Time dependent low-energy deuterium interactions with lithiated graphite plasma-facing surfaces

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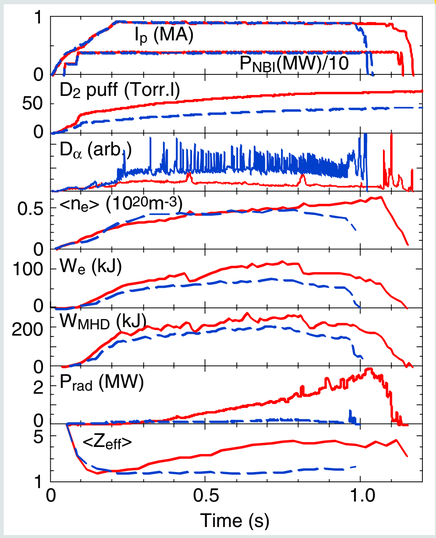
P09B

Abstract

Background

- Lithium dramatically improves plasma performance in fusion devices, in part, by reducing deuterium recycling.
- We investigate surface chemistry of Li-graphite to understand what mechanisms allows Li to enhance plasma performance.
- X-ray photoelectron spectroscopy (XPS) can be used to observe lithium/ deuterium interactions with oxygen and carbon on ATJ graphite.
  - Li-O-D interactions occur at 533.0 ± 0.6 eV.
  - Li-C-D interactions occur at 291.4 ± 0.6 eV .

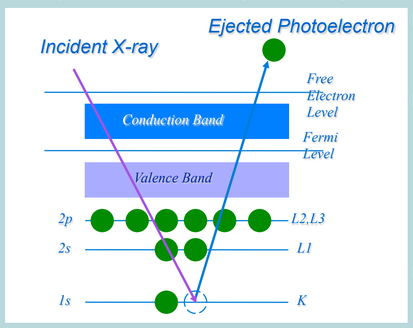


Objectives

- Instead of LiD bonding as seen in pure liquid Li, graphite introduces additional complexities.
- Show that Li-O-D and Li-C-D bonds become "saturated" with D at fluences between 3.8 and 5.2×10^{17} cm^{-2}.
- Demonstrate the use of advanced in-situ facilities to better measure saturation dynamically.

① Surface Characterization Methods

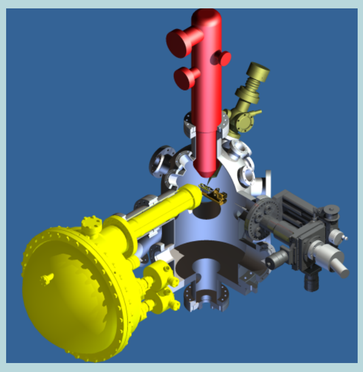
X-ray Photoelectron Spectroscopy: *The photoelectric process*



- *XPS spectral lines are identified by the shell from which the electron was ejected (1s, 2s, 2p, etc.).*
- *The ejected photoelectron has kinetic energy:*

  KE = hv – Binding E – Work Function

- *Energy of ejected photoelectron reveals characteristic information regarding the elemental composition of the substrate.*



PRIHSM schematic showing future capabilities

Experiment Methods

- XPS performed before and after each process (dwell=0.8ms, step=0.05eV).
- Processes include:
  - Li deposition via lithium evaporator.
  - D^+ bombardment (irradiation) (500 ev/amu).

PRIHSM Facility

Particle Radiation Interaction with Hard and Soft Matter

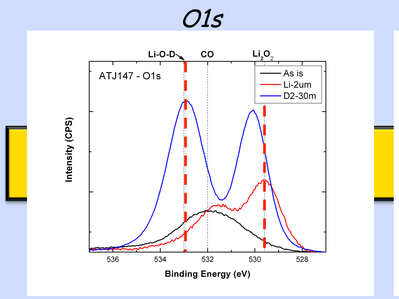
In-situ capabilities include:

- XPS
- UPS
- BS-ISS
- FS-ISS
- DRS
- Evaporation
- ARUPS
- ARPES

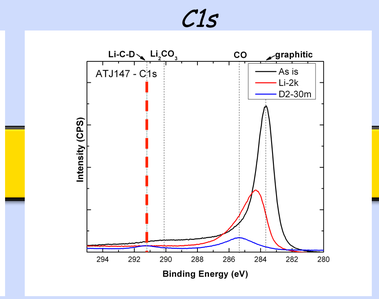
② Deuterium-Lithium Surface Chemistry

ATJ graphite surface chemistry with 2μm lithium deposition and D^+:

O1s



C1s



ATJ graphite sample substrate (black) with 2μm lithium conditioning (red), and 30 min deuterium bombardment (blue). Control studies isolate the effect of each process

What happens?

