Surface chemistry analysis of NSTX private flux region and upper vessel tiles

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RSSEL

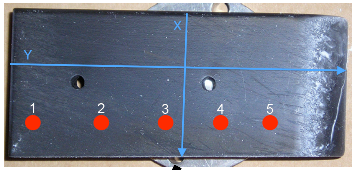
PURDUE ENGINEERING

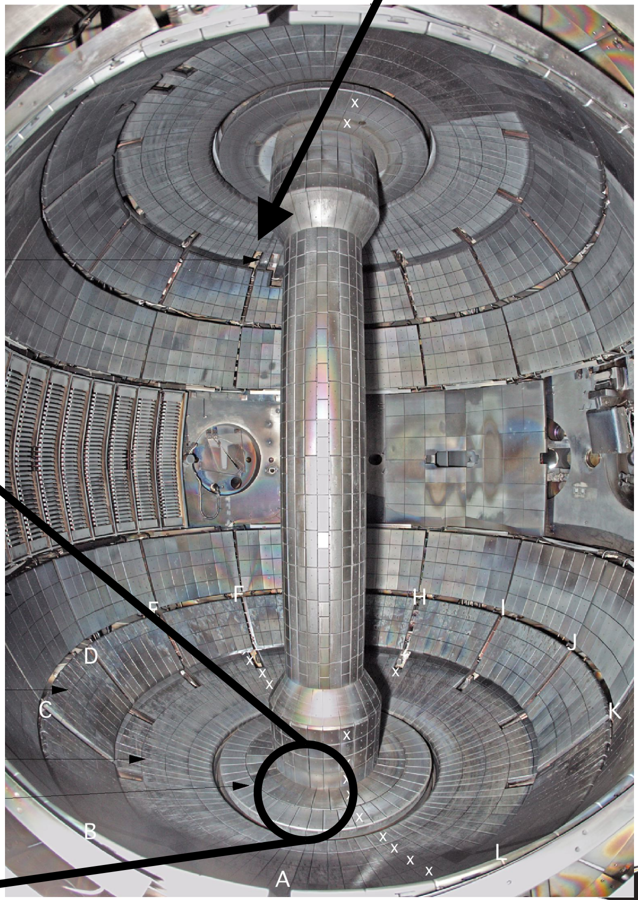
Outline

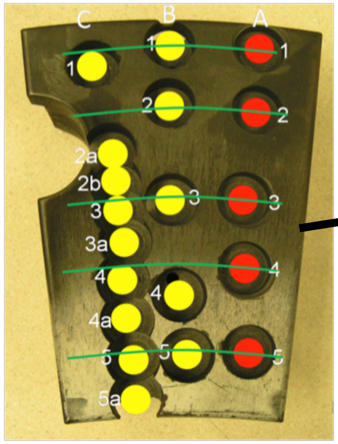
- Background on NSTX tiles
- Offline control experiments
- NSTX tile analysis
  - Cleaning process on various tiles
  - NSTX tile deuterium pumping locations
  - Effect of high-temperature annealing
  - Lithium ‘thickness’ dependence
- Conclusions

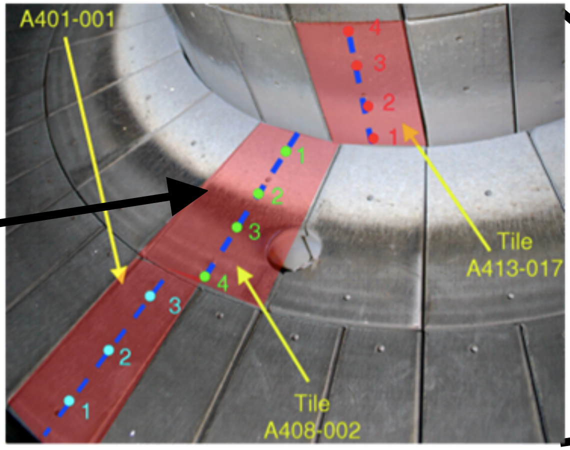
Background on NSTX tiles

- Four 2008 NSTX tiles were used in analysis.
  - Tiles passivate upon air exposure
- Many plasma configurations were used throughout the campaign.
  - Strikepoint locations
  - Wall conditioning (boronization, glow discharge, lithiumization)
- Brief procedure:
  - X-ray photoelectron spectroscopy (XPS) to examine surface chemistry
  - Clean with 1 keV Ar sputtering/annealing









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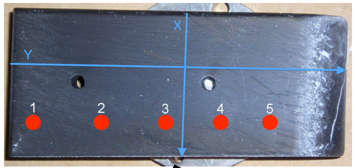
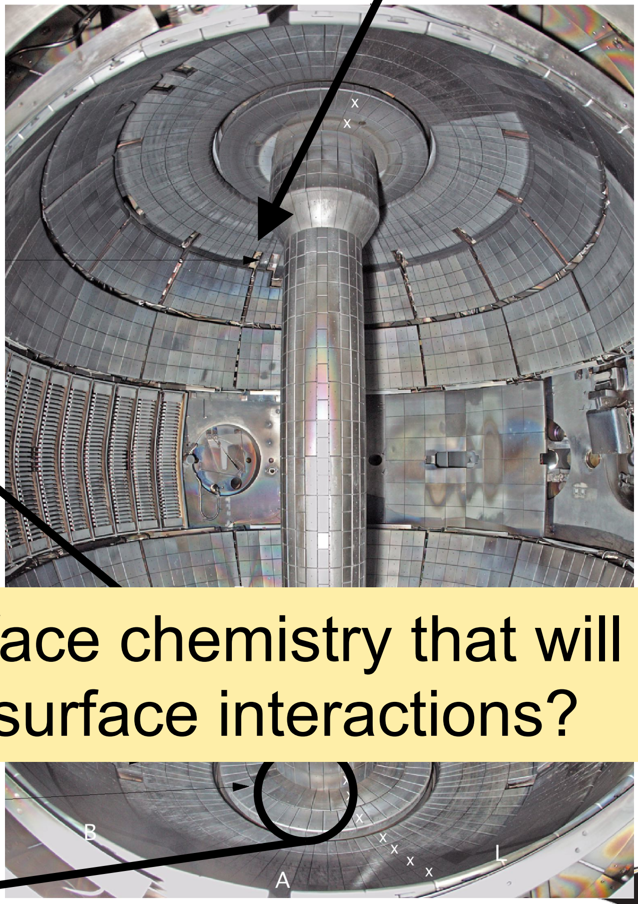
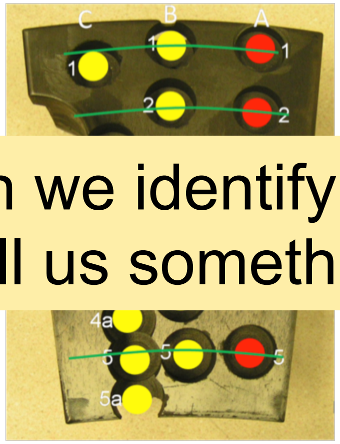
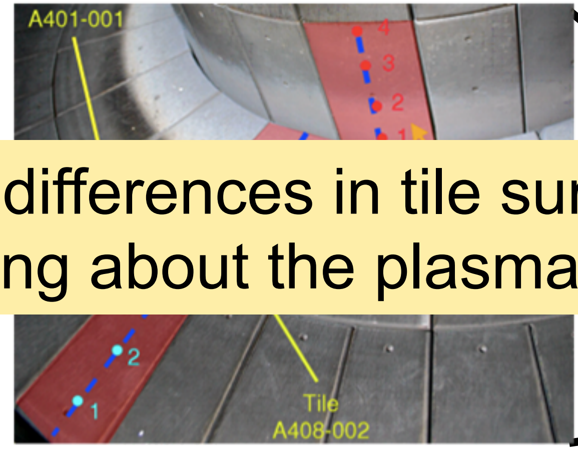




