Review of APS-DPP 2010 results:

Ultraviolet photoelectron spectroscopy analysis of lithium and deuterium interactions with graphite

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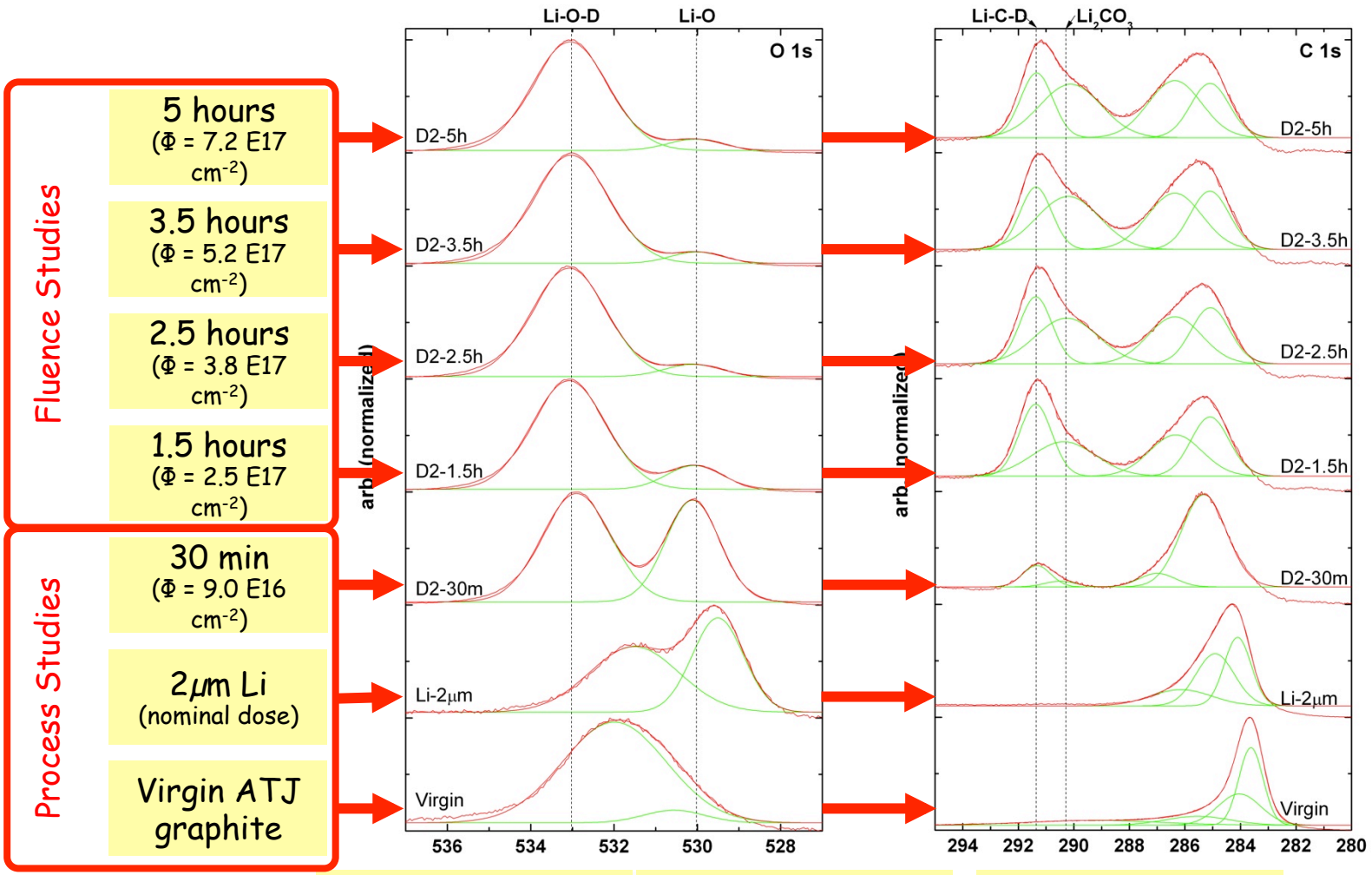
Outline

- Review of XPS results
- Why use UPS?
- Review of D. Ensling UPS studies
- Initial results
- Conclusions

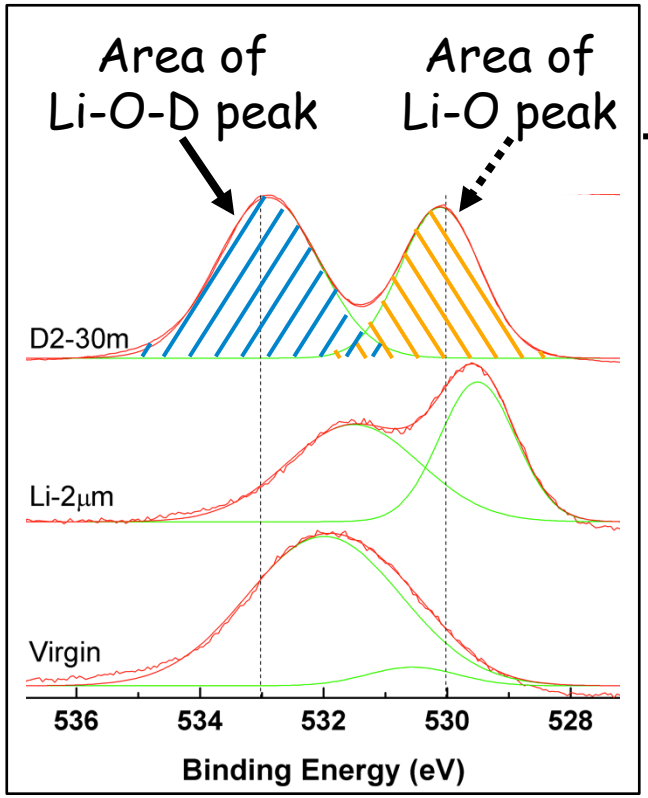
Review of XPS results – Chemistry



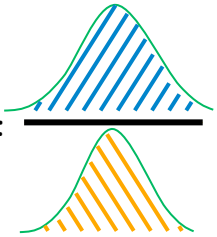
- Li-O-D peak (533.0 ± 0.6 eV) increases with D fluence
- Li-O peak (530.1 ± 0.6 eV) decreases with D fluence
- Li-C-D peak (291.4 ± 0.6 eV) increases

