Deciphering energetic deuterium ion interactions with lithiated ATJ graphite

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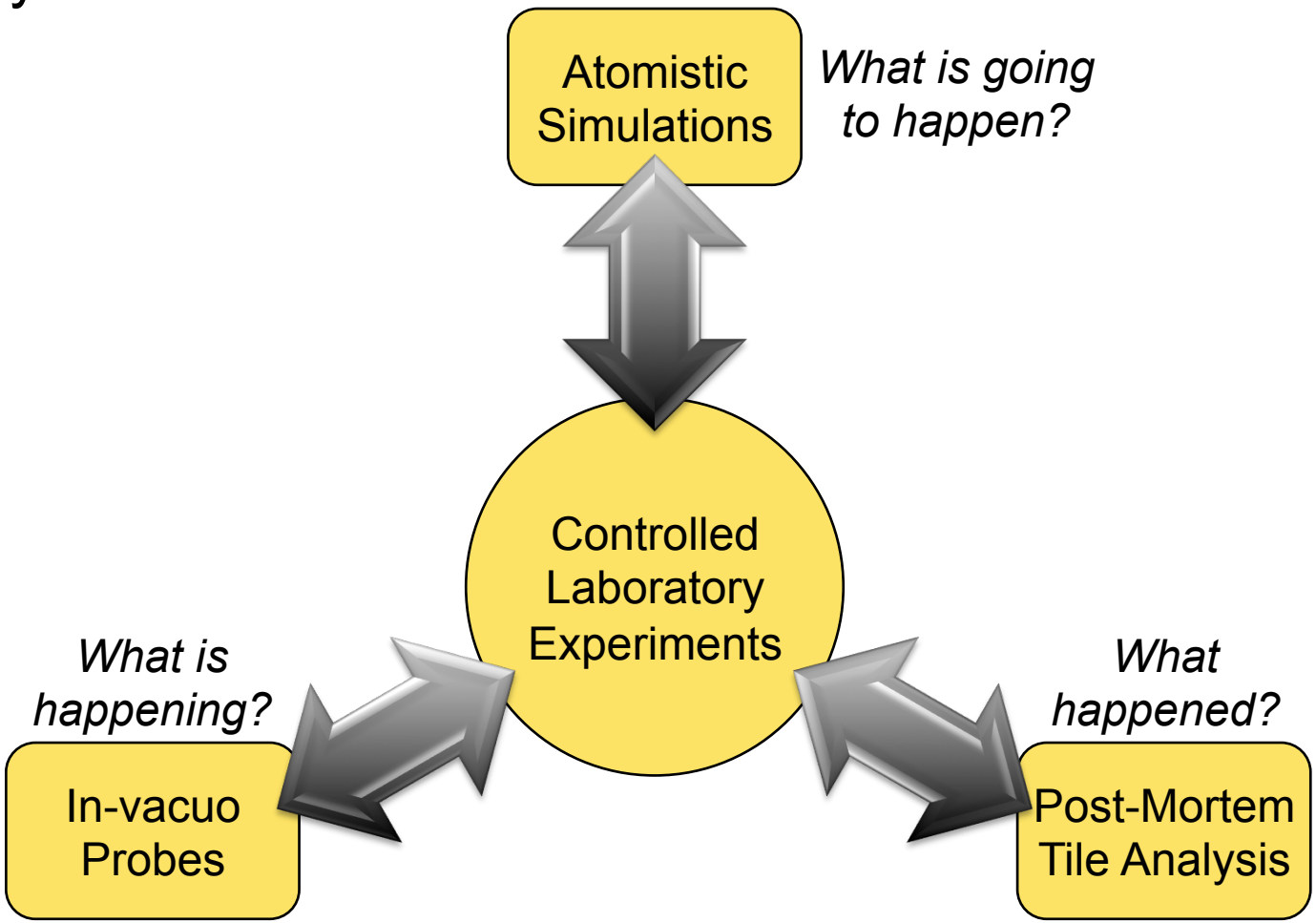
PURDUE ENGINEERING

Outline

- Motivation
- Analysis techniques
- X-ray photoelectron spectroscopy (XPS)
  - Controlled laboratory experiments
  - Post mortem tile analysis
  - Deuterium fluence studies
- Mid-point conclusions
- Ultraviolet photoelectron spectroscopy (UPS)
  - Review of work
  - Controlled experiments
- Summary and future work