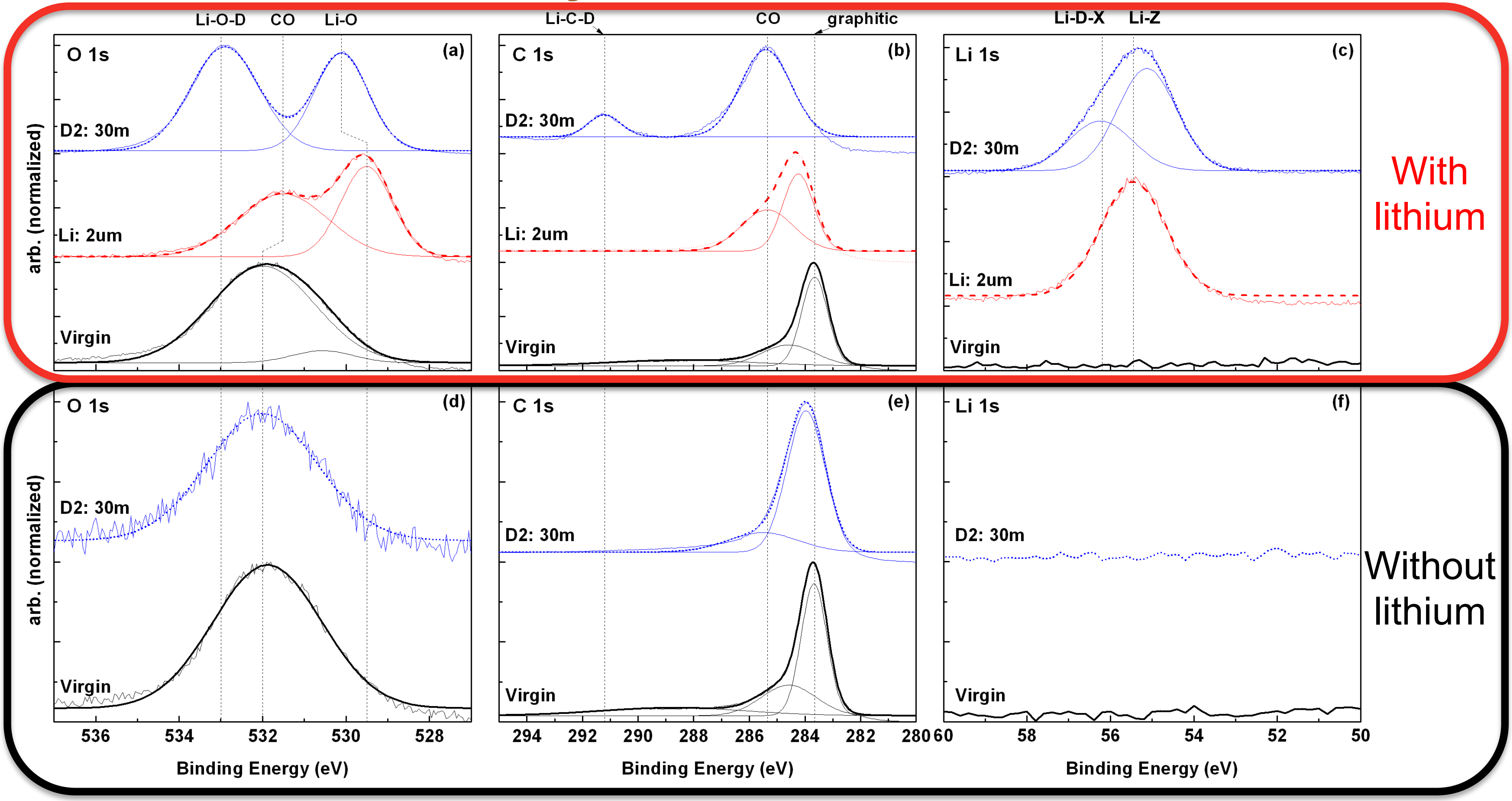




Can we identify differences in tile surface chemistry that will tell us something about the plasma surface interactions?

Offline controlled experiments

…Allow us to identify deuterium related interactions

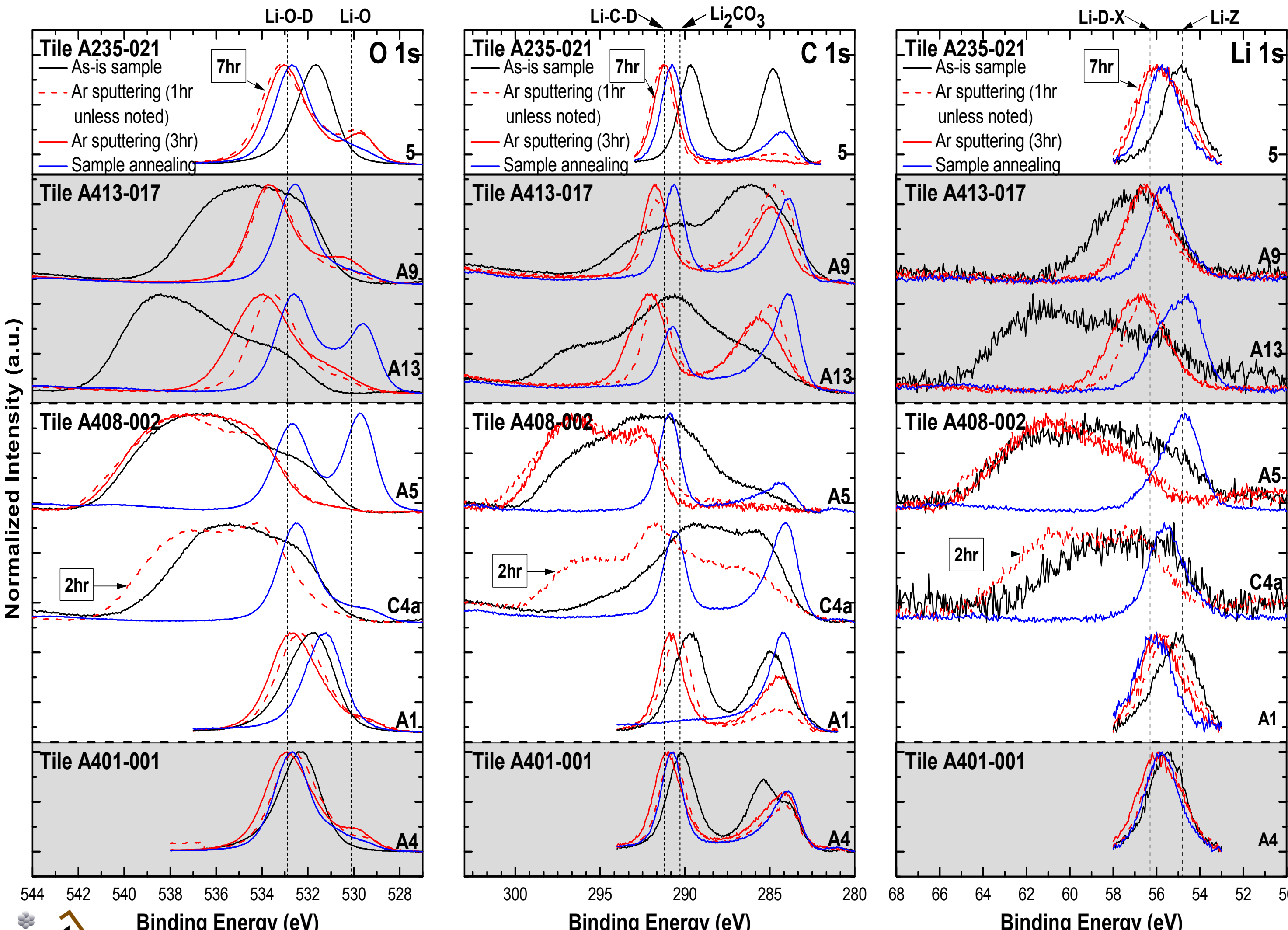


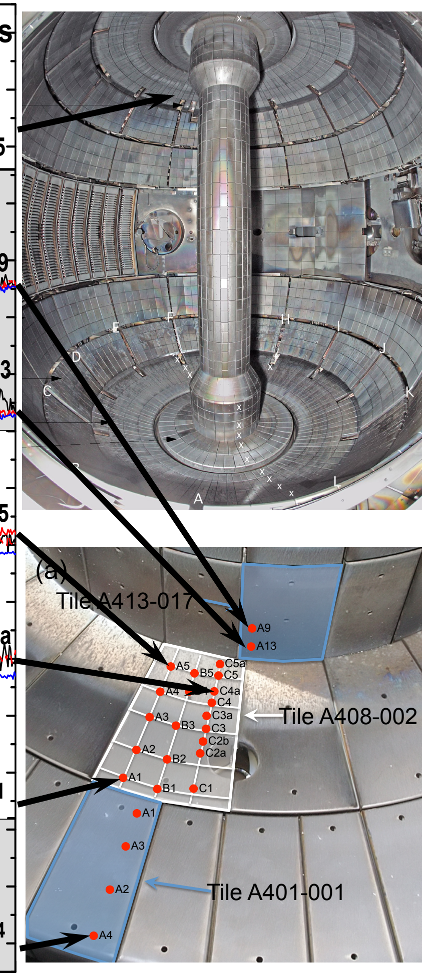
The formation of new peaks or a reproducible peak shift is an indication of a chemical change.

