temperatures beyond 150°C due to the decomposition of solid state electrolyte in the presence of molten lithium.

With the successful demonstration of β-Li_3PS_4 electrolyte to extract hydrogen isotopes from D infused lithium, a similar test was performed using the LLZO high temperature solid ionic conductor as it is capable of working in molten lithium at the operating temperature of the blanket in the fusion reactor (~500°C). This ionic conductor has previously been demonstrated to be stable at 800°C in molten lithium[11] and due to this reason it is considered as a good candidate for the current application.[11] In this experimental set-up, a LLZO pellet was pressed and then gold paint was applied to one side of the pellet to reduce the contact resistance with the counter electrode.[11] The pellet was then placed on top of the H infused lithium and the cell was assembled for the experiment and performed inside of the glovebox. The cell was then heated up to 550°C and allowed to thermally equilibrate. Next, an appropriate voltage was applied to the cell and the corresponding RGA signal clearly showed the release of hydrogen from lithium infused with hydrogen and behaved similarly to the low temperature experiment employing β-Li_3PS_4 as the Li ion conductor. During the experiment the cell voltage was turned off (labeled as OCV in Figure 6) to show that the release of hydrogen from the sample stopped during this portion of the experiment. These results indicate the proposed electrochemical method of utilizing a solid state ionic conductor at high temperatures can be used for the extraction of hydrogen isotopes from molten lithium blanket in a fusion reactor.

Additionally, process modeling was performed to understand the energy requirements and electrode areas needed for decomposing different concentrations of tritium (Table 2). Assumptions for the model are outlined in Table 1. This analysis looked at increasing LiT concentrations from 1 – 10,000 ppm of LiT and assumed incremental improvements in LiT cell properties that would be expected when moving to larger systems with steady state operation. The electrode area and power increased, but were not values that are unreasonably large for the amount of tritium being extracted. Electrochemical reduction potentials for the half-cell reactions at the reaction temperature are calculated using HSC Chemistry. The processing rate considered for the model is 1000 kg of blanket material processed per hour. It is assumed that the current efficiency is low at 25% to account for lithium oxidation as an alternate anode reaction. The overpotentials are assumed to be 200 mV and -200 mV on the anode and cathode, respectively. These overpotentials are used as typical for moderately complex electrochemical reactions. Assumptions have been made about the progression on improving electrolyte materials. It is assumed that the process would likely be adopted using low concentrations of LiT and then scaled up to larger concentrations later. It is assumed during this progression in LiT concentration that the ionic conductivity of the electrolyte will increase and the thickness of the electrolyte required will decrease. Cell power along with the lithium transport rate and the tritium production rate were also calculated and these values were found to scale linearly with the LiT concentration. See supporting information, Table S1, and Table S2 for model description, assumptions, parameters, and additional calculations

Figure 7 is based on the cell areas and concentrations in Table 2 and shows how the model predicts that electrolyte area will scale with increase in LiT concentration. The model results illustrate that improvement in the electrolyte properties would allow a 5 order of magnitude scaling in the lithium concentration that can be handled while the electrolyte area would only need to increase by 2 orders of magnitude. The cell areas projected are not unreasonable for electrochemical cells and indicate that the direct LiT process is feasible if improved electrolyte materials can be developed.

Conclusion

In this work, we successfully demonstrated a proof of concept for the extraction of H and D from molten lithium via a new electrochemical process utilizing solid ionic conductors in direct contact with the metal. This electrochemical process could potentially simplify the hydrogen isotope extraction from the breeder blanket through the elimination of mechanical parts and machinery (i.e. centrifugal contactors) in contact with corrosive materials (i.e. molten halide salts) at temperatures in excess of 500°C in a high radiation environment. Current work is focused on scaling up the synthesis of the LLZO electrolyte to make larger surface areas and still maintain the electrolysis reaction. Additional work will focus on the extraction of hydrogen isotopes using this methodology from a flowing lithium blanket.

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Figure 1. Comparison of the molten salt extraction process[12] (left) with the proposed process (right) utilizing solid ionic conductors for the separation of hydrogen isotopes from molten lithium.

Figure 2. (left) Design of the electrochemical cell utilized for the electrolysis reactions. (right) cell design shown with the capilary line that was attached to an RGA.

Figure 3. In-situ XRD monitoring the formation of the c-LLZO phase from the Li_2CO_3, La_2O_3, and ZrO starting materials. The c-LLZO and La_2O_3 diffraction peaks are denoted by the (*) and (x) symbols respectively. The additional peaks are due to the presence of ZrO_2.

Figure 4. (left) H infused lithium composite. (middle) Experimental set-up for the electrochemical experiments. (right) photo of the sealed electrochemical cell.

Figure 5. (top) Current profile for the electrolysis and the cell configuration. (bottom) Voltage profile and RGA analysis for the electrolysis of a D infused lithium composite with a β-Li_3PS_4 solid ionic conductor.

Figure 6. (top left) photo of the H infused lithium composite before the electrolysis (top right) LLZO pellet with the gold electrode on top of a H infused lithium composite, and (bottom) Combined electrochemical and RGA analysis of the electrolysis of H infused lithium demonstrating the release of H_2 at 500°C using the LLZO solid electrolyte. OCV – open circuit voltage.

Figure 7. Electrode surface area needed as a function of LiT concentration for processing 1 tonne per hour of Li blanket material

Table 1. Model Parameters for the LiT Electrolysis Process Calculations

Model Parameters			
Symbol	Value	Units	Origin
$U_{LiT,400°C}$	-2.82	V vs. H2 Electrode	HSC Chemistry
$U_{Li,400°C}$	-3.22	V vs. H2 Electrode	HSC Chemistry
F	96485	C/gmol	Physical Constant
n	1	moles e-/mole LiT	Stoichiometry
M_{LiT}	9.956	g/gmol	Physical Constant
$W_{blanket}$	1000	kg/hr	Assumption
ε_{LiT}	0.25		Assumption
η_{LiT}	0.2	V	Assumption
η_{Li}	-0.2	V	Assumption

Table 2. LiT model results for a progression in electrolyte conductivity and thickness

LiT Conc (ppm)	Cell Voltage (V)	Electrolyte Conductivity (S/cm)	Electrolyte Thickness (cm)	Cell Area (cm^2)	Cell Power (W)	Li Transported (g/hr)	T_2 Produced (g/hr)
1	3.0	0.0001	0.01	489	32	2.78	0.303
10	3.0	0.0002	0.01	2447	323	27.8	3.03
100	3.0	0.0005	0.01	9789	3230	278	30.3
1000	3.0	0.001	0.005	24471	32305	2780	303
10000	3.0	0.005	0.005	48945	323040	27802	3029