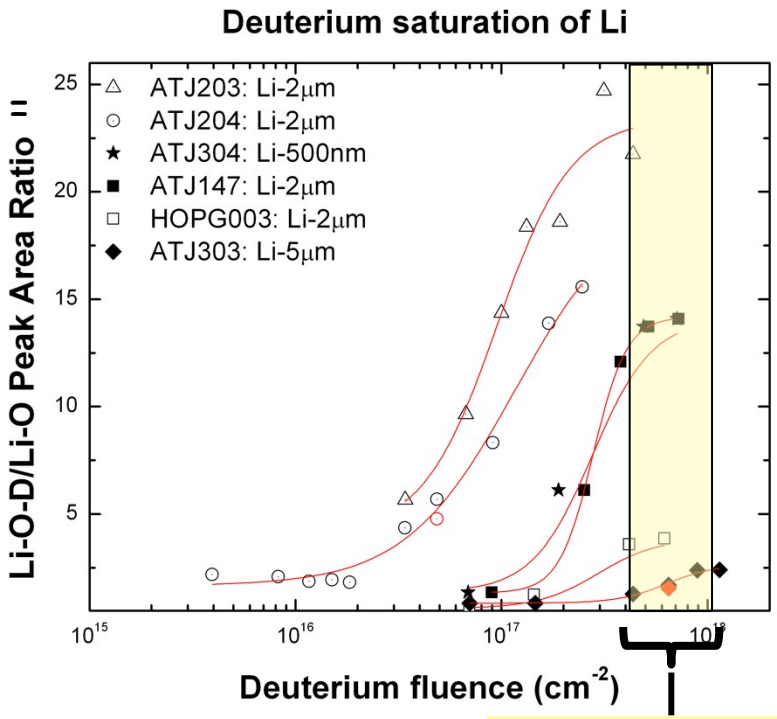
Review of XPS results – Saturation

- How do we calculate saturation?



$$\frac{\text{Li-O-D}}{\text{Li-O}} =$$



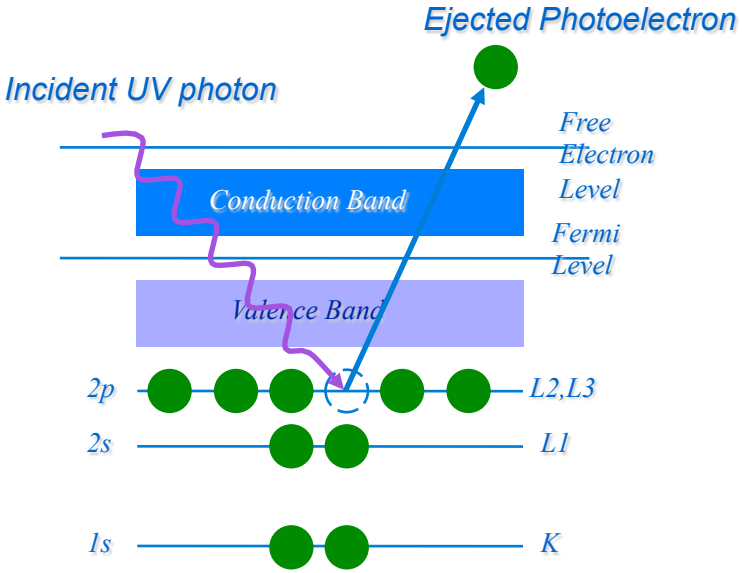


Deuterium saturation: The point at which deuterium no longer induces changes in surface chemistry. Defined as the fluence at which the normalized derivative < 10% between consecutive irradiations .

A nominal lithium thickness of 2μm becomes saturated with D at a fluence of ~10^{17}cm^2. NSTX lithium depositions (10s-100s nm per cycle) likely saturate after a single discharge (10^{17} cm^{-2}).

Why ultraviolet photoelectron spectroscopy?

- XPS ejects core level photoelectrons.
  - Results provide consistent elemental data with qualitative chemical interpretation.
  - Probing depth <10 nm.
- UPS ejects valence level photoelectrons.
  - Chemistry occurs at the valence level.
  - Probing depth <1 nm.



Why ultraviolet photoelectron spectroscopy?

- Objective:
  - Show that C does or does not influence Li-O-D interactions
  - Show that O does or does not influence Li-C-D interactions
- Plan:
  - Perform initial UPS experiments on post mortem samples.
  - Validated technique for our materials prior to purchasing and installing new equipment for full *in-situ* studies.