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

Cleaning process on various tiles

-- As is
-- Ar sputtered
-- Annealed





C.N. Taylor, et al., Nucl. Fusion, submitted 2011.

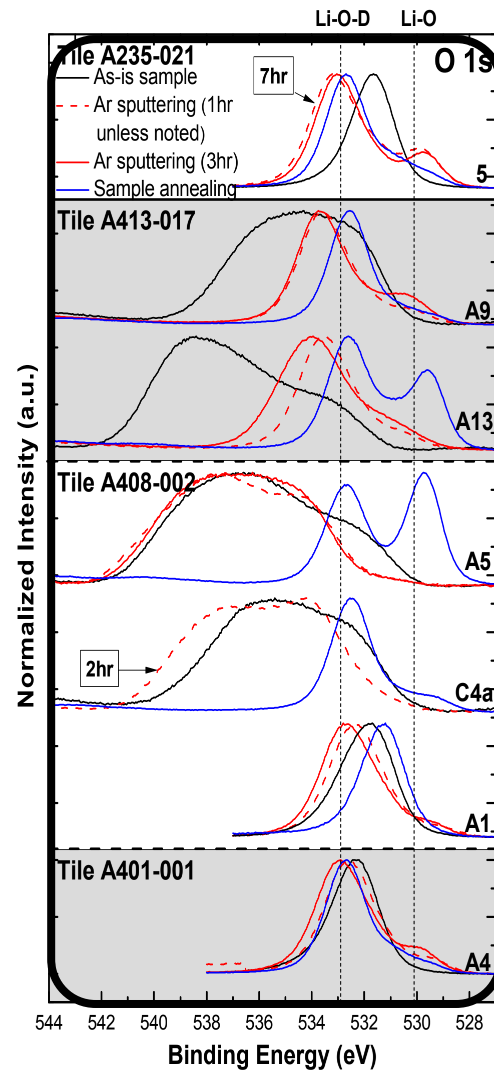
Cleaning process on various tiles

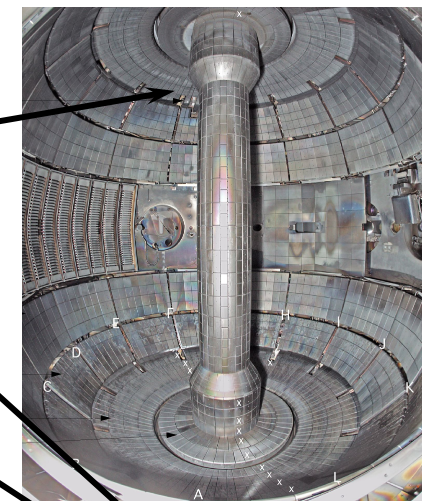
As is spectra are inconsistent

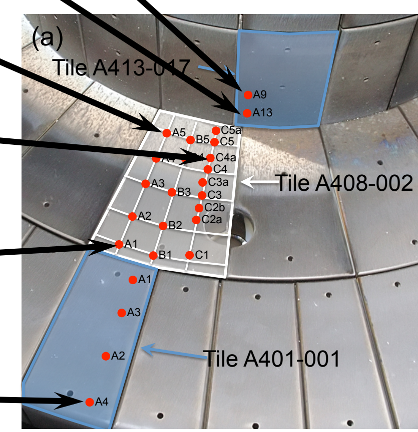
-- As is
-- Ar sputtered
-- Annealed

Consider the "as is" spectra (black).

- *As is spectra are not consistent from tile to tile.*
- *Spectra uncharacteristic of passivated Li-graphite, graphite, or lithium.*
- *Unusual broad XPS peaks*







C.N. Taylor, et al., Nucl. Fusion, submitted 2011.

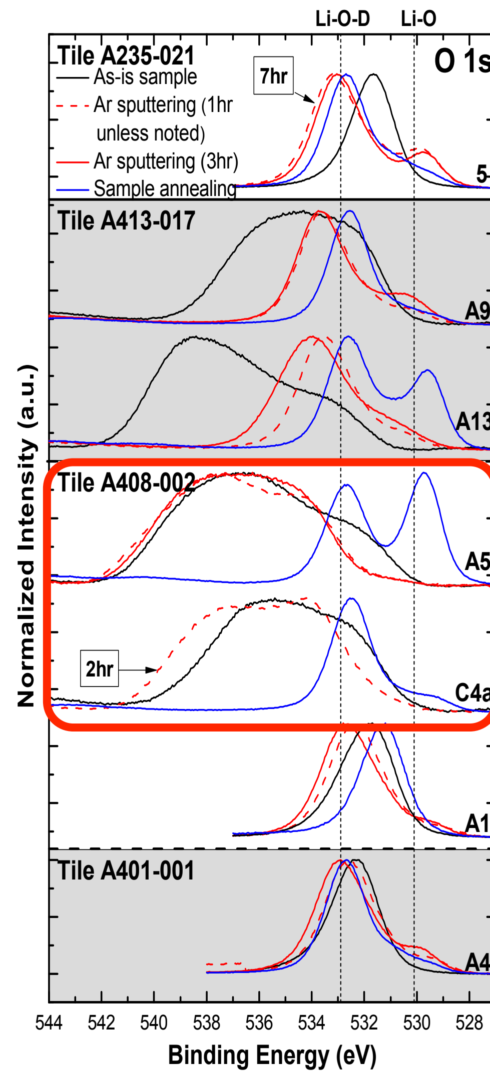
Cleaning process on various tiles

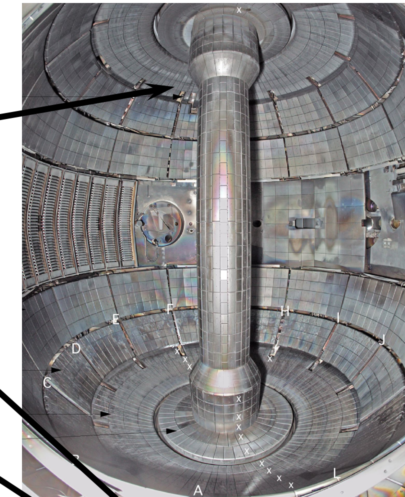
Sputter cleaning recovers "most" locations

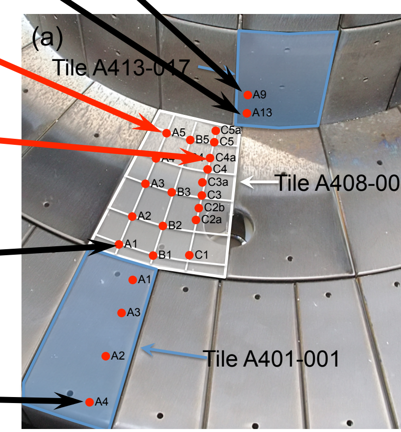
-- As is
-- Ar sputtered
-- Annealed

Consider the "Ar sputtered" spectra (red).

- *Sputter cleaning (red peaks) does not 'repair' broad spectra for all samples*







C.N. Taylor, et al., Nucl. Fusion, submitted 2011.

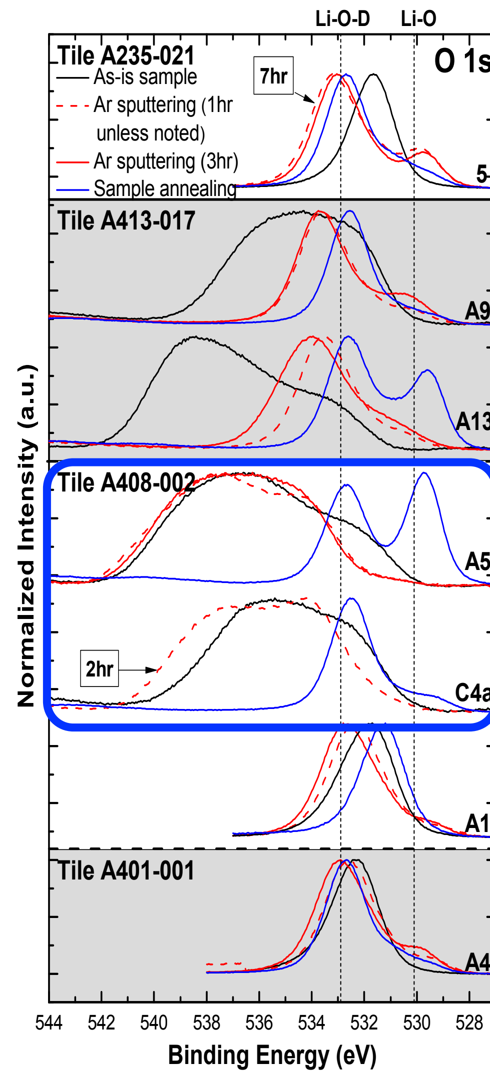
Cleaning process on various tiles

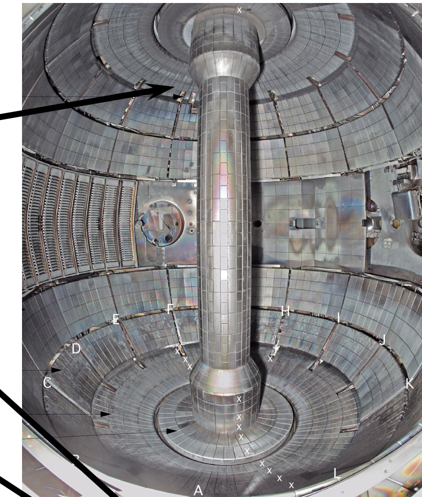
Sputtering and heating repairs *all* samples

