



Abstract

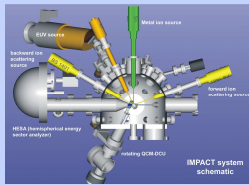
Lithium has been considered a potentially viable plasma-facing surface enhancing the operational performance (low-recycling regimes) of fusion devices such as TFTR and NSTX^{1,2}. Although extensive work has been done with lithium in the solid and liquid states³, only limited effort has been done in understanding how lithium surfaces behave on various metallic and graphitic substrates.

This work presents initial results on the chemical and physical properties of lithiated graphitic surfaces in support of NSTX Li studies. In particular, lithium coatings are used in NSTX runs to enhance energy confinement. Questions remain on the role of lithiated surfaces and multi-material interactions at the plasma edge. Processes of interest are the erosion of lithiated graphite surfaces, diffusion of Li into graphite and D-retention of lithium-covered surfaces. These processes consist of spatial scales from a few monolayers at the vacuum/film interface to 100's nm deep. Studies are conducted in the IMPACT experimental facility. IMPACT is designed to study in-situ how multi-component surfaces evolve under particle irradiation. Techniques include: low-energy ion scattering spectroscopy (LEISS), direct recoil spectroscopy, X-ray photoelectron spectroscopy and in-situ erosion diagnosis. In this paper detailed LEISS and XPS studies of lithiated graphitic surfaces simulating conditions in NSTX are presented.

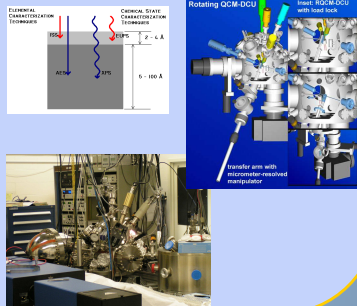
1. D. Mansfield, Nucl. Fusion 41, 1823 (2001).
2. R. Majeski, R. Doerner, T. Gray, R. Kaita, R. Maing, et al. Phys Rev. Lett. 97, 075002 (2006).
3. J.P. Allain, M. Nieto, M.D. Coventry, R. Stubbers, D.N. Ruzic, Fusion Engineering and Design 72, 1-3, (2004) 93-110.

IMPACT Experimental Setup

The IMPACT (Interaction of Materials with energetic Particles and Components Testing) is designed to study heterogeneous surfaces at the micro- and nano-scale with in-situ metrology during surface modification by energetic charged and neutral particles at low energies (5-1000 eV) and thermal metal atoms (e.g., Sn, Li).



- IMPACT is equipped with various excitation sources including: EGN4 e-beam evaporation source (thermal Li) with an in-line flux meter. The Li vapor source is then cross-calibrated *in-situ* with a microbalance that sits on an axis perpendicular to the evaporator line of sight.
- Several ion sources including an NTI 1403 alkali-metal (Li) energetic source, electron source and several X-ray sources are used as either "probes" or "modifiers".
- Surface concentration is probed by low-energy ion scattering spectroscopy and XPS (X-ray photoelectron spectroscopy). XPS also provides the chemical state of surface species with depth resolution of about 1-2 nm.
- Sputtering induced by energetic ions is measured in-situ with a rotating quartz crystal microbalance dual-control unit (QCM-DCU).
- Rotation allows for a direct *in-situ* measurement of the multi-species sputtered flux collected on the oscillator.



¹ J.P. Allain, et al. Rev. Sci. Instrum. 78, 113105 (2007)

Results

Erosion from lithiated surfaces have been studied as shown in the lower left figure. Lithium yields from both solid and liquid state of lithium have been measured. In addition, some measurements of erosion from lithiated surfaces on graphite have also been completed and agree with known physical sputtering reduction data of lithiated graphite¹ (also see M. Racic et al. P3-48). Lithium is also found to intercalate as demonstrated by work of Sugai et al.² Results of this work also corroborate this using both low-energy ion scattering spectroscopy and X-ray photoelectron spectroscopy.