Oxygen:

Li and O interactions are manifest at 530.1 eV in the XPS spectrum.

Li, O, and D interactions, on a graphite substrate, are manifest at 533.0 eV.

Carbon:

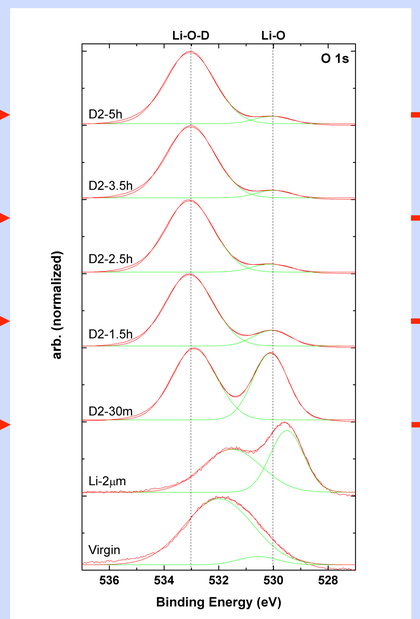
Li, D, and C interactions are manifest at 291.4 eV.

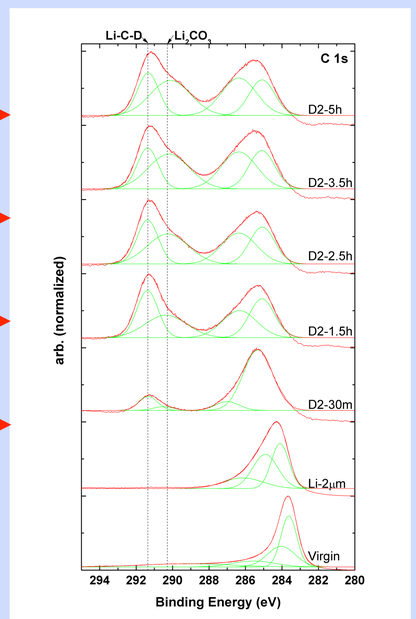
Time dependent deuterium fluence affects dominating chemical bonds:

As ***deuterium fluence is increased,*** relative peak heights change. Beyond 3.5 hr, changes become less apparent.

XPS Spectra: • Peak shift • New peak = Change in chemical bonds/interactions

- 5 hours (Φ = 5.2 E17 cm^{-2})
- 2.5 hours (Φ = 3.8 E17 cm^{-2})
- 1.5 hours (Φ = 2.5 E17 cm^{-2})
- 30 min (Φ = 9.0 E16 cm^{-2})





•Li-O-D peak (533.0 ± 0.6 eV) increases

•Li-O peak (530.1 ± 0.6 eV) decreases

•Li-C-D peak (291.4 ± 0.6 eV) increases

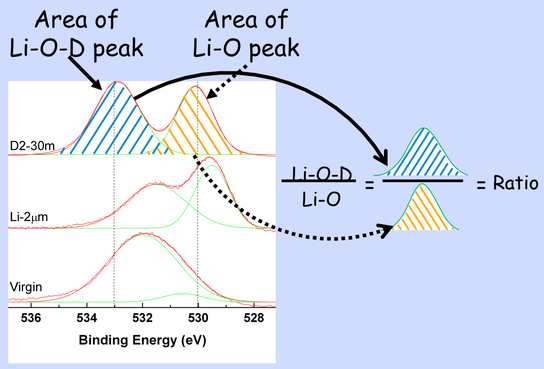
C.N. Taylor, B. Heim, J.P. Allain, Journal of Applied Physics 109, 053306 (2011)

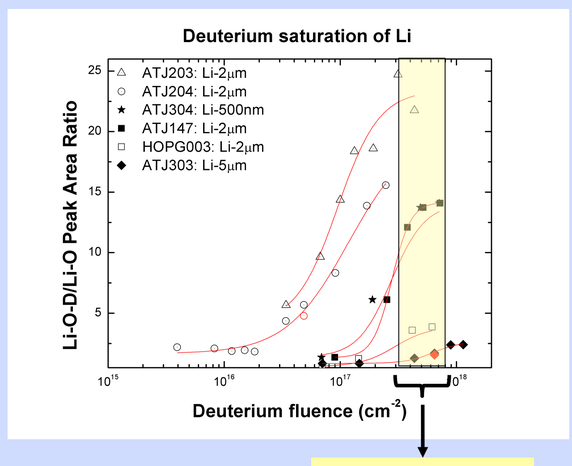
③ Deuterium Saturation

Deuterium saturation:

Solid lithium on graphite plasma-facing components have a finite deuterium storage capacity. Understanding and parameterizing deuterium saturation impacts the procedures for lithium wall conditioning, and assessing feasibility of advanced Li regimes.

