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# Hydrogen retention in lithium on metallic walls from "in vacuo" analysis in LTX and implications for high-Z plasma-facing components in NSTX-U

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#### ABSTRACT

The application of lithium to plasma-facing components (PFCs) has long been used as a technique for wall conditioning in magnetic confinement devices to improve plasma performance. Determining the characteristics of PFCs at the time of exposure to the plasma, however, is difficult because they can only be analyzed after venting the vacuum vessel and removing them at the end of an operational period. The Materials Analysis and Particle Probe (MAPP) addresses this problem by enabling PFC samples to be exposed to plasmas, and then withdrawn into an analysis chamber without breaking vacuum. The MAPP system was used to introduce samples that matched the metallic PFCs of the Lithium Tokamak Experiment (LTX). Lithium that was subsequently evaporated onto the walls also covered the MAPP samples, which were then subject to LTX discharges. In vacuo extraction and analysis of the samples indicated that lithium oxide formed on the PFCs, but improved plasma performance persisted in LTX. The reduced recycling this suggests is consistent with separate surface science experiments that demonstrated deuterium retention in the presence of lithium oxide films. Since oxygen decreases the thermal stability of the deuterium in the film, the release of deuterium was observed below the lithium deuteride dissociation temperature. This may explain what occurred when lithium was applied to the surface of the NSTX Liquid Lithium Divertor (LLD). The LLD had segments with individual heaters, and the deuterium-alpha emission was clearly lower in the cooler regions. The plan for NSTX-U is to replace the graphite tiles with high-Z PFCs, and apply lithium to their surfaces with lithium evaporation. Experiments with lithium coatings on such PFCs suggest that deuterium could still be retained if lithium compounds form, but limiting their surface temperatures may be necessary.

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#### 1. Introduction

The value of lithium plasma-facing components (PFC) to improve plasma performance has long been appreciated. For example, lithium wall conditioning produced the largest increase in the fusion "triple product," i.e., the product of density, confinement time, and temperature, in the Tokamak Fusion Test Reactor (TFTR) [1]. Approaches for introducing lithium into TFTR included lithium pellet injection and laser ablation of lithium in an in-vessel cru-

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cible. The locations that served as limiters for TFTR discharges depended on the plasma radius. When the radius was varied, different PFC surfaces could be coated with lithium from the plasma in a process called "painting." The PFCs in TFTR were made of graphite, including the surfaces that served as plasma limiters. Over a decade later, lithium was evaporated onto the graphite PFCs of the National Spherical Torus Experiment (NSTX). Unlike TFTR, NSTX operated with divertor plasmas, and improvements to discharge performance were observed as well [2].

Experiments with lithium walls have also been conducted in tokamaks with metal ("high-Z") walls. The PFCs in the Current Drive Experiment-Upgrade (CDX-U) were stainless steel, and a fully-toroidal "tray" at the bottom of the vacuum vessel was filled with

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liquid lithium to serve as a limiter. Lithium evaporated from the tray also coated up to 50% of the plasma-contacting area. When this occurred, the largest increase over predictions for energy confinement scaling in Ohmic tokamak plasmas was achieved [3]. The CDX-U device was modified to include a conducting shell that conforms to the plasma shape and renamed the Lithium Tokamak Experiment (LTX). The shell has a stainless steel liner that forms the LTX PFC. As in CDX-U, discharge performance was enhanced with the application of lithium wall coatings [4]. The relationship between plasma performance and recycling was inferred from variations in the hydrogen-alpha emission as PFC conditions changed.

Open questions remain, however, about the relationship between the detailed characteristics of the PFCs themselves and plasma performance. An example of why they are difficult to answer is presented in Section 2, where the complexities of the deuterium retention mechanism when lithium is applied to graphite PFCs are described. As a more expeditious means for determining PFC characteristics that better reflect conditions during plasma operations, a diagnostic called the Materials Analysis and Particle Probe (MAPP) was developed. A description of MAPP and its use in characterizing LTX PFCs with lithium coatings is provided in Section 3. The results of surface analysis of samples exposed to lithium evaporation in LTX are also included. Conclusions and implications for lithium coatings on high-Z PFCs are presented in Section 4.

#### Challenges of characterizing lithium applied to plasma facing components

Explanations for the improved confinement with the application of lithium to PFCs in magnetic confinement devices appeared to be straightforward. Lithium is a highly reactive metal that is expected to readily bind hydrogenic species. This leads to low-recycling walls, which in turn results in hotter edge plasmas and broader electron temperature profiles. Electron temperature gradients (ETGs) have long been associated with drift wave instabilities that could degrade energy confinement [5]. Reducing wall recycling would reduce ETGs, and thus improve plasma performance.