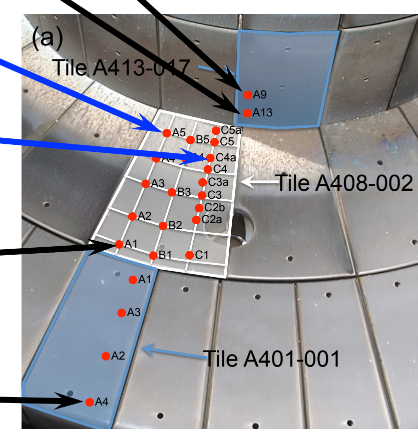
-- As is
-- Ar sputtered
-- Annealed

Consider the "Annealed" spectra (blue).

- *Heating to ~550ºC (blue peaks) transforms and 'repairs' broad spectra, revealing Li-O-D and Li-C-D peaks characteristic of deuterium retention.*







C.N. Taylor, et al., Nucl. Fusion, submitted 2011.

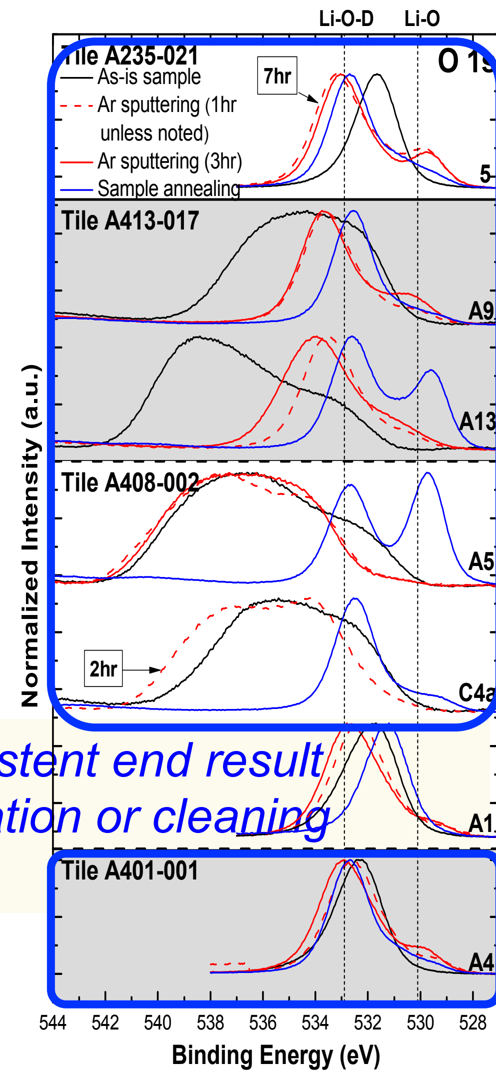
Cleaning process on various tiles

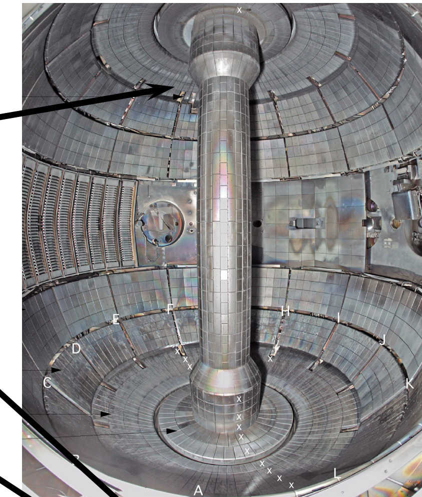
Consistent chemistry regardless of tile location

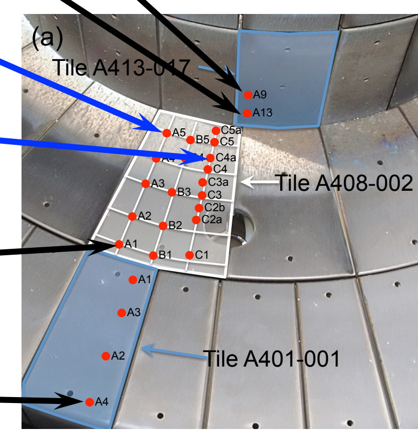
-- As is
-- Ar sputtered
-- Annealed

Consider the "Annealed" spectra (blue).

Cleaned peaks (in blue) show consistent end result regardless of sample extraction location or cleaning process!





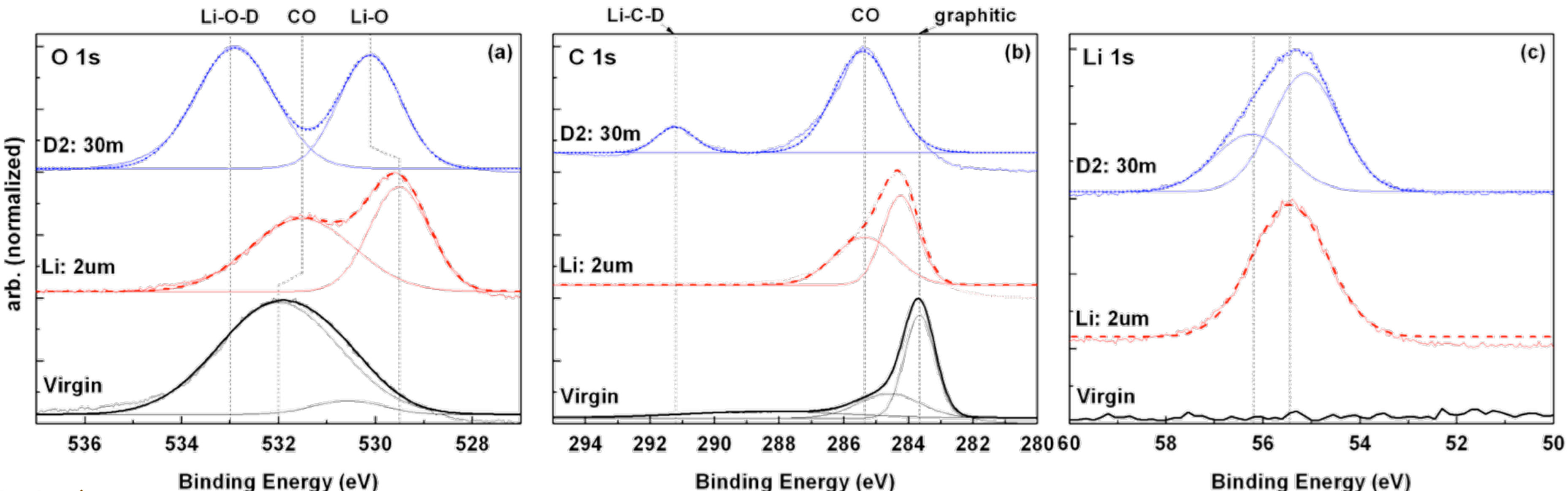


See: J.P. Allain PI2.06

C.N. Taylor, et al., Nucl. Fusion, submitted 2011.