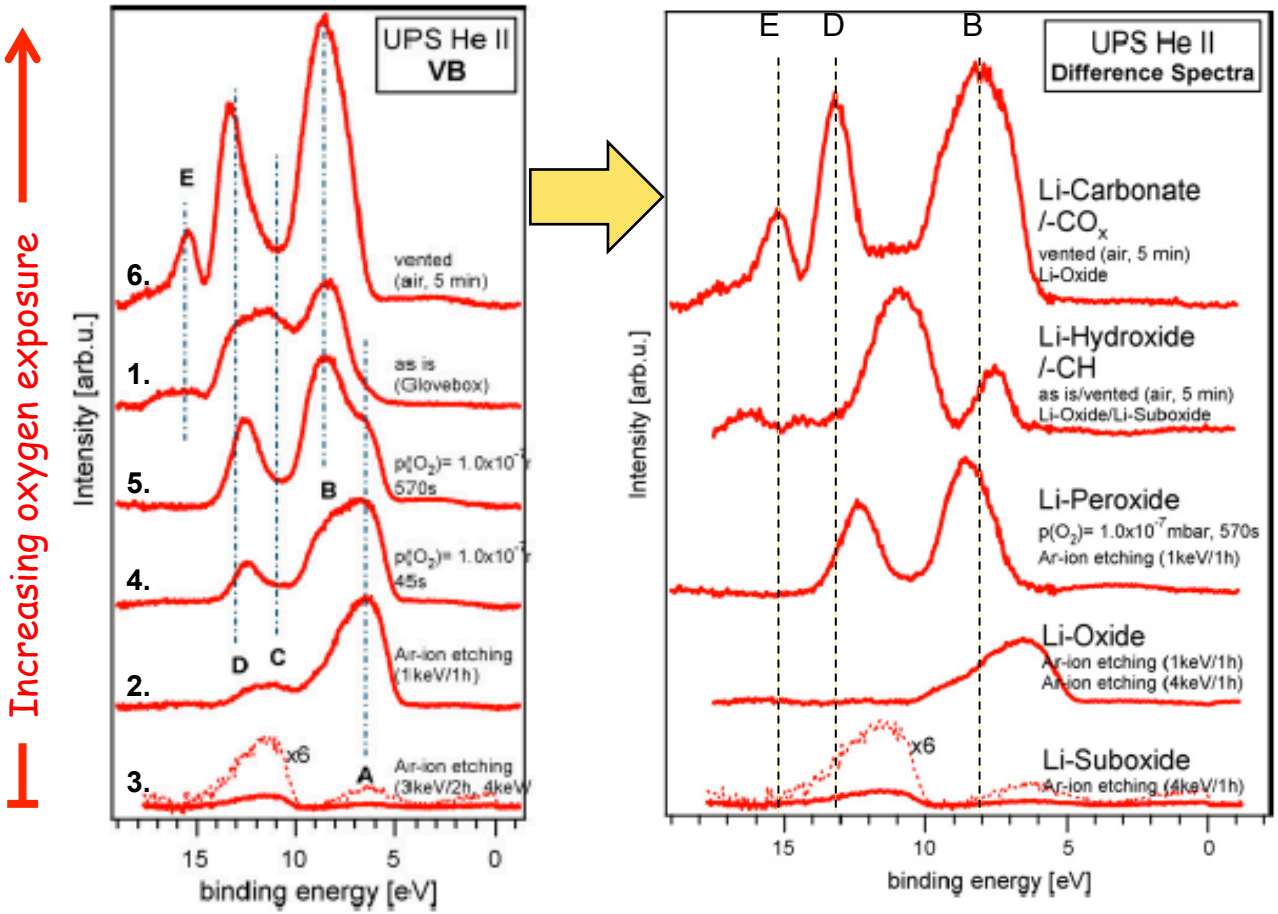
Review of D. Ensling UPS studies

Process:

1. UPS of “virgin” lithium
2. Ar cleaning (1 keV, 1hr)
3. Ar cleaning (3 keV, 2hr)
4. O exposure (45 sec)
5. O exposure (570 sec)
6. Air exposure (5 min)

Identification of peaks lines:

- **A** (6 eV) and **C** (10.5eV): O 2p
- **B** (8.5 eV): O 2p/C 2sp
- **D** (11.5 eV): C 2sp/O 2p
- **E** (15.2 eV): C 2s



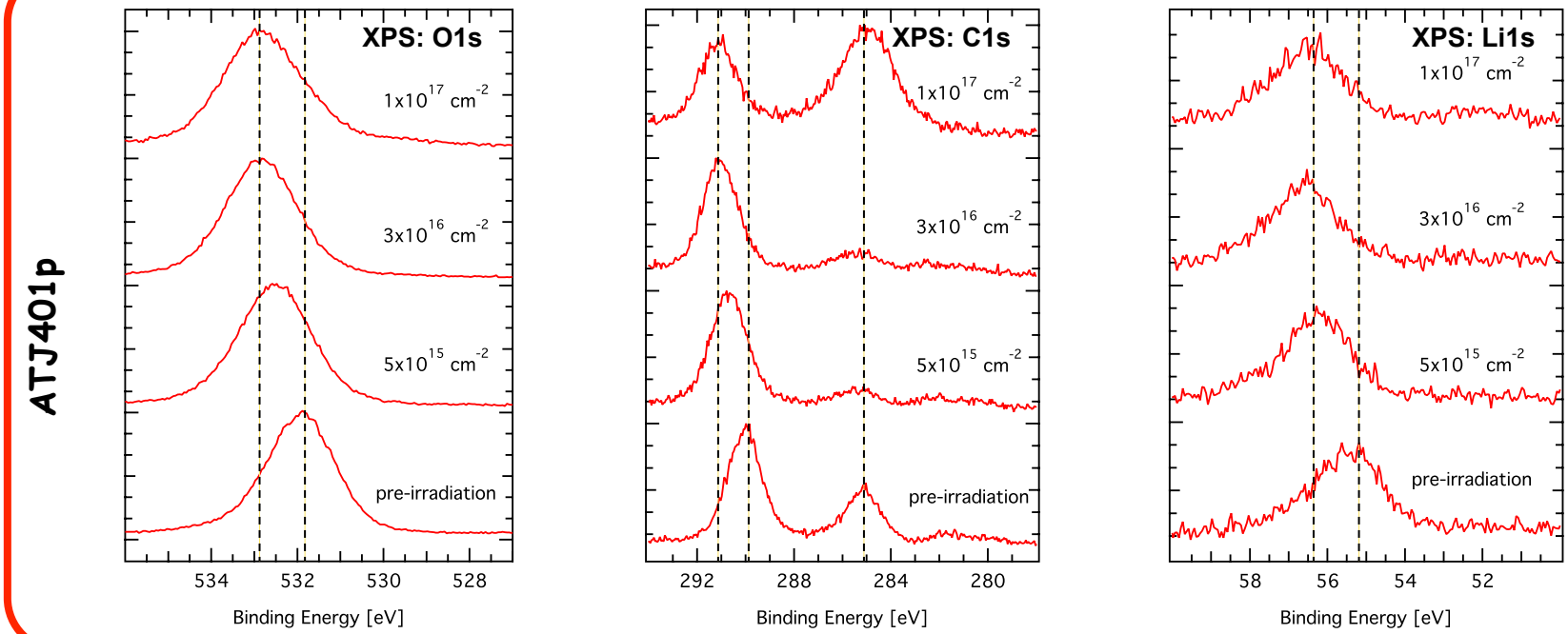
Initial results

Samples:

- Mo205p – Porous molybdenum exposed to ~1 week of NSTX plasmas via the sample analysis probe. Lithium conditioning preceded deuterium plasma discharge. Sample removed and analyzed at Purdue. Note that MAPP will allow for in-vacuo characterization at NSTX, thus simplifying this procedure.
- ATJ401p – ATJ graphite sample exposed to simultaneously with Mo205p to NSTX plasmas.
- ATJ147a – ATJ graphite with 2μm lithium deposition and D_2^+ irradiation at Purdue University. Post-mortem UPS analysis follows long term air exposure and includes Ar sputtering for surface cleaning.

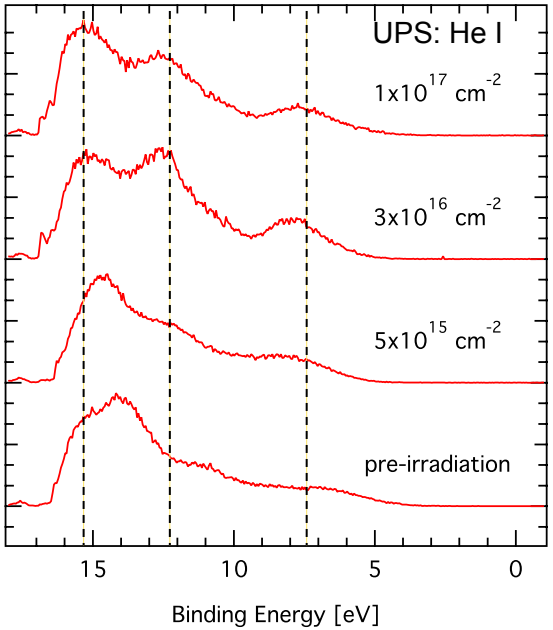
Initial results – XPS

- XPS survey to verify the presence of Li
  - Second peak (Li-O) in O1s buried in shoulder?

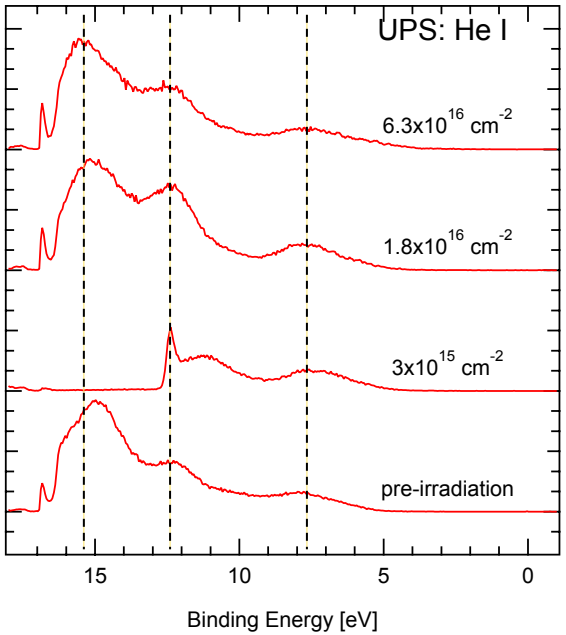


Initial results – UPS

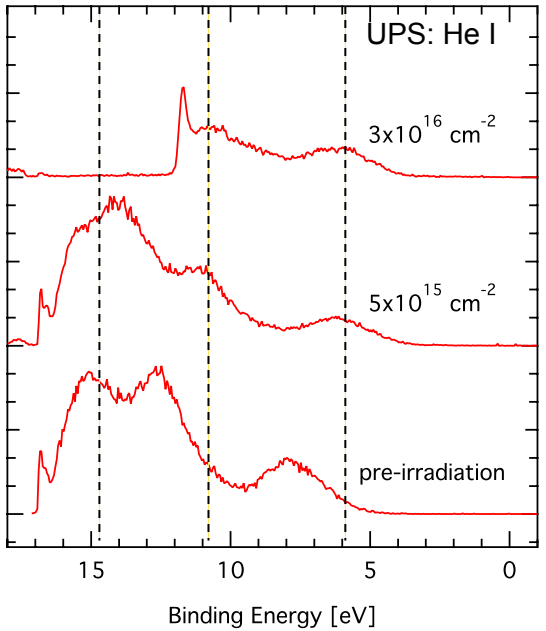
ATJ401p



ATJ147a



Mo205p



Identification of peaks lines:

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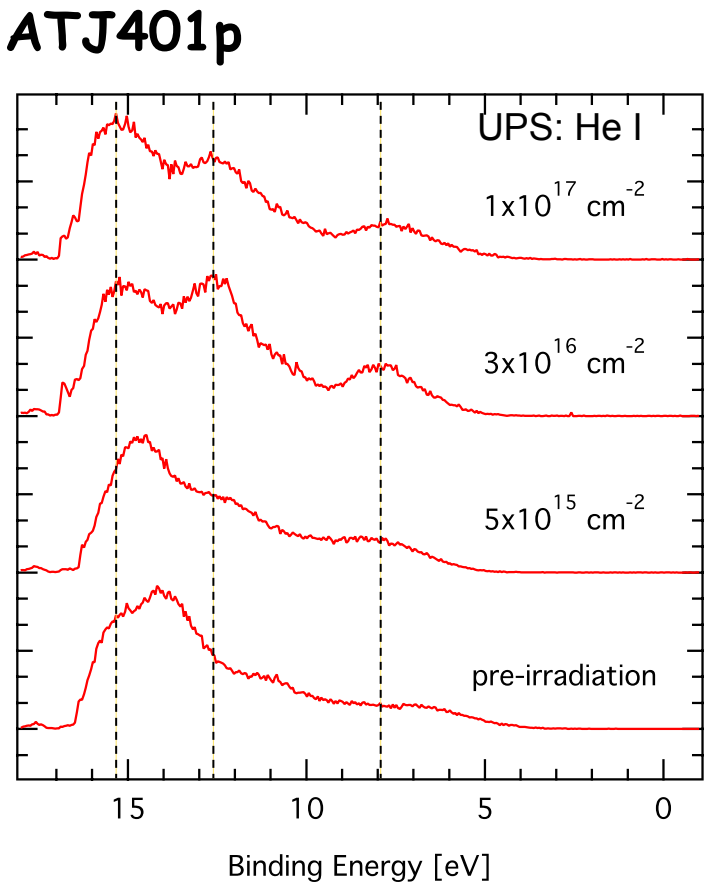
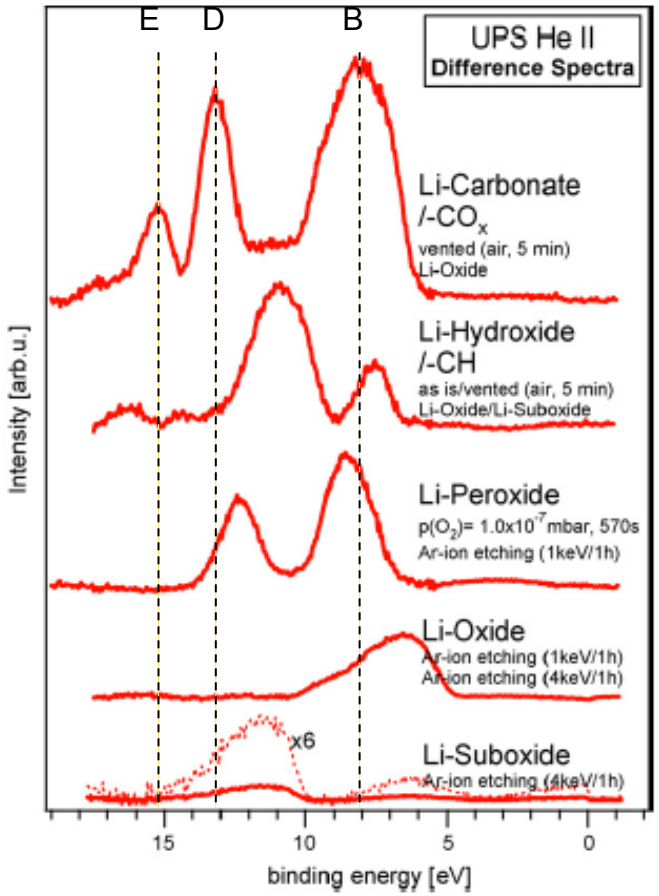
- **B** (8.5 eV): O 2p/C 2sp
- **D** (11.5 eV): C 2sp/O 2p
- **E** (15.2 eV): C 2s

Notes:

- Peaks have 3 prominent peaks resembling (though not perfectly) Li-Carbonate peaks found by Ensling.
- Spurious spectra occurred for unknown reasons.
- Since UPS probes <1 nm, it is expected that Mo probe sample will be similar to ATJ samples.

Initial Results





- Peaks from post-mortem probe samples resemble Li-Carbonate peaks seen by D. Ensling.