Low recycling walls were also needed for so-called "supershots" in TFTR, and lithium pellet injection was a technique for achieving them [6]. The favorable confinement scalings of the supershots have been explained through the reduction of turbulence through sheared flows. The reduced ion thermal diffusion from suppression of the toroidal ion temperature gradient (ITG) driven modes then led to the high ion temperatures obtained in supershots [7].

The choice of lithium as a PFC then seemed to be clear. The high chemical reactivity of lithium enabled retention of hydrogen from the plasma by the formation of lithium hydride. This simple picture should not hold, however, if the lithium were applied to graphite PFCs. Lithium intercalates into graphite, and does not have the same characteristics as lithium in its elemental form. As a result, lithium intercalated into the graphite should not necessarily volumetrically convert to lithium deuteride as observed when a macroscopically–thick (5 mm) liquid lithium sample was exposed to a high deuterium fluence [8].

The actual mechanism for the retention of deuterium in graphite PFCs was determined only after analysis of NSTX PFCs. X-ray photoelectron spectroscopy (XPS) measurements were performed after the removal of lithium compounds formed after exposure to air. They revealed chemical complexes involving lithium, oxygen, and carbon [9]. Quantum-classical molecular dynamics (QCMD) modeling was conducted for deuterium bombardment of surface matrices of mixed atoms. Simulations were performed for surfaces composed of lithium, oxygen, and carbon as determined from XPS data. Calculations of deuterium bombardment were also conducted for surfaces consisting only of lithium and carbon [10].

When lithium and carbon alone were included in the QCMD calculations, the percentage of bound deuterium that shared charges with lithium was even lower than the surface fraction of lithium relative to the carbon. This is consistent with what would be expected when lithium intercalates into graphite and binds with carbon. For a surface that included lithium, oxygen, and carbon, however, the percentage of deuterium that shared charges with lithium was again low. The fraction that shared charges with oxygen, however, was not only greater than the percentage of deuterium, but exceeded the fraction of oxygen in the original surface composition.

The QCMD results were thus able to account for the unexpected effectiveness of lithium coatings on graphite to retain deuterium. They demonstrated that binding deuterium in a system that includes lithium, carbon, and oxygen was much more effective for deuterium retention than lithium deuteride formation. The data that supported the modeling, however, were only obtained after the end of an entire NSTX operational period. A PFC tile had to be taken out of the vacuum vessel and treated to remove the lithium compounds that formed after prolonged exposure to air. It then had to be assumed that the exposed surface reflected PFC conditions during plasma operations. This demonstrates the value of analyzing PFC samples immediately after plasma exposure, to reduce the uncertainties from assumptions needed to interpret surfaces exposed to an entire run.

# 3. Hydrogen retention by lithium oxide from in vacuo sample exposure and analysis with materials analysis and particle probe

To address the need for in vacuo PFC analysis, i.e., without venting the vacuum vessel and removing PFCs, the MAPP was developed [11]. This system was designed to permit up to four samples to be exposed to plasmas. The samples can then be withdrawn, without breaking vacuum, into an analysis chamber equipped with several surface analysis capabilities. They include x-ray photoelectron spectroscopy (XPS), low-energy ion scattering spectroscopy (LEISS), and direct recoil spectroscopy (DRS). Because the samples are attached to heaters that can be controlled separately, they can be analyzed individually with thermal desorption spectroscopy (TDS) or temperature programmed desorption (TPD).

The MAPP diagnostic was originally constructed for use on NSTX. During the outage for the construction of the NSTX-Upgrade (NSTX-U), the MAPP was installed on LTX to study samples introduced into the plasma chamber [12]. The samples were made of materials that included stainless steel to match the conducting shell surface that constitute the LTX PFCs. Lithium evaporated onto LTX walls also covered the samples. These samples were subsequently exposed to LTX discharges, and withdrawn into analysis chamber under vacuum and analyzed using XPS.

Fig. 1a shows the atomic ratios of lithium to oxygen ( $R_{\rm Li,O}$ ), as determined from XPS measurements of the Li(1s) and O(1s) photoelectron peaks, as a function of exposure time to the residual gases within LTX after lithium deposition on the shell surfaces. The larger uncertainties at shorter exposure times were the result of lower statistics for the XPS data, due to the limited time over which the spectra could be obtained. Within several hours after lithium evaporation,  $R_{\rm Li,O}$  reaches a level indicative of a surface that is primarily lithium oxide (Li<sub>2</sub>O). This is consistent with earlier measurements of the oxidation of lithium when exposed to water vapor [13].

The  $R_{\rm Li,O}$  remains essentially unchanged for nearly 100 h under a partial pressure of water of about  $2\times 10^{-9}$  torr, and then only slowly begins to decrease. A salient observation is that in terms of LTX plasma performance, a lithium oxide PFC surface does not appear be substantially different from more lithium-dominated surfaces closer to the time lithium was applied. Fig. 1b shows the

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