NSTX tile analysis

- So what do these peaks mean?
- Reminder:
  - From controlled studies, peaks have been identified that are associated with deuterium retention.
    - Li-O (529.5 eV) is converted to Li-O-D (532.9 eV)
    - Li-C-D forms at 291.2 eV

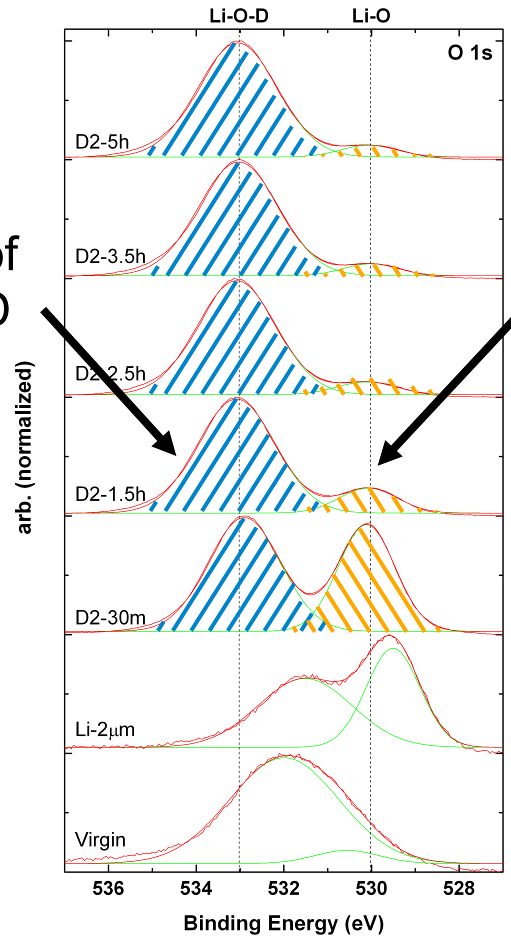


C.N. Taylor, B. Heim, J.P. Allain, Journal of Applied Physics 109, 053306 (2011)

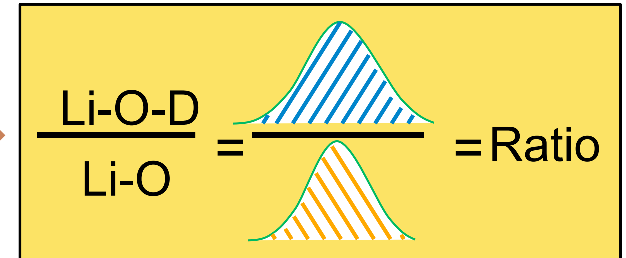
Deuterium saturation of lithiated graphite

- How is saturation calculated?

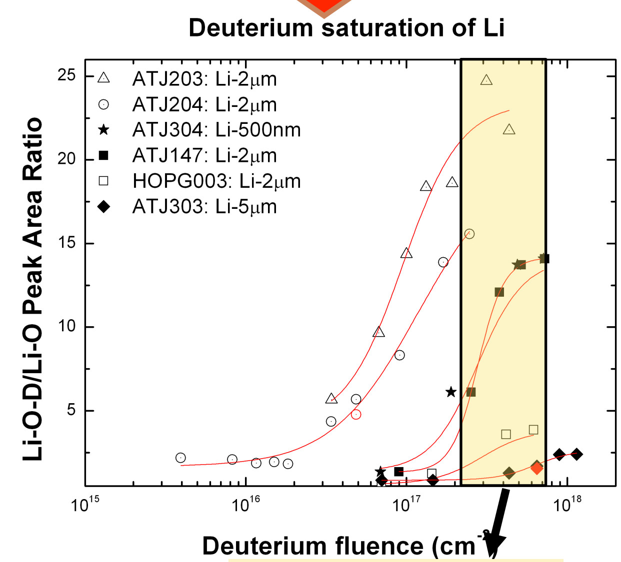
Area of Li-O-D peak



Area of Li-O peak



Li-O bonds are consumed and converted into Li-O-D interactions!



Saturation region
Fluence ~ $10^{17}cm^{-2}$

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

NSTX tile D pumping locations

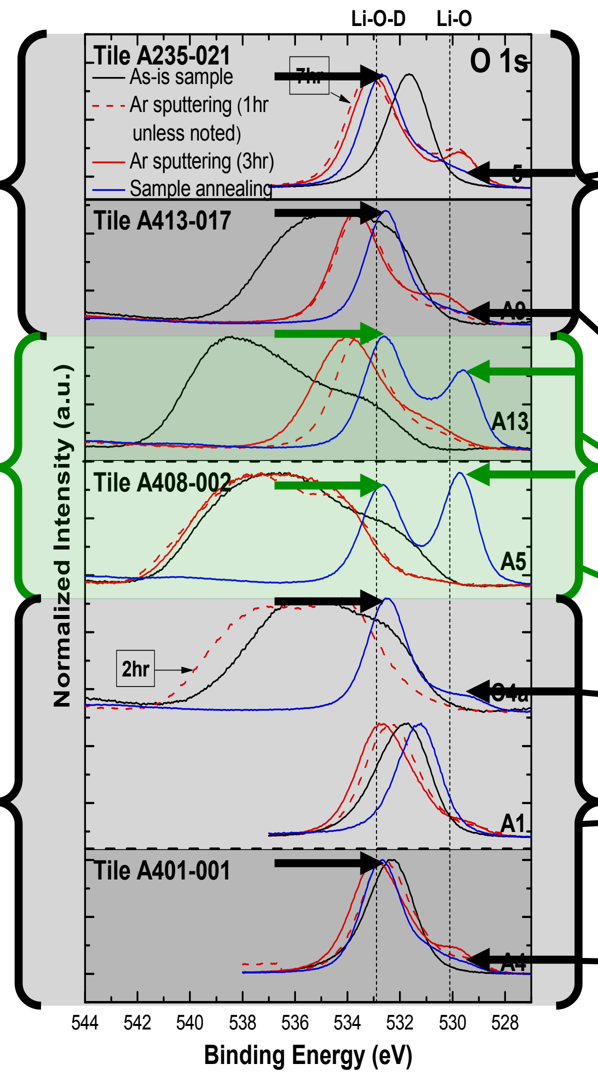
Samples ± 2.5 cm from corner aren't saturated

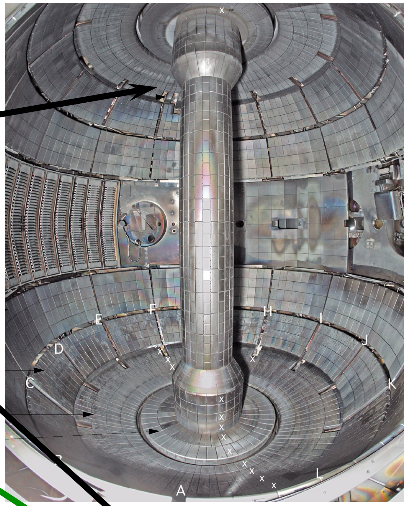
-- As is
-- Ar sputtered
-- Annealed

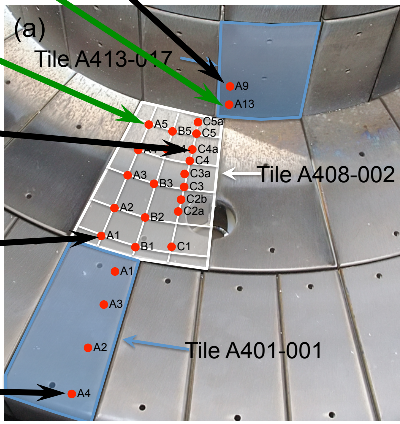
Deuterium-saturated, regions of high-recycling

Locations of persistent deuterium pumping

The Li-O-D peaks dominate at the expense of Li-O. Locations where Li-O is depleted are likely saturated and do not further aid in D retention.







C.N. Taylor, et al., Nucl. Fusion, submitted 2011.