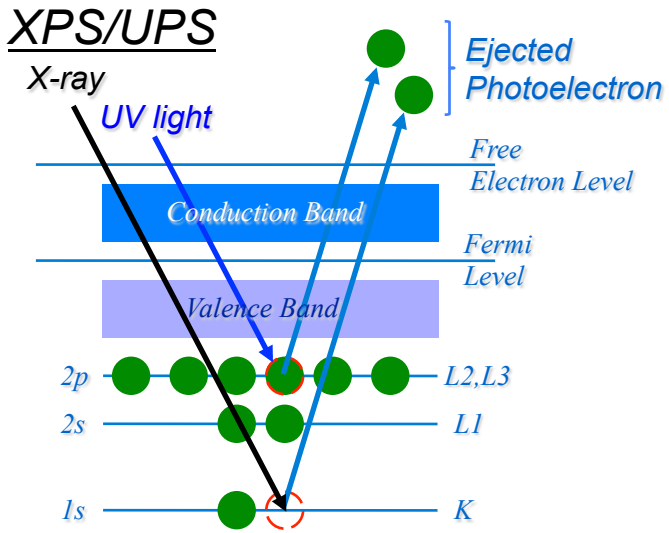
Motivation

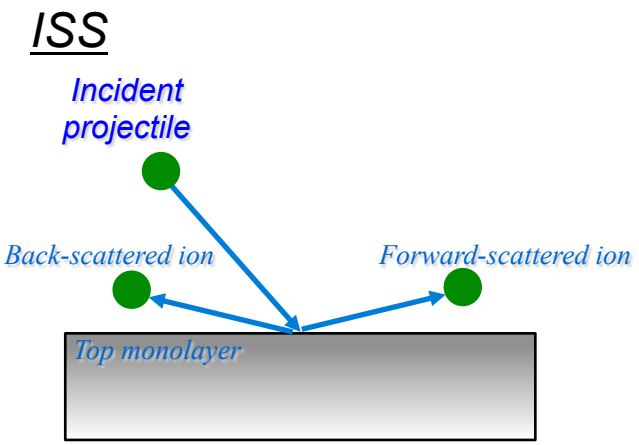
- Connecting laboratory experiments to the tokamak edge.
- Why in-situ in the context of lithium?



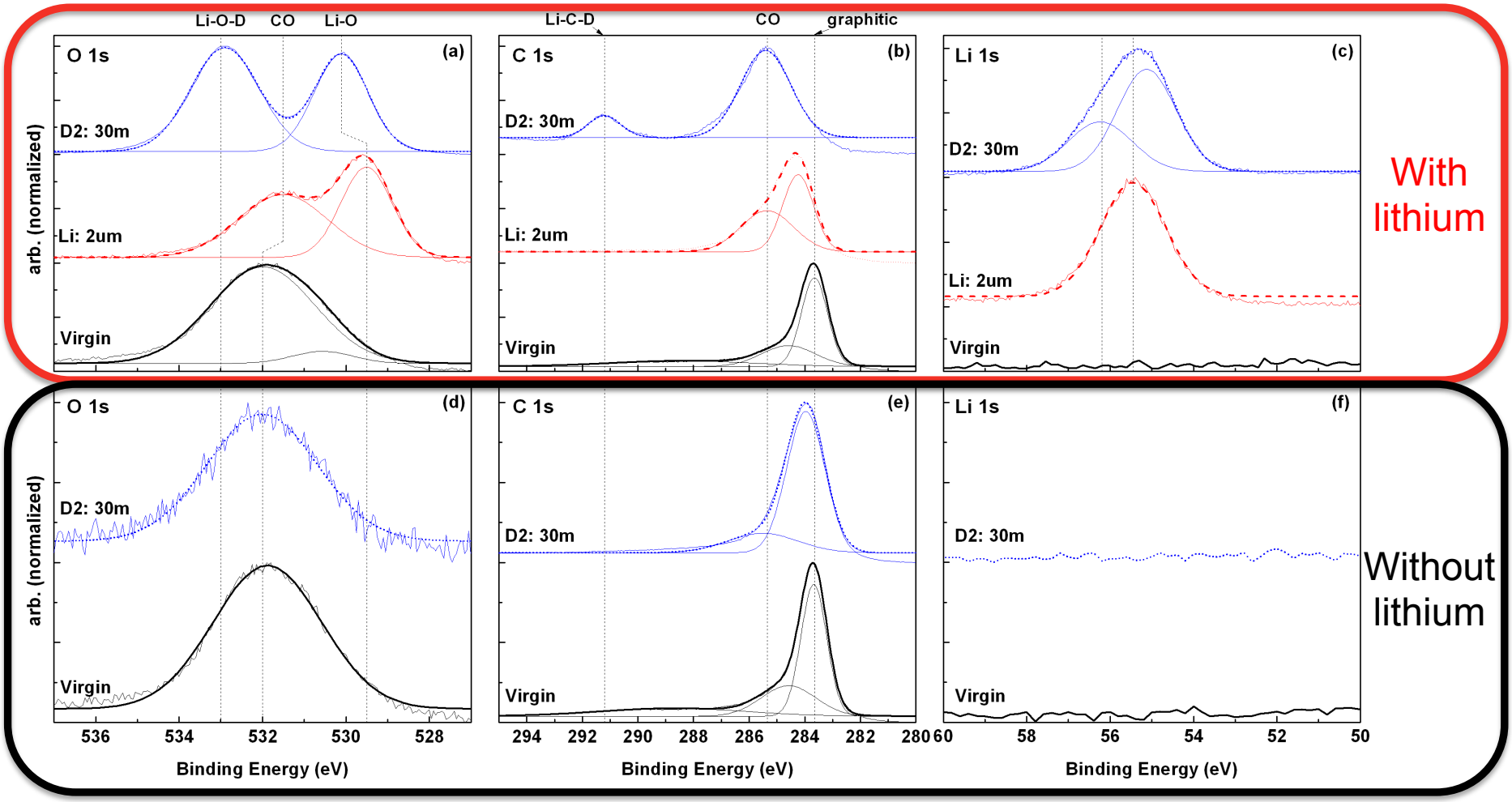
Analysis Techniques

- X-ray Photoelectron Spectroscopy (XPS)
  - X-rays interact with sample inducing core photoelectron ejection. Photoelectron energy is characteristic of sample material/element.
  - Chemical information from top 5-10 nm.
- Ultraviolet Photoelectron Spectroscopy (UPS)
  - UV light excites and ejects valence electrons. Photoelectron energy is characteristic of inherent chemical binding state of material.
  - UPS probes the top < 1nm of a surface.
- Ion-Scattering Spectroscopy (ISS)
  - Ions scatter from top monolayers (2-5 Å). Those projectile ions that survive neutralization are detected.
  - Provides qualitative identification of surface species when target elements are more massive than the projectile.
- We look for changes in spectral peaks to identify changes in our samples.