How is saturation calculated?





Deuterium saturation:

The fluence at which the normalized derivative < 10% between consecutive irradiations .

What happens

- A given lithium layer is capable of "holding" a finite amount of deuterium.
- Any deuterium that is added to the system does not affect the binding chemistry (as observed through XPS).
- For various samples, the magnitude of the area ratios have high variability (unknown causes).
- However, saturation fluence for all samples remains within <1 decade region.
- D saturation occurs from 2 x 10^{17} and 8 x 10^{17} cm^{-2}.

Sample Parameters

Sample	Lithium *	D fluence	XPS scans
ATJ203	2 μm	4E17 cm^{-2}	7
ATJ204	2 μm	2E17 cm^{-2}	10
ATJ304	500 nm	7E17 cm^{-2}	4
ATJ147	2 μm	7E17 cm^{-2}	5
HOPG001	2 μm	3E17 cm^{-2}	7
ATJ303	5 μm	1E18 cm^{-2}	6

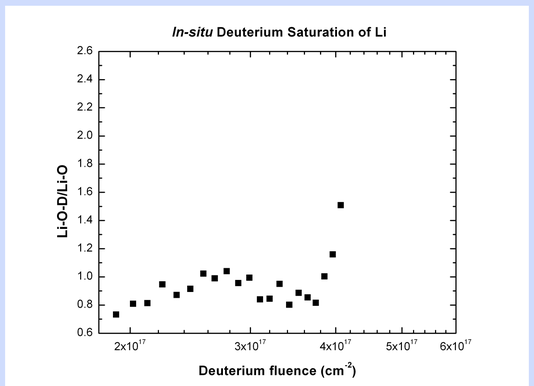
*Thicknesses such as 2 μm are nominal and neglect surface effects such as porosity, morphology, or intercalation.

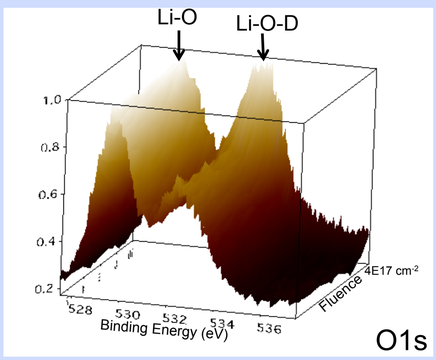
C.N. Taylor et al., J. Nucl. Mater. (2010), doi:10.1016/j.jnucmat.2010.09.049
J.P. Allain, D. Rokusek, et al., J. Nucl. Mater. 390-391 (2009) 942

④ *In-situ* Measurement of Saturation

Continuous Analysis

- After lithium was deposited on ATJ graphite, 48 consecutive XPS were performed during the 300 minute irradiation. Final deuterium fluence reached 4 x 10^{17} cm^{-2}.





- The Li-O-D peak (533 eV) begins to dramatically grow at a fluence of 3.8 x 10^{17} cm^{-2}.
- The O peak formerly located at 532 eV gradually shifts to 533 eV with increasing fluence.
- The initial ratio increase and decrease is partially a result of intercalation and peak transition from 532 to 533 eV.

⑤ Conclusions

- ❑ The point at which lithiated graphite saturates with deuterium can be observed in XPS spectra by comparing ratios of integrated peak areas.
- ❑ Continuous analysis in the PRIHSM facility provides a unique diagnostic to measure saturation. Saturation was found to occur
- ❑ A nominal lithium thickness of 2μm becomes saturated with D at ~10^{17}m^{-2}.
- ❑ Lithium thickness is a fundamental parameter in deuterium retention. *A **nominal minimum threshold*** thickness exists between 100 and 500 nm.
- ❑ Deuterium does not bind directly with lithium, but lithium always binds with ***oxygen and carbon***, when present.

Discussion

- ❑ NSTX lithium depositions (10s-100s nm per cycle) likely saturate after a single discharge (10^{17} cm^{-2}).
- ❑ On graphite, Li, O, and D interactions are manifest at 533.0 eV. Peak dominates with larger D fluence, indicating that a given lithium thickness has a finite deuterium capacity. Li, D, and C interactions are manifest at 291.4 eV. Relative peak energy increases with increased D fluence.
- ❑ Li and O interactions, on a graphite substrate, are manifest at 530.1 eV in the XPS spectrum. Peak diminishes with larger D fluence, indicating that D^+ binds with and consumes the Li-O bonds.
- ❑ Future work will: 1) utilize ultraviolet photoelectron spectroscopy (UPS) to probe outer valence electrons, 2) systematic study of deuterium retention for various substrates, and 3) examine the effects of surface morphology.

Acknowledgements

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