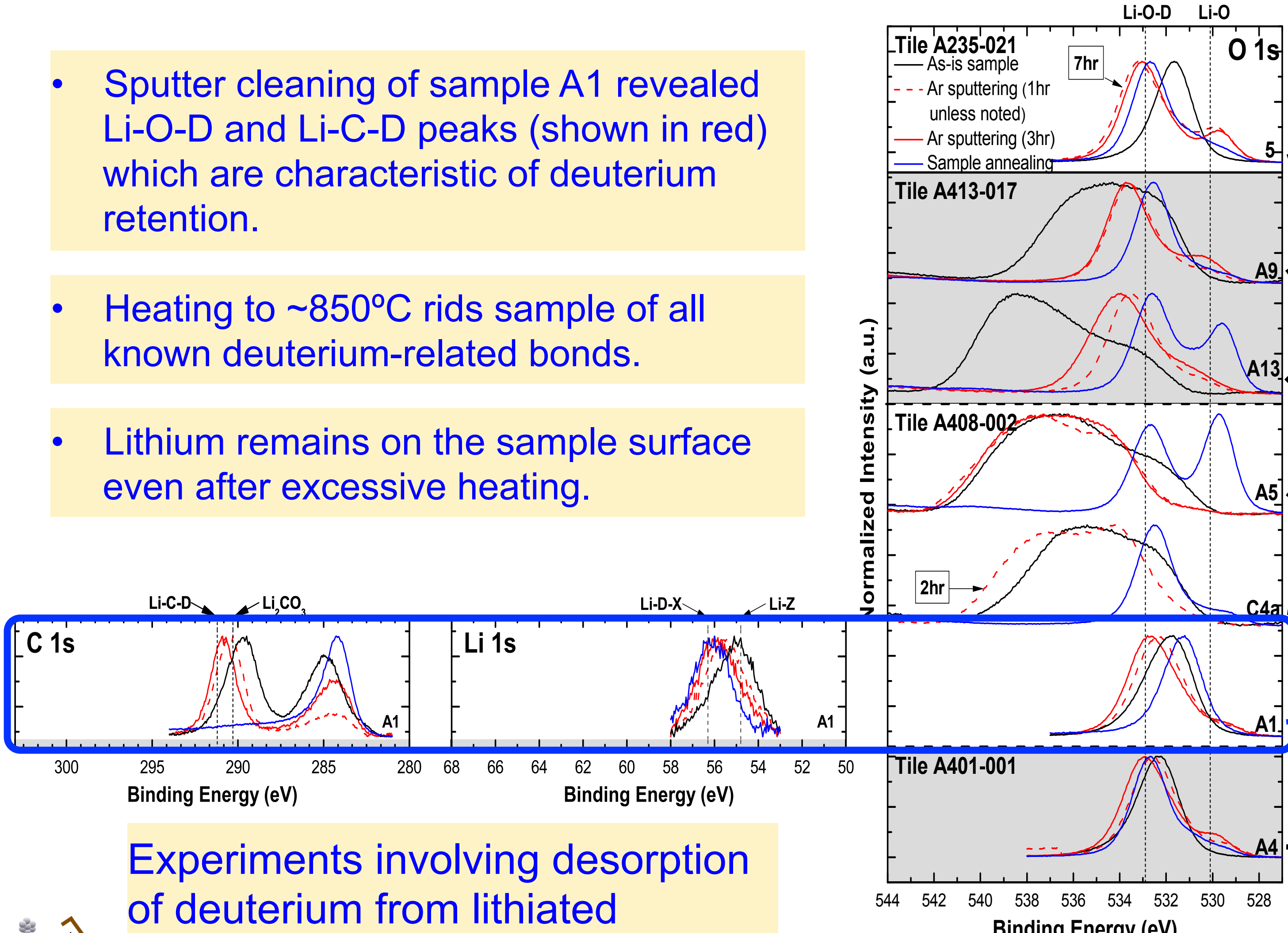
Effect of high-temperature annealing

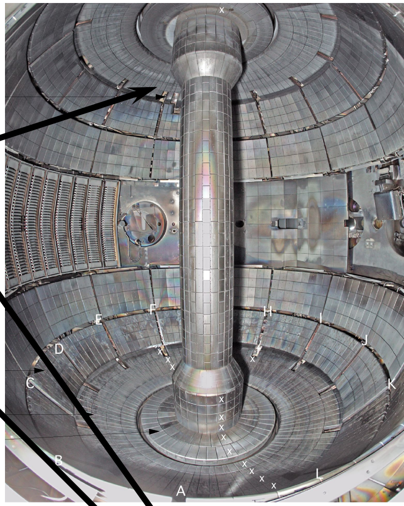
Heating to ~850ºC desorbs D-related interactions

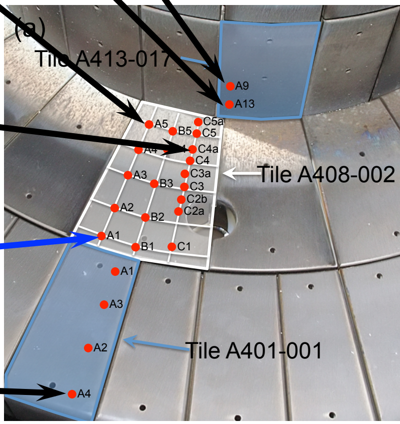
- Sputter cleaning of sample A1 revealed Li-O-D and Li-C-D peaks (shown in red) which are characteristic of deuterium retention.

- Heating to ~850ºC rids sample of all known deuterium-related bonds.

- Lithium remains on the sample surface even after excessive heating.





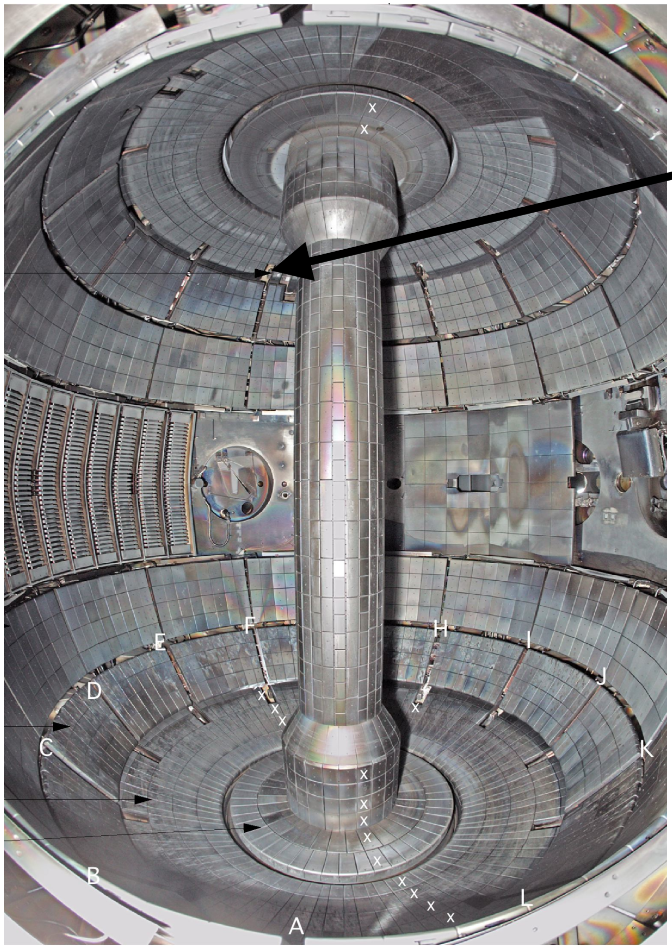


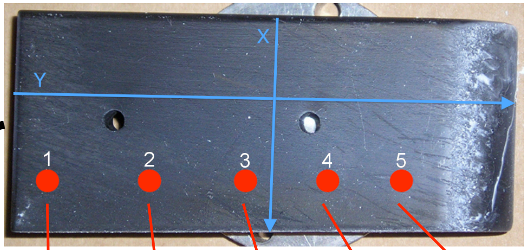
Experiments involving desorption of deuterium from lithiated graphite should heat to ~850ºC.

C.N. Taylor, et al., Nucl. Fusion, submitted 2011.

Lithium ‘thickness’ dependence

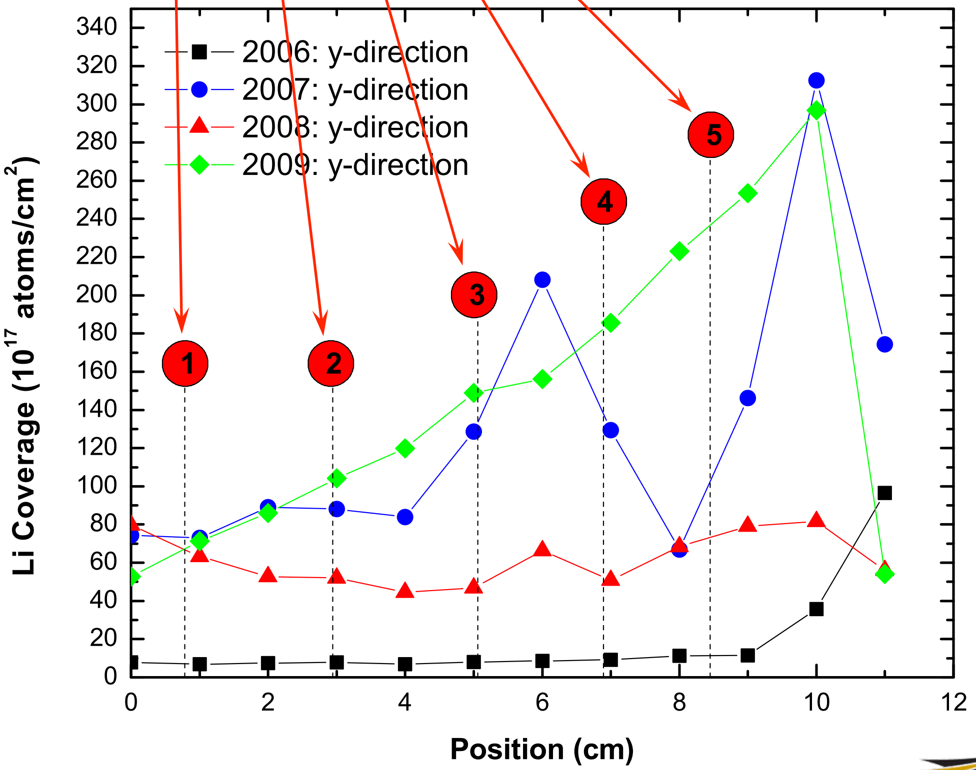
Upper vessel tile has large lithium coverage gradient