XPS: Controlled laboratory experiments

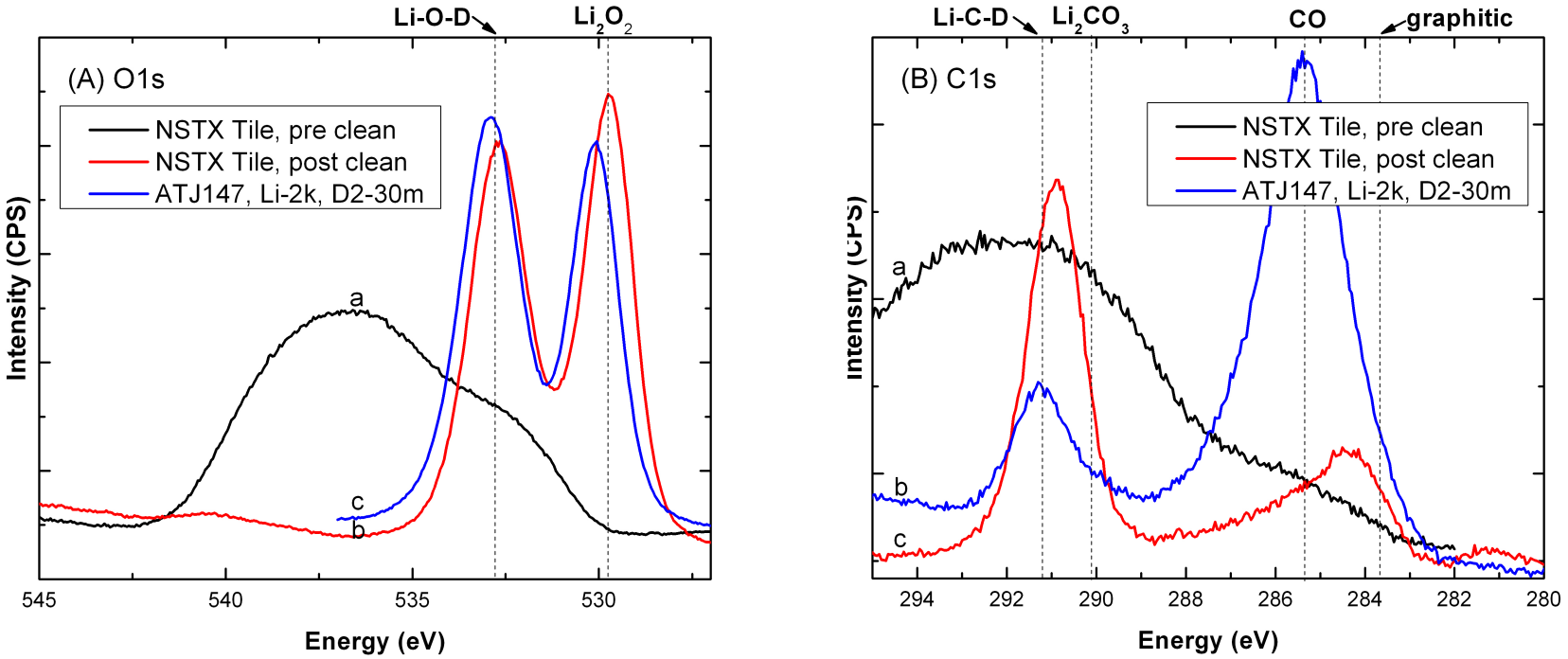


The formation of new peaks or a reproducible peak shift is an indication of a chemical change.

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

XPS: Post mortem tile analysis

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

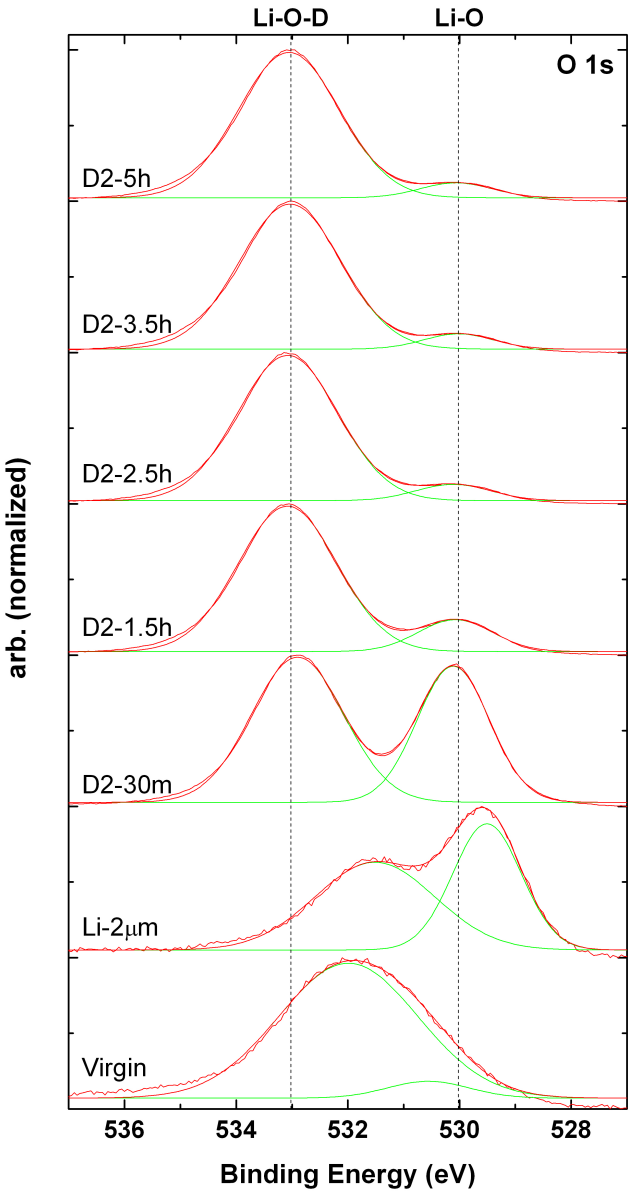


Post mortem tiles exhibited broad peaks; amorphous nature and surface contamination are suspected contributors.