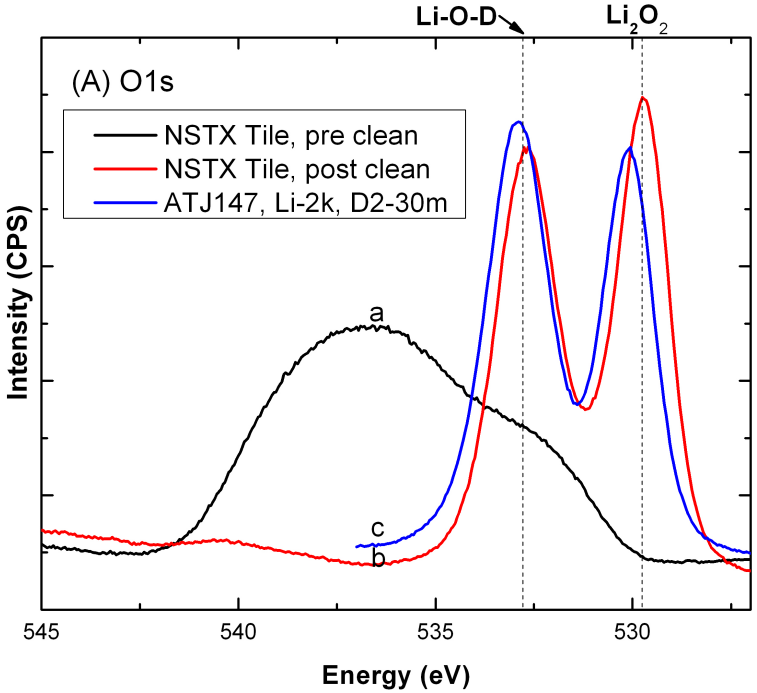
Conclusions

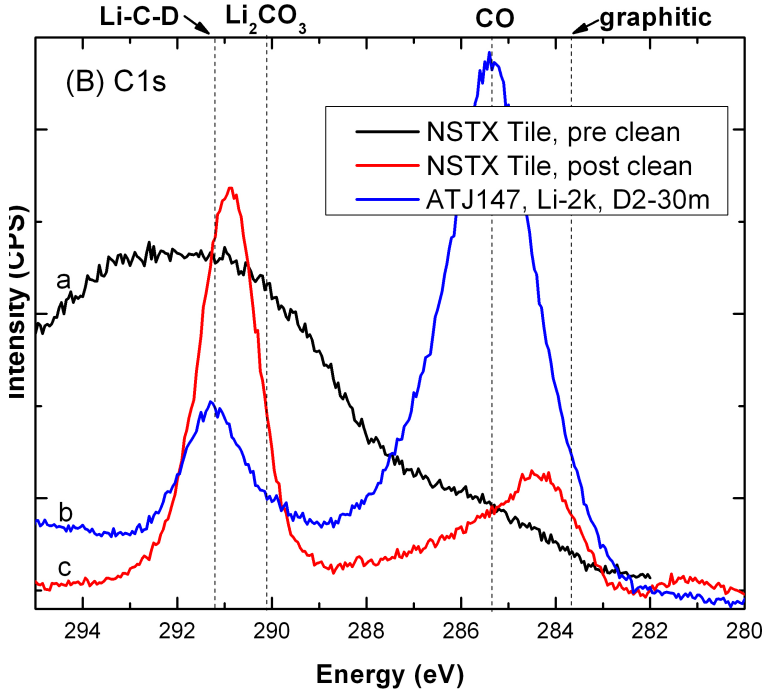
- X-ray photoelectron spectroscopy
  - We can observe the peaks responsible for D retention.
    - Li-O-D at 533.0 eV
    - Li-C-D at 291.4 eV
  - We cannot conclusively isolate O from C-related bonds C from O-related bonds.
- Ultraviolet photoelectron spectroscopy
  - Replicated results similar to other studies, thus validating this method for lithiated graphite.
  - Will now begin a full campaign of controlled studies
  - Equipment will arrive shortly!

EXTRAS

Results – Connect to reality

- How do offline, control experiments compare to post mortem tiles?



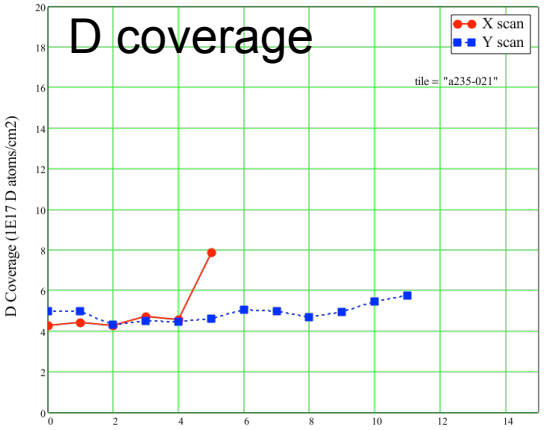


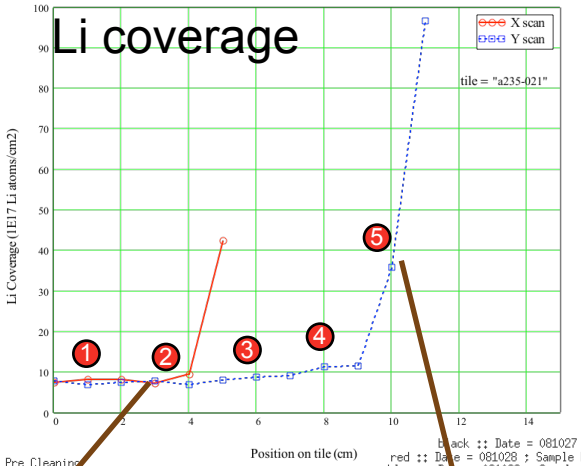
Post mortem tiles exhibited broad peaks; amorphous nature and surface contamination are suspected contributors.

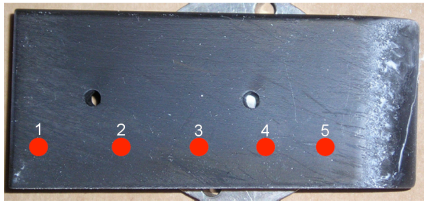
After cleaning procedure (Ar sputtering and heating), peaks reflect those discovered in controlled experiments.

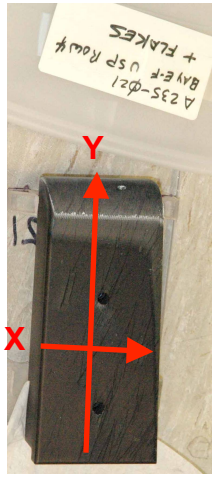
Comparisons of Ion Beam data with XPS

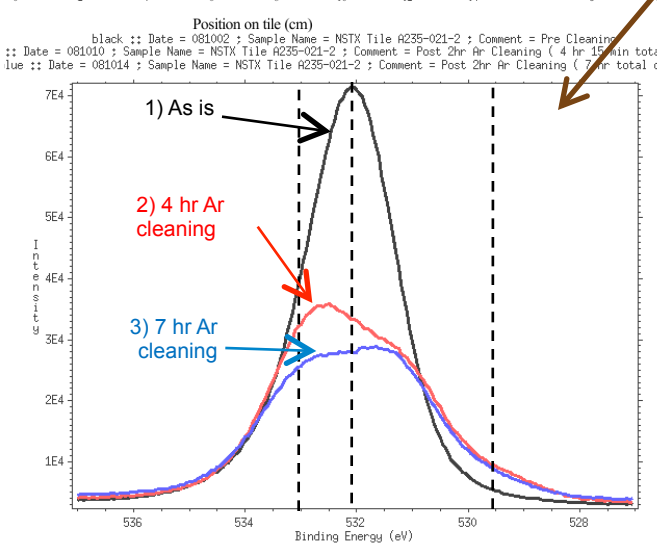
- Lithium dependence on surface chemistry