Nuclear reaction ion-beam analysis

Probing depth:
~15 μm for Li
~7 μm for D

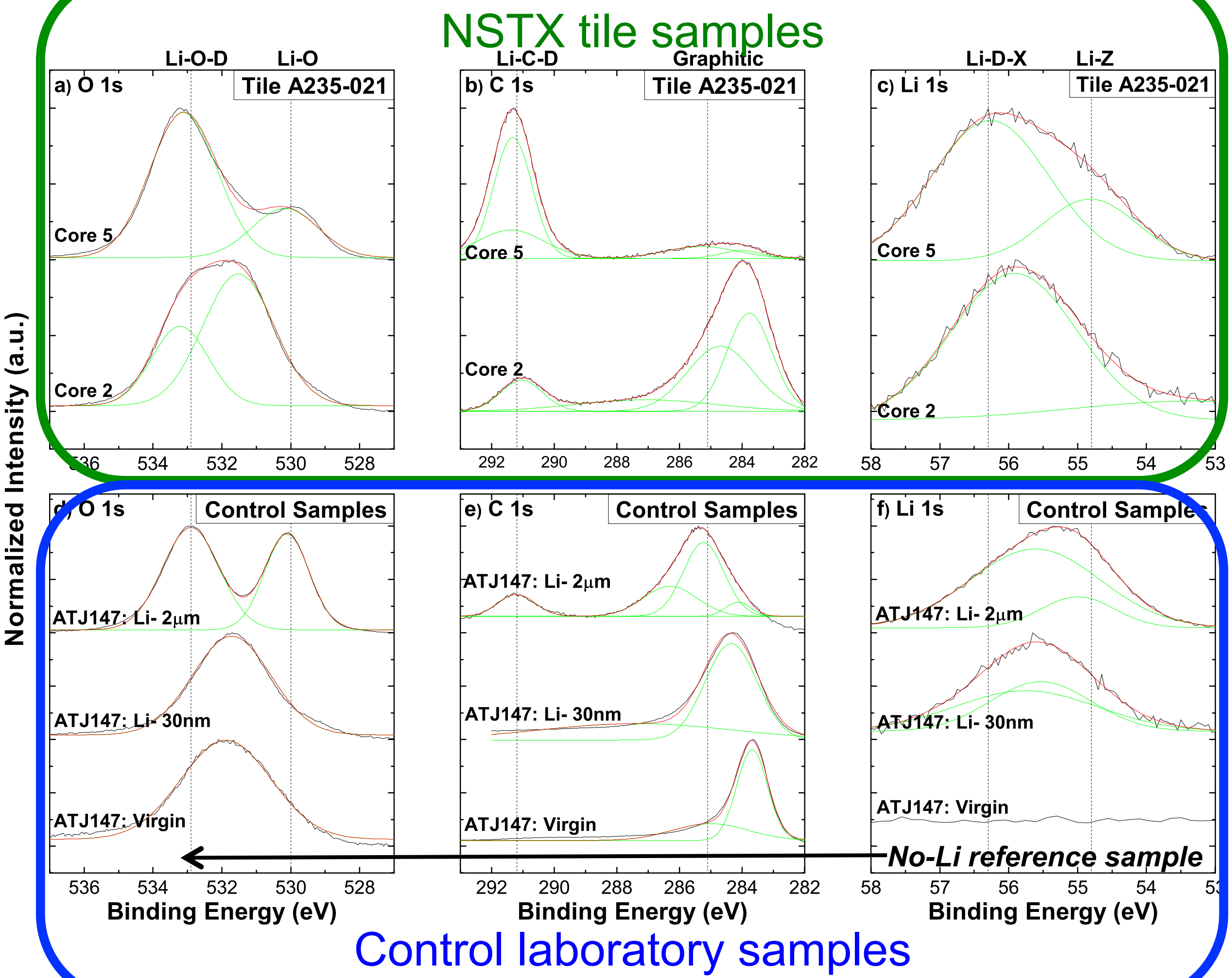


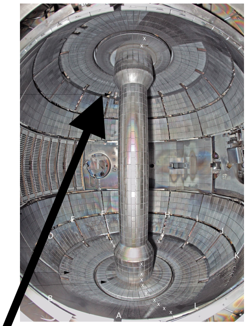
C.N. Taylor, et al., Nucl. Fusion, submitted 2011.

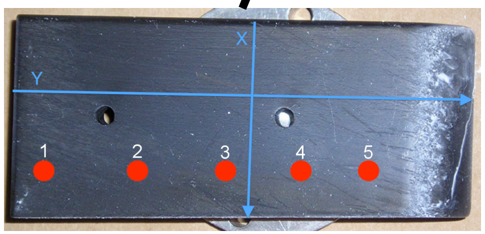
W. Wampler, unpublished data, 2009

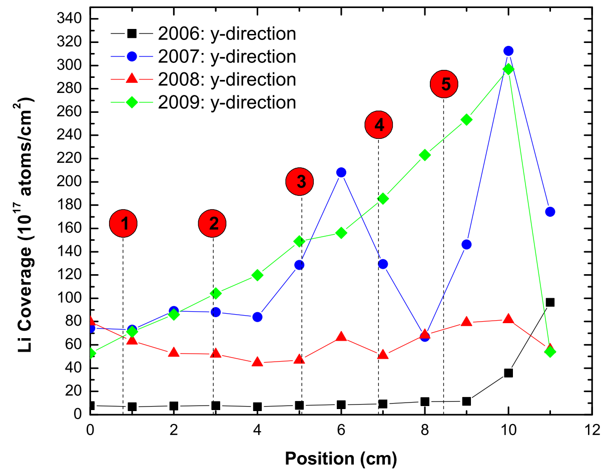
Lithium ‘thickness’ dependence

Control experiments find minimum Li threshold









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Lithium 'thickness' dependence