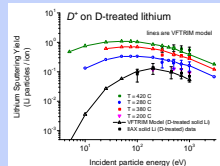


FIG. 1 Sputter yield of lithium from D bombardment

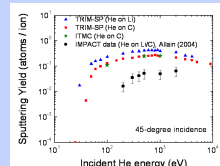


FIG. 2 Sputter yield of lithiated graphite

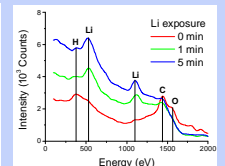
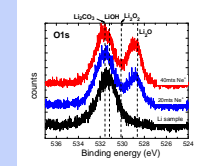
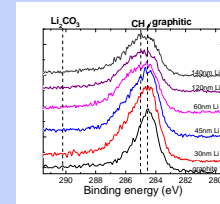


FIG. 3 LEISS spectra from Li-C

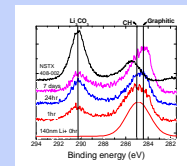
- XPS analysis on NSTX tiles showed presence of C, O, B and Li
- C 1s, O 1s and Li 1s lines showed presence of lithium carbonate on NSTX tiles
- Metal oxide formation (Li₂O) is not evident in Li1s and O1s XPS spectra
- LiOH may be also present on the top surface (oxygen from air)
- The strong presence of Li₂CO₃ and/or LiOH can be due to exposure of the tiles to air
 - Li easily oxidizes in the presence of air



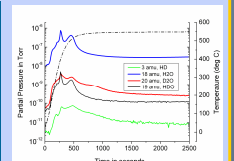
XPS analysis of Li metal showed LiOH on surface



- In-situ XPS analysis of Li doping on graphite showed absence of carbonate and presence of peroxide.
- In-situ analysis showed peroxide layer is formed immediately after the deposition even at UHV
- Lithium peroxide functionality is found to be absent in Li metal
- The formation of carbonate functionality can be related to the interaction of peroxide with carbon in the presence of oxygen and water in atmosphere.

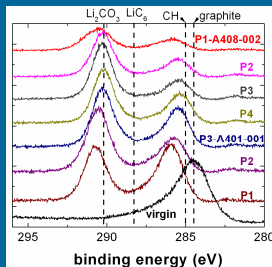
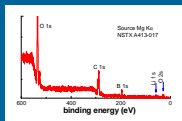
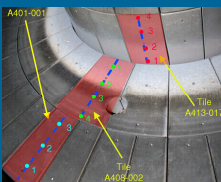


- ATJ graphite samples exposed to Li vapor deposition at about 10 nm/min
- Samples are then exposed to high-intensity D₂ irradiation at 500 eV/amu
- Samples characterized ex-situ (transferred under argon ambient) in the Omicron Cluster System at Purdue University's Birck Nanotechnology Center
- Peaks in D-containing species reflect decomposition of surface hydroxides
- Signs of oxygen reduction by presence of lithium is evident; further work must clarify role of D species



Surface Characterization and Analysis

- NSTX tiles selected from divertor floor exposed to lithium evaporation. Analysis completed ~ 3 months after plasma discharges.
- In addition to tile analysis, *in-situ* studies in IMPACT studied Li vapor interaction with ATJ graphite surfaces using similar Li deposition rates as those in NSTX Li shots



Photoelectron spectra of carbon from lithiated ATJ graphite NSTX tile sample. Analysis points corresponds to picture on top left. XPS analysis of NSTX tiles showed strong presence of Li₂CO₃

- XPS and ISS studies of in-situ Li doping in UHV is essential for getting further information about
 - How long the Li layers remain on the substrate surface?
 - Is there any change in surface chemical state over time?
 - When do the oxide layers form?
 - Is there any Li passivation effects over time even in UHV?
 - What happens to the Li if the tiles are exposed to air?
- In-situ studies have been carried using IMPACT facility at ANL
 - Li deposited on graphite and SS targets at 1mm/min
 - XPS analyses have been carried out at various levels of doping
 - XPS studies of air exposed samples
 - Also studied the oxidation effects of 'as received' Li metal

Summary and Future Work

- Lithiated surfaces have demonstrated unique properties for their use in limited and diverted fusion devices
- Liquid lithium work has been extensive, however its use in actual tokamak environments is limited
- The ability for lithiated surfaces to pump D and provide "low-recycling" regimes that dramatically improve performance in current fusion machines remains lithium's most attractive attribute
- Understanding how lithiated surfaces will pump D in a complex tokamak plasma remains our most challenging task in using Li as a plasma-facing surface
- Outstanding Issues and Future Work:
 - What is the role of peroxide (530 eV, Li₂O₂) in the chemistry of lithiated layers on graphite and their ability to pump D?
 - At what Li dose can one expect the dominance of the peroxide to deteriorate and thus this increase the probability for pumping D?
 - What is the role of He discharge cleaning and can it modify lithiated graphite to mitigate peroxide formation?
 - What is the role of lithiated graphite on other pure Li properties?
 - For example: Li secondary sputtered ion yield: Carbon tends to neutralize ion emissions thus decreasing Li ion yields
 - Further work will address these issues and also understand the synergistic role of energetic He and D at low threshold level energies (50-150 eV) on D pumping by lithiated surfaces (on top of: C, Mo or W)

Acknowledgements

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