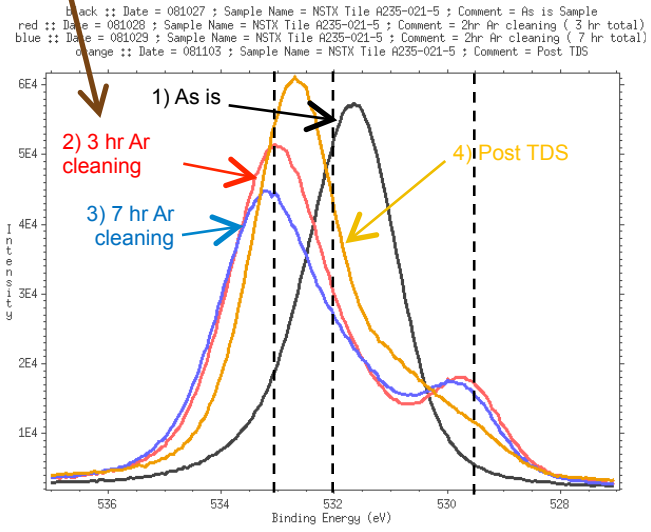












Ion Beam Analysis of Li and D on Tiles from NSTX, W. Wampler, 2006

NSTX Tile A235-021

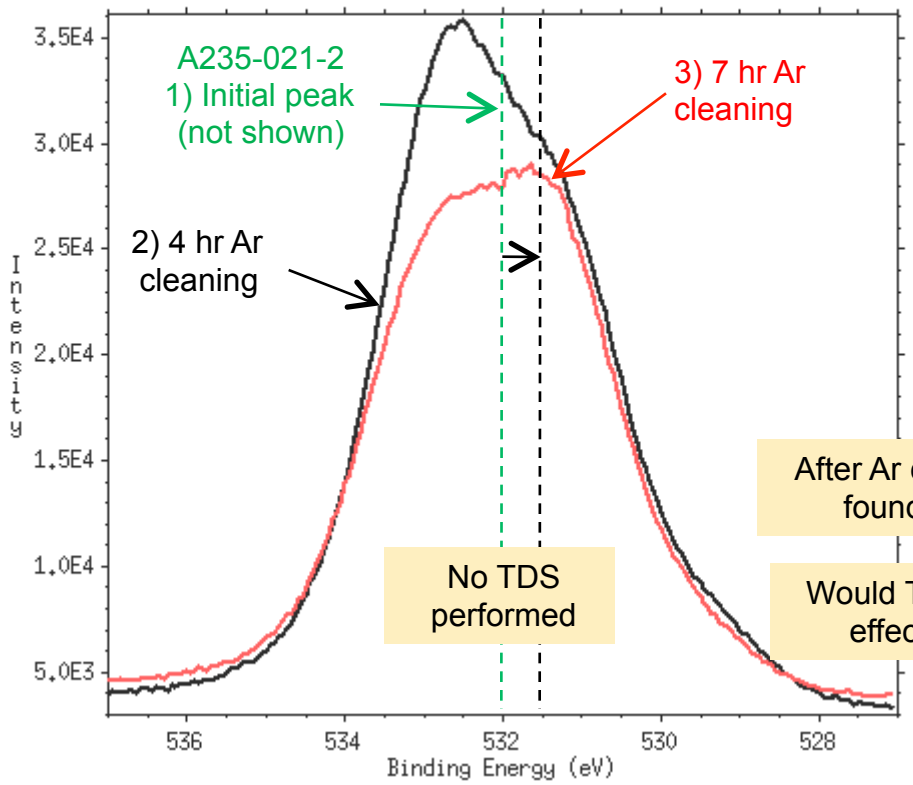
AJT139 vs. post-mortem tile near LITER

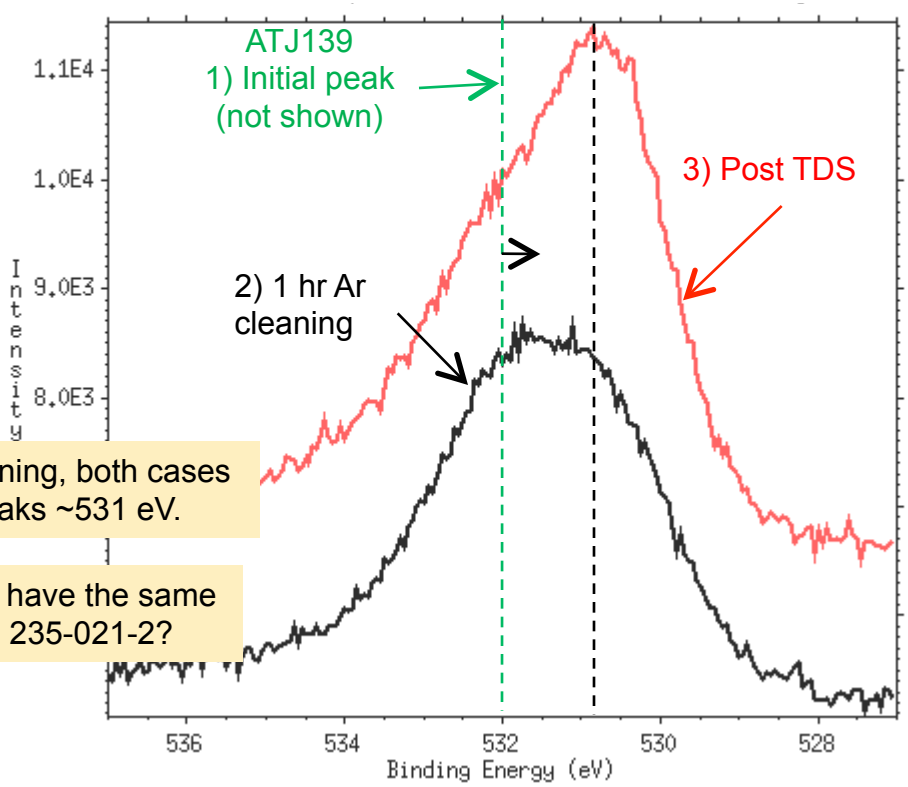
NSTX Tile A235-021-2

•Staged Ar cleaning

ATJ139

- Lithium conditioning
- 6 NSTX NB plasma shots
- Ar cleaning
- TDS performed at Purdue