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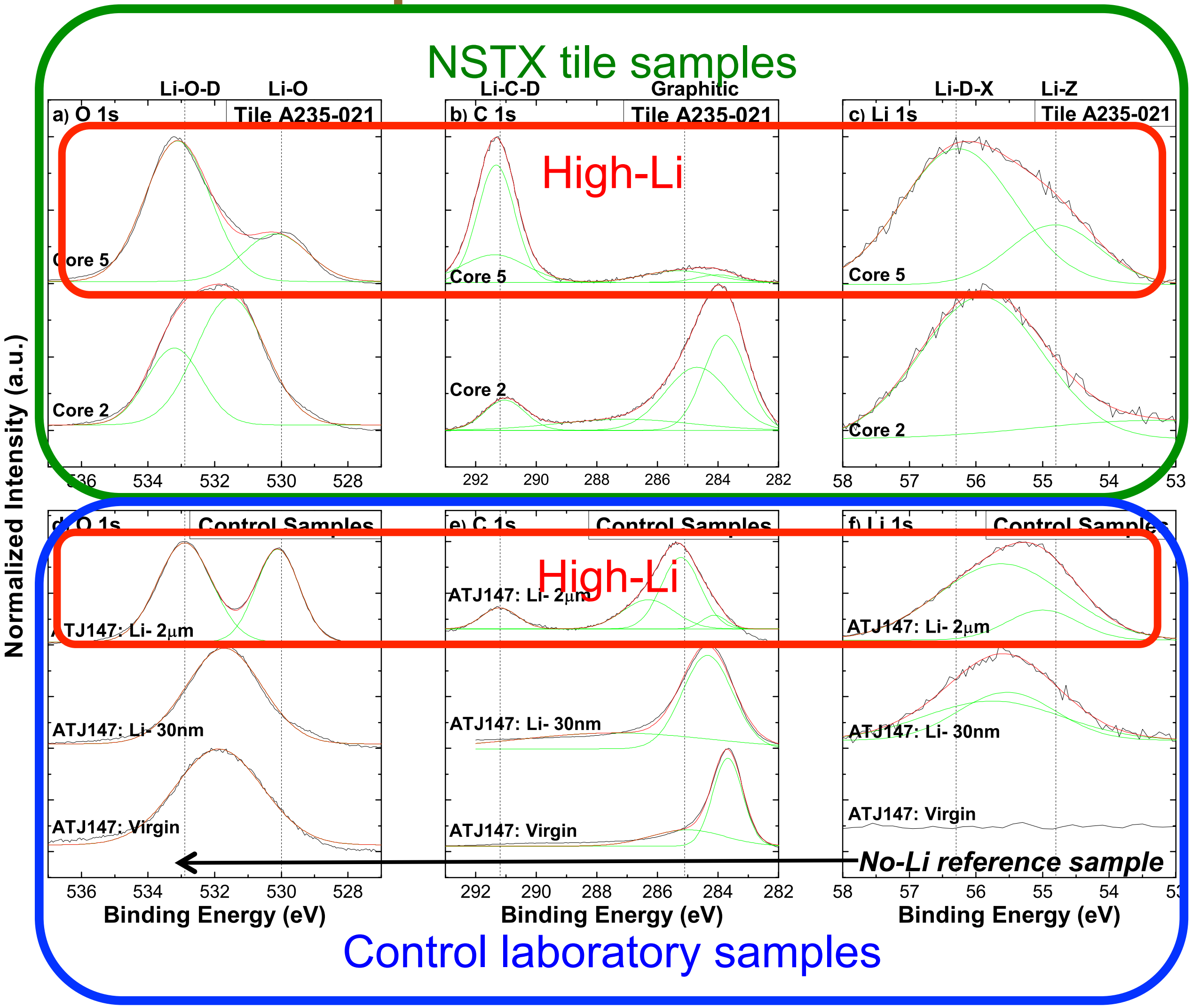

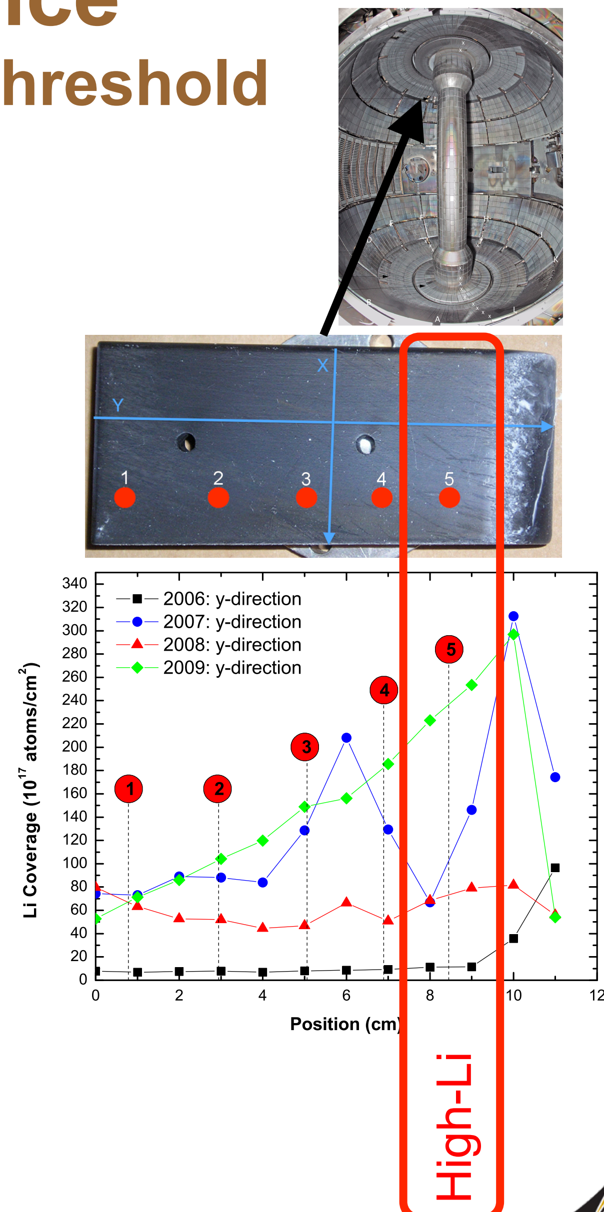

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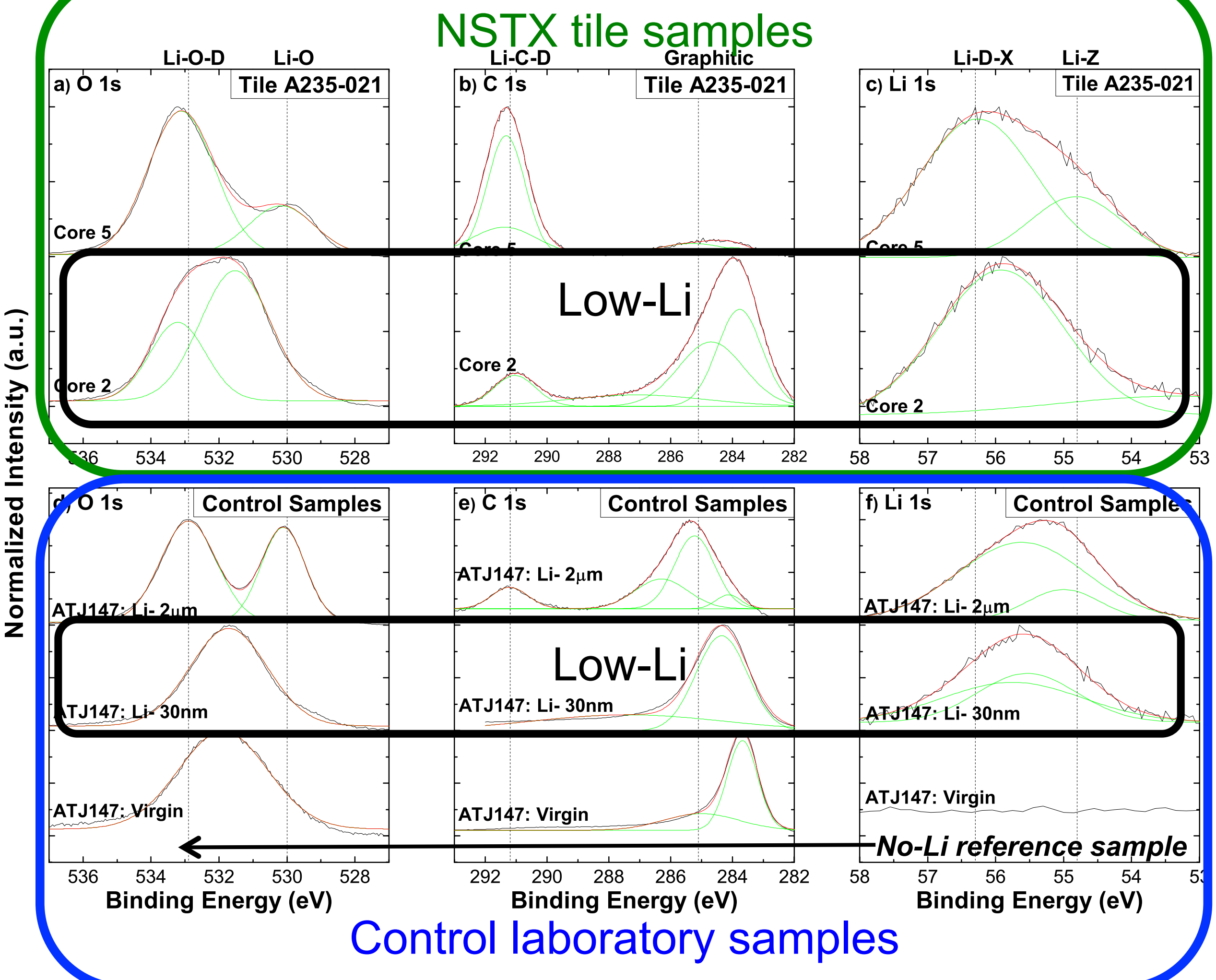

