Experimental setup for analysis of sorption and desorption of tritium in liquid lithium under different external conditions

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Abstract. An original complex device has been designed and created specially for this research. Some of tritium sorption and thermal desorption in liquid lithium experiments has been successfully done already. First experiment series were carried out under reduced pressure (vacuum) to rule out other operating gases (like Ar) that may squeeze out tritium from lithium. Experiments showed good tritium desorption from lithium which highly depends on the temperature. A proportional gas counter tritium monitor was used to collect data from tritium thermal desorption experiments.

1. Introduction
The concept of using flowing liquid metals for all plasma facing components has been proposed as a potential and final solution to the critical plasma – material interaction issues [1]. Liquid lithium is a perspective material and it’s intended to be used in the DT fusion devices (Jet, Tokamak) as a cooling agent, which protects plasma facing components from complete thermal destruction. For example, temperature at the surface of divertor (inside the lower part of the plasma chamber) during fusion reaction can reach 800 °C [2, 3] and has to be constantly cooled down. Besides the divertor, plasma facing limiters are important as well. The most popular material in use for today’s Tokamak first wall components is graphite [4]. In liquid limiter case, limiter represents a non stop flowing liquid metal wall between fusion plasma and first solid wall of the plasma chamber. And one of the main candidates for this solution is lithium [4-6]. Lithium also is one of the most advantageous blanket materials not only because of its high heat transfer property to compose a self – cooled fusion reactor system but it also has a high tritium breeding ratio [7]. Around 7 grams of tritium per year as a by-product is generated in the Li loop [8]. Therefore, the concept of liquid lithium is one of the most suitable and perspective materials as than others. The main idea of the liquid metal cooling concept is that liquid lithium is flowing over the plasma facing components and cools it down. During this process lithium gets polluted (adsorbing) with plasma chamber gases (including fuel) and with chemically eroded particles of divertor surface as well. In order to not disrupt complex process of nuclear plasma, concentration of tritium in lithium shall not exceed certain acceptable standards (less than 1 appm) [9]. If too much fuel is absorbed, replenishment the plasma at a fast enough rate may be

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impossible [10]. Lithium keeps a very strong ionic bonding with hydrogen isotopes [7]. That fact makes it difficult to recover or extract tritium back from lithium waste. Development of tritium recovery method is one of the most important issues for the design of self-cooled Li blanket loop [7].

There are several published methods for the tritium separation from liquid lithium, like permeation window method [7-9, 11, 12], liquid eutectic method [11], molten salt recovery method [9, 11], cold trapping method [9, 11] and various distillation methods [9]. Some authors also list another one method as an individual method called gettering method [9, 13]. But technically, permeation window method in itself includes not only tritium separation but also collection which usually is done by the adsorption. This means, that permeable window in itself is or it has integrated tritium adsorbers, commonly referred as getters. That’s why listing a gettering method as a separate method is not quite necessary, basically it’s a part of the same permeation window method. Each of these tritium separation methods has its own drawbacks which mostly associated with insufficiently low concentration of tritium recovery. For example, the cold trap process can only reduce tritium concentration to about 400 appm, which is far above the ITER designed goal of reducing tritium concentration in lithium to ~1 appm [9]. Nevertheless, experimentally proved that using yttrium permeation window method it’s possible to reduce tritium concentration even from 0.0007 to 0.04 ppm [7], which is more than enough for ITER designed goal. But this method is quite slow even with few grams of lithium and must be done in laboratory conditions with great care. This means it would not be so easy to deal with hundreds of tons of liquid lithium which need to be separated from tritium as soon as possible. That’s why these all listed methods are promising methods for tritium separation and none of these individual methods hasn’t been denied or accepted as the best yet.

The particular research aims is to study tritium sorption and thermal desorption processes in molten lithium under different external conditions (temperature ramp, magnetic field, atmosphere and others). Investigate lithium - tritium chemically bonded forms which also includes a number of polymorphic forms (LiₙT) [14]. Develop and optimize conditions for effective separation of tritium from lithium.

2. Experimental

0.20 g of solid pure lithium of quoted purity 99.9 % was placed in a 15.7 cm³ stainless steel reactor tube and hermetically closed. All procedures were done in a hermetic glove box with an argon atmosphere. A vacuum of 0.01 mbar was chosen as the working atmosphere for the reactor tube. Reactor tube with lithium was placed in the programmable electric furnace and started a ramp heating to 220 °C at 10 °C / min. After reaching temperature of 220 °C, a 10 MBq of tritium was injected into reactor. After 2 hour of constant heating at 220 °C reactor was slowly cooled down to the initial room’s (20 °C) temperature at the 13 °C / h. At this point thermal desorption process can be started. Lithium reactor was slowly re-heated to 750 °C at 240 °C / h and this temperature was held constant for 2 hours. During this process liquid lithium reactor was continuously purged thru by the purge gas (He + 0.1% H₂). After 2 hour of constant heating and purging at 750 °C, reactor was slowly cooled down (40 °C / h) to initial room’s temperature without interrupting the purging. Thermally desorbed tritium was detected by the tritium monitor (Mab Solutions TEM-2100A) and all temperature data was collected by the data logger (Agilent 34970A). P10 (Ar + 10 % CH₄) was used as a counting gas for tritium monitor. A liquid nitrogen cold trap was used to prevent possible entering of lithium vapour in detector during purging process. To avoid possible moisture of the purge gas, a hot zinc granule furnace (400 °C) was placed right after the cold trap. An empty Drechsel bottle (2.3 L) was used to dilute the initial high concentrations of tritium. Figure 1 shows schematic diagram of the experimental apparatus and setup. Figure 2 shows the temperature program for sorption of tritium in liquid lithium. Whole sorption experimental process including ramp heating and cooling to 20 °C ran for 18 hours.
3. Results and discussion

Purging tritium from the cold reactor (Figure 3) shows that large amount of tritium (3.52 MBq) is not adsorbed or chemically bonded with lithium even after two hour of heating at 220 °C in which lithium is in liquid state.

Purging tritium during thermal desorption (Figure 4) shows that tritium release rate rapidly increases by increasing the temperature of the reactor tube. That fact demonstrates good tritium release tendency from liquid lithium in effect of temperature. Maximum tritium release rate reaches 2.08 kBq / s and it’s approximately the same (2.40 kBq / s) as purging the cold reactor from none adsorbed tritium (Figure 3). Total amount of released tritium during thermal desorption is 4.20 MBq (Figure 4).

Total amount of purged tritium including non adsorbed and thermally desorbed tritium is 7.72 MBq. Initial amount of injected tritium was 10 MBq, which means, that quite large amount of tritium (2.28 MBq) still stays in lithium. The fact shows that single thermal desorption process does not provide so simple and straightforward separation of tritium. And this type of thermal distillation has to be used combined with other methods or other external conditions, like magnetic field and various atmosphere gases in the reactor that may help to squeeze out tritium from lithium. A minimal amount of lithium was found in the cold trap, which means that lithium vapours may be transferred thru the system during thermal purging despite the fact that lithium has quite low vapour pressure. No lithium vapour was observed in the system tubes located after the cold trap, indicating that lithium vapour easily condenses on the cooler surfaces. Further investigations under different external conditions are in progress.
4. Conclusions
Experiments showed that tritium release from liquid lithium during thermal desorption is highly dependent on the temperature. Single thermal desorption process does not provide so simple and straightforward separation of tritium.

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