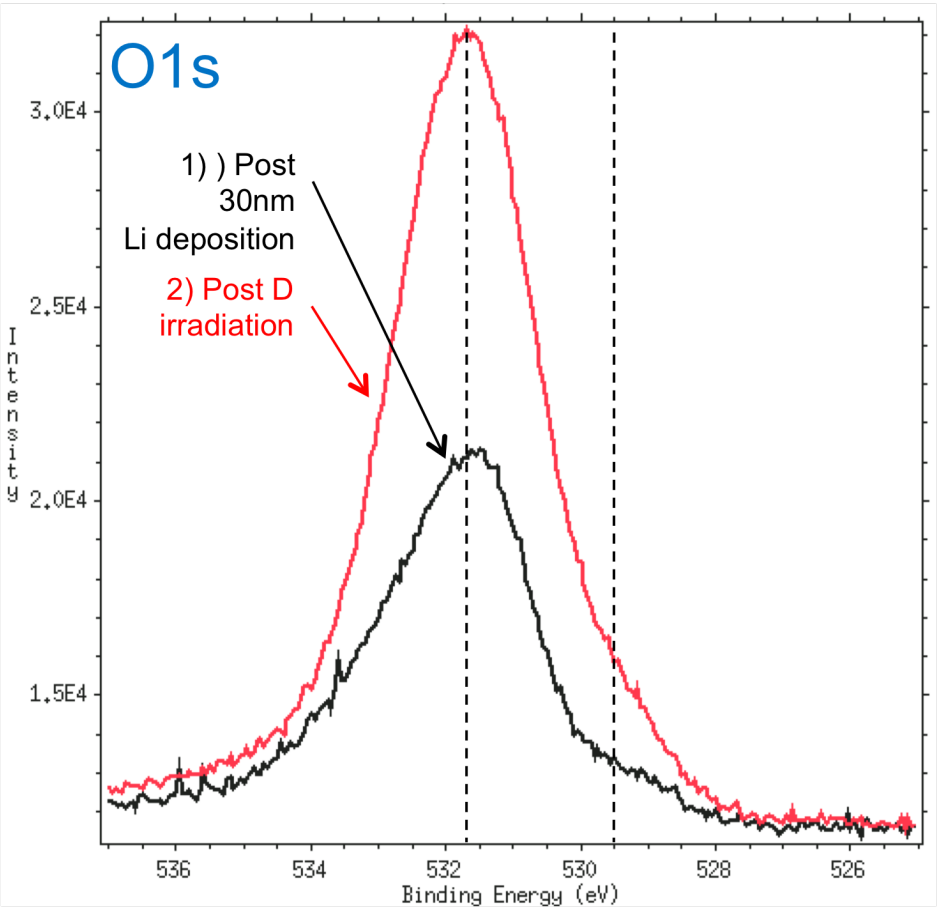


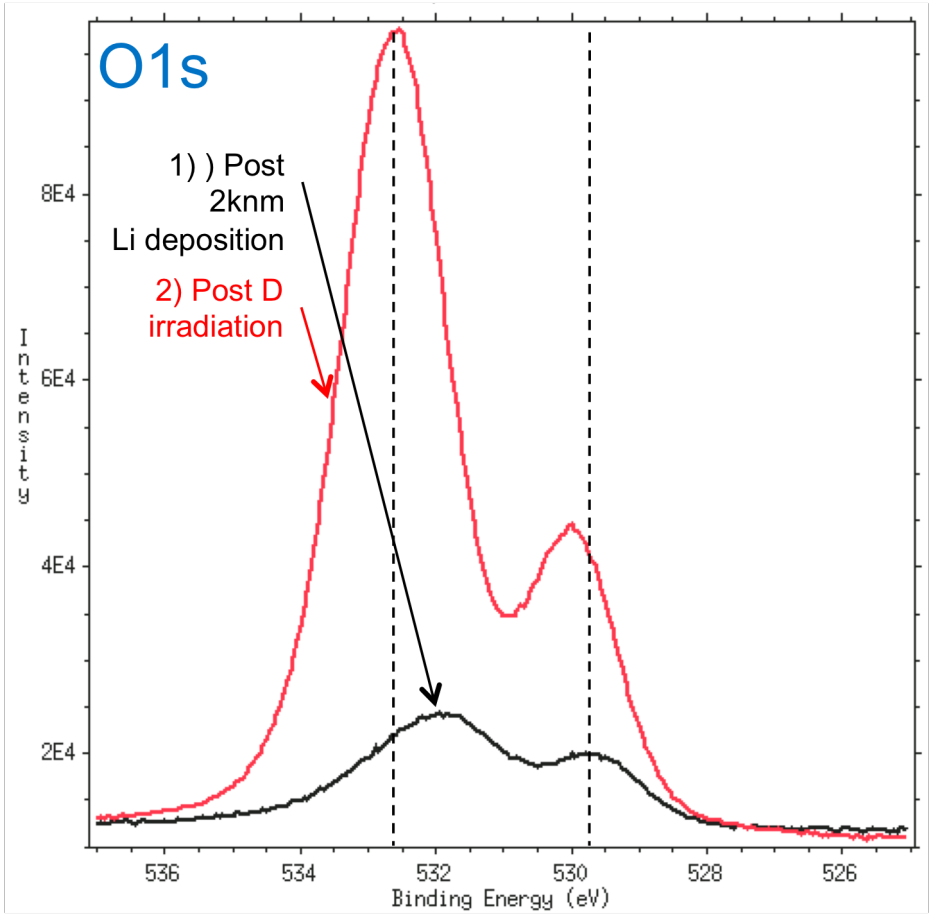


After Ar cleaning, both cases found peaks ~531 eV.

Would TDS have the same effect on 235-021-2?

Lithium dose affects Li-D-O-C functionality





Li-30nm post deposition, post D irradiation

Li-2000 nm post deposition, post D irradiation

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