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

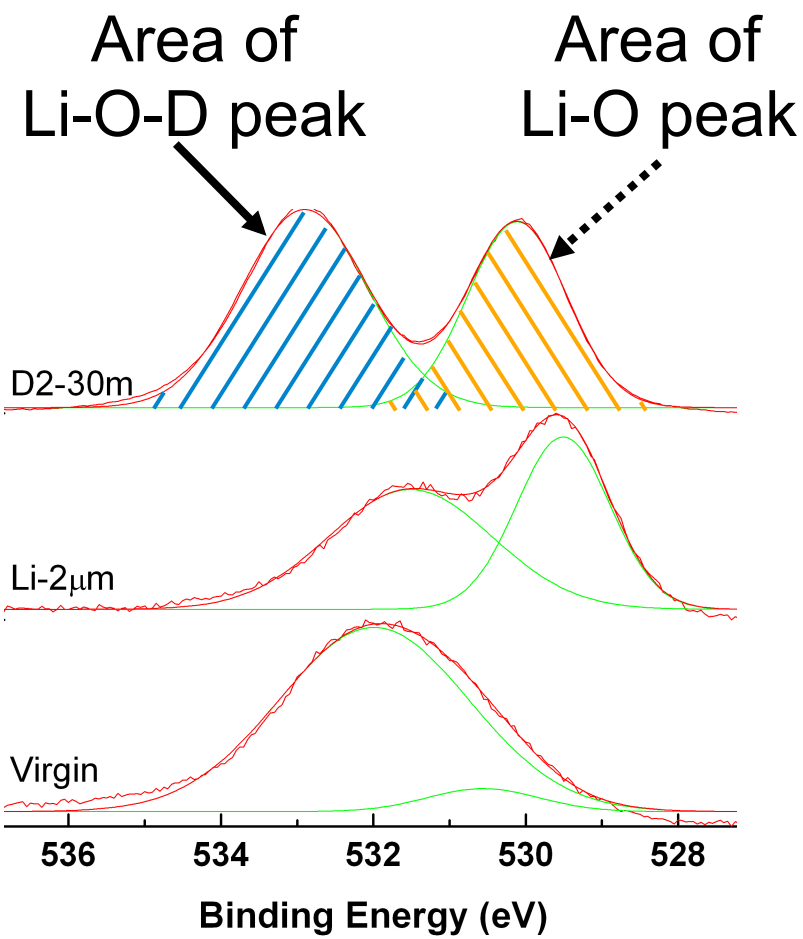
XPS: Deuterium fluence studies

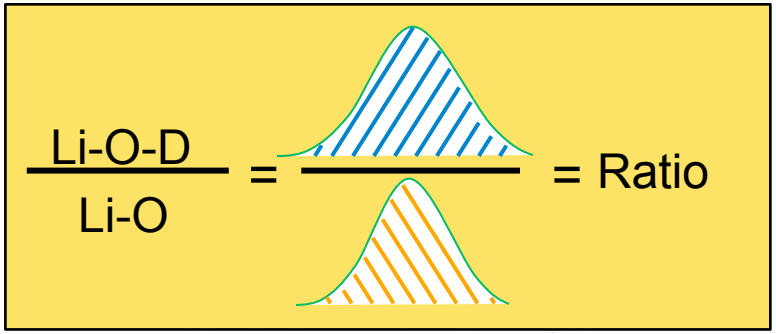
- Introducing deuterium alters surface chemistry of lithiated graphite.
- What happens as more and more deuterium is introduced?
- Can lithium-deuterium saturation be observed through XPS?

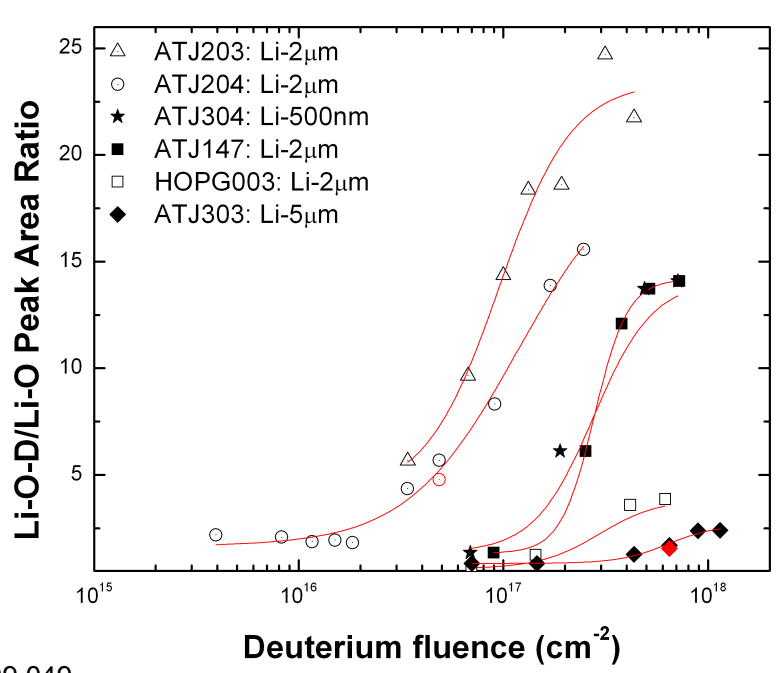


XPS: Deuterium fluence studies

- How is saturation calculated?

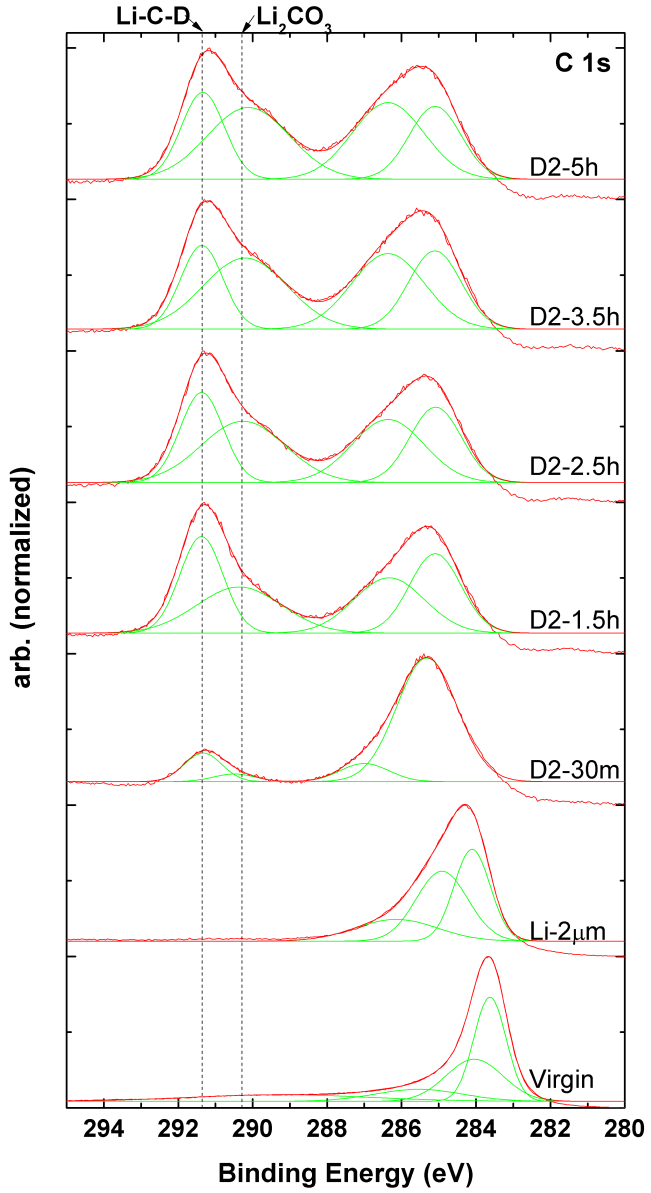




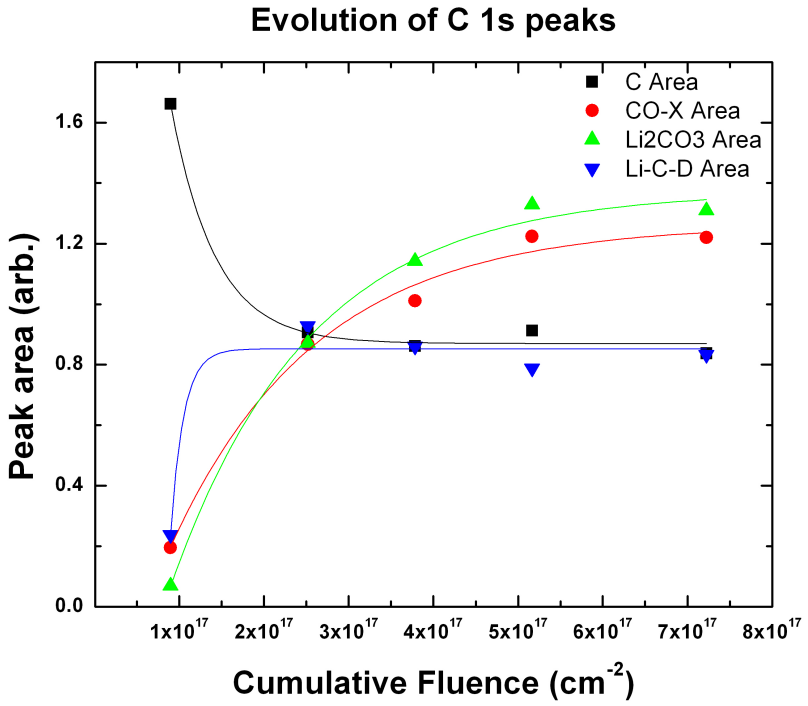


C.N. Taylor et al., J. Nucl. Mater. (2010), doi:10.1016/j.jnucmat.2010.09.049

XPS: Deuterium fluence studies



- What happens in the C1s spectral range as deuterium fluence increases?



(Mid-point) Conclusions

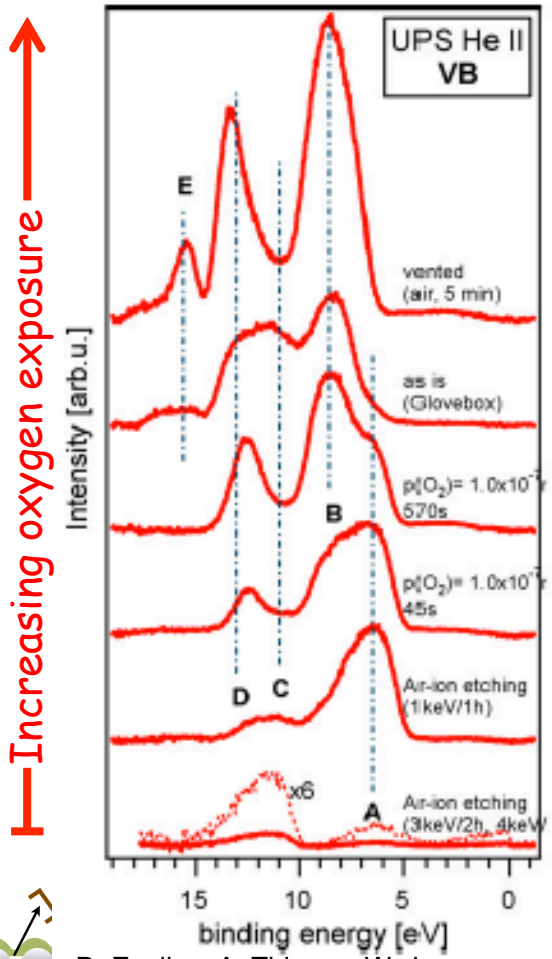
- Deuterium does not bind directly with lithium, but lithium always binds with oxygen and carbon, when present.
  - Atomistic simulations show complementary findings *(see talk by P.S. Kristic)*
- Li-D-O and Li-O interactions can be identified in photoelectron spectra.
- A link between offline laboratory experiments and NSTX wall tiles has been found.
  - Heating a cleaned NSTX tile reveals two peaks that correspond to the Li-D-O and Li-O binding states.
- The point at which lithiated graphite saturates with deuterium can be observed in XPS spectra by comparing ratios of integrated peak areas.
  - A nominal lithium thickness of 2μm becomes saturated with D at ~10^{17}cm^{-2}. Lithium thickness is a fundamental parameter in deuterium retention.
- First step to understand the underlying mechanism(s) by which lithium retains deuterium.

Next steps?

- Are the mechanisms for deuterium retention here unique to lithiated graphite? What mechanisms cross over to alternate materials?
- Chemistry occurs at the valence electron shell. Lets expand our diagnostics to interrogate this region.
- Chemistry of top most surface provides more relevant date to corroborate atomistic modeling.

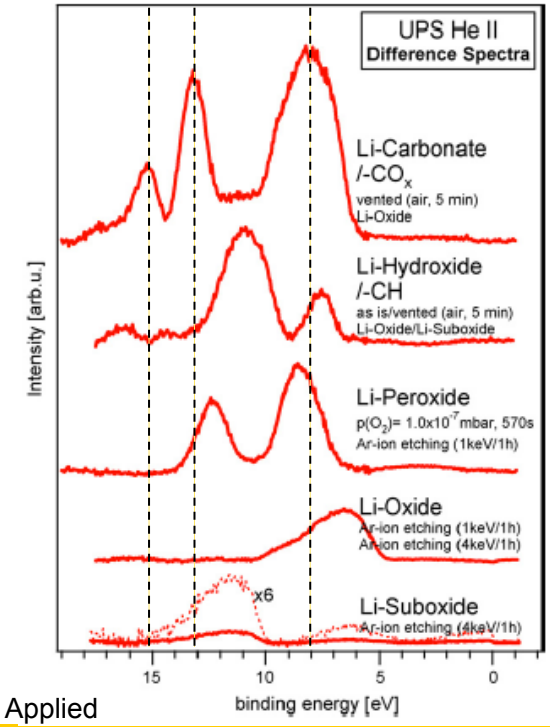
UPS: Initial results

D. Ensling examined lithium surfaces with UPS. Sample was loaded into vacuum under inert atmosphere, cleaned twice via Ar sputtering, and then exposed to two controlled amounts of O2. Afterwards, the sample was exposed to air. UP spectra were collected at each stage of the experiment.

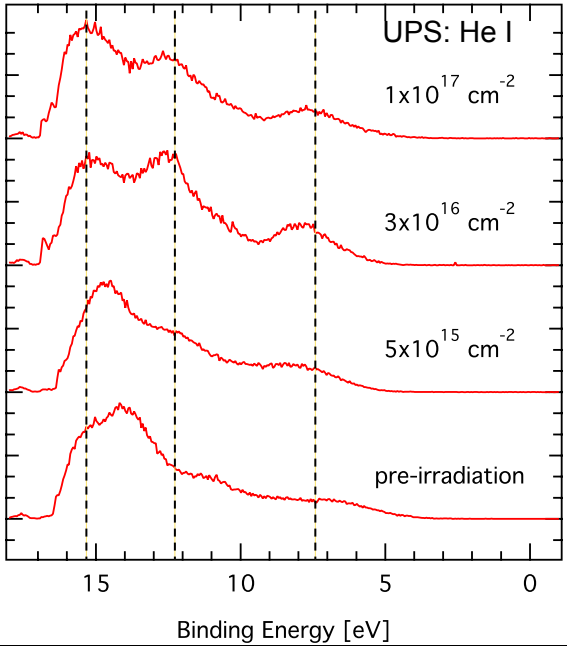


Identification of peaks lines (left)

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



ATJ401p

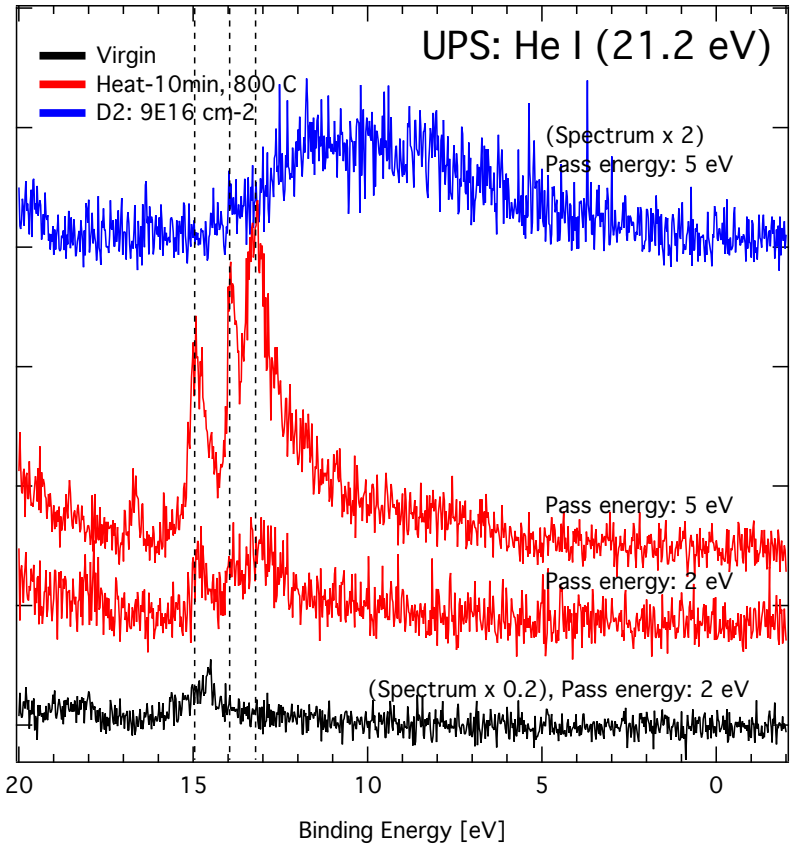


- Monitored a lithiated sample with respect to sputter cleaning and found peaks that resembled carbonates.
- Motivation for additional studies.

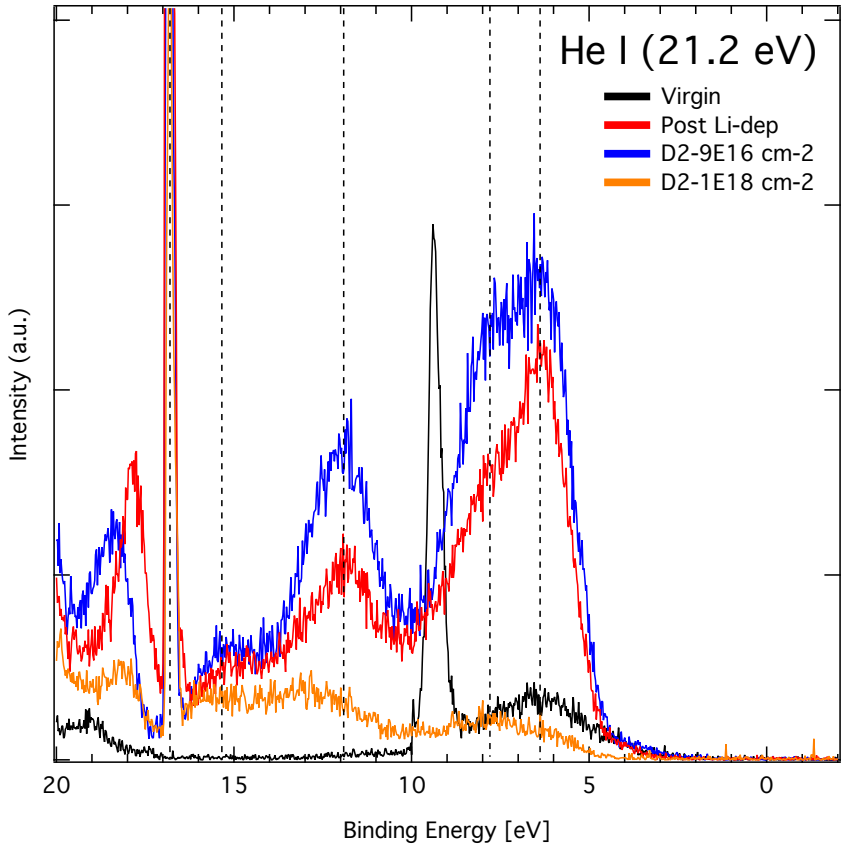
D. Ensling, A. Thissen, W. Jaegermann, Applied Surface Science 255 (2008) 2517–2523

UPS: Control experiments

AT307: No Lithium, D2: 8x10^{16} cm^{-2}



AT309: Li dep, D2: 8x10^{16} cm^{-2}



Heating sample removes surface oxides.

- Lithium deposition results in peaks at 6.5, 7.8, 12.0, 18 eV.
- Deuterium irradiation accentuates the peaks at 7.8 eV and shifts peak from 18 to 18.5 eV.

Summary and future work

- X-ray photoelectron spectroscopy
  - Deuterium does not bind directly with lithium, but lithium always binds with oxygen and carbon, when present.
    - Atomistic simulations show complementary findings (see talk by P.S. Kristic)
  - Li-D-O and Li-O interactions can be identified in photoelectron spectra.
  - A link between offline laboratory experiments and NSTX wall tiles has been found.
    - Heating a cleaned NSTX tile reveals two peaks that correspond to the Li-D-O and Li-O binding states.
  - The point at which lithiated graphite saturates with deuterium can be observed in XPS spectra by comparing ratios of integrated peak areas.
    - A nominal lithium thickness of 2μm becomes saturated with D at ~10^{17}cm^{-2}. Lithium thickness is a fundamental parameter in deuterium retention.
  - First step to understand the underlying mechanism(s) by which lithium retains deuterium.

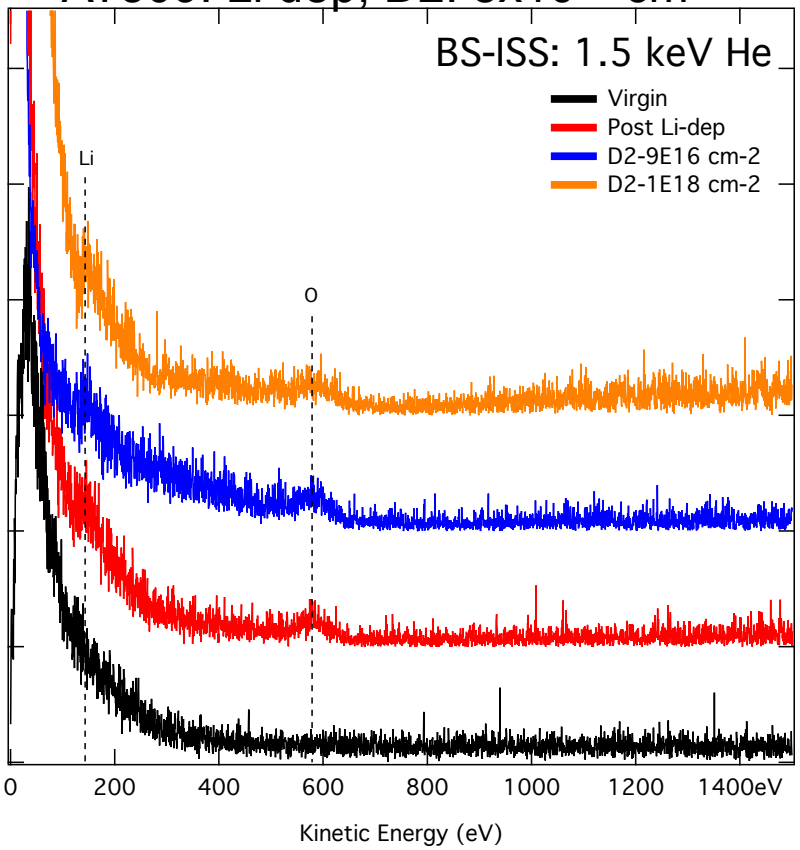
(Repeated)

- Ultraviolet photoelectron spectroscopy
  - First look at controlled experiments indicate that we can use UPS to decipher and de-convolute binding mechanisms identified with XPS.
- Ion scattering spectroscopy
  - Use forward-scattering ISS to as an additional surface characterization technique. Complementary technique, direct recoil spectroscopy (DRS) can directly detect hydrogen on the surface.
- Future work
  - Expand parameter space and continue to systematically use UPS and ISS to identify all binding mechanism responsible for deuterium retention.
  - Systematically investigate deuterium retention via thermal desorption spectroscopy for low and high lithium and deuterium doses, respectively.

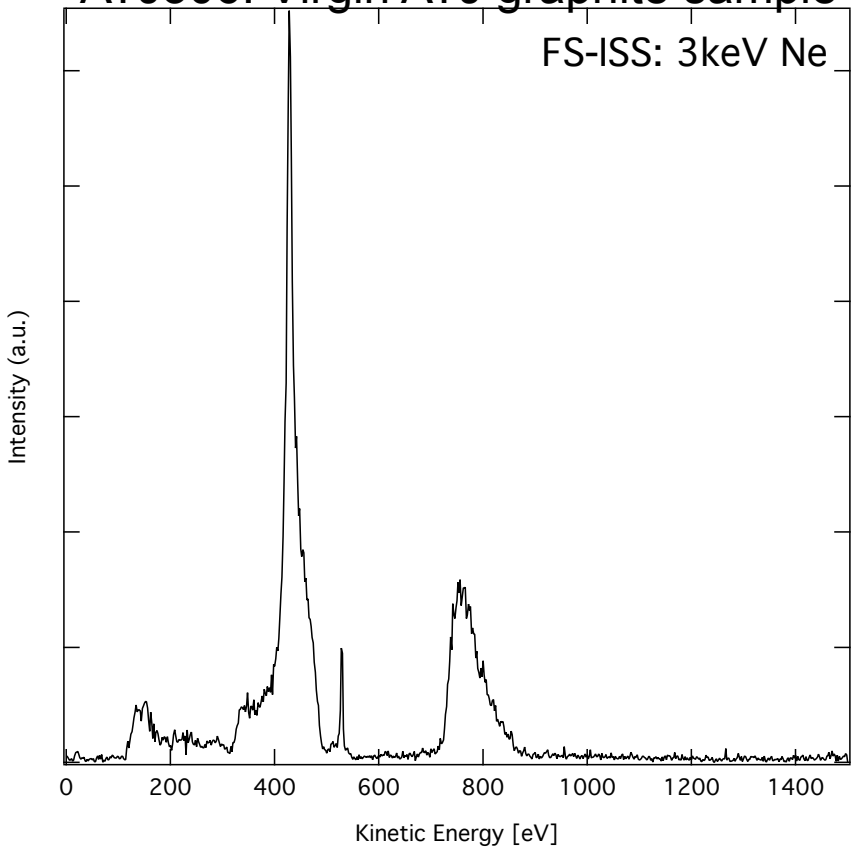
EXTRA SLIDES

ISS: Back- vs. forward-scattering

AT309: Li dep, D2: 8x10^{16} cm^{-2}



ATJ306: Virgin ATJ graphite sample



Back scattering ISS does not reveal much information for ATJ graphite due to its

Forward scattering ISS combines scattered ions and direct recoils. Spectra shows many features that will be investigated in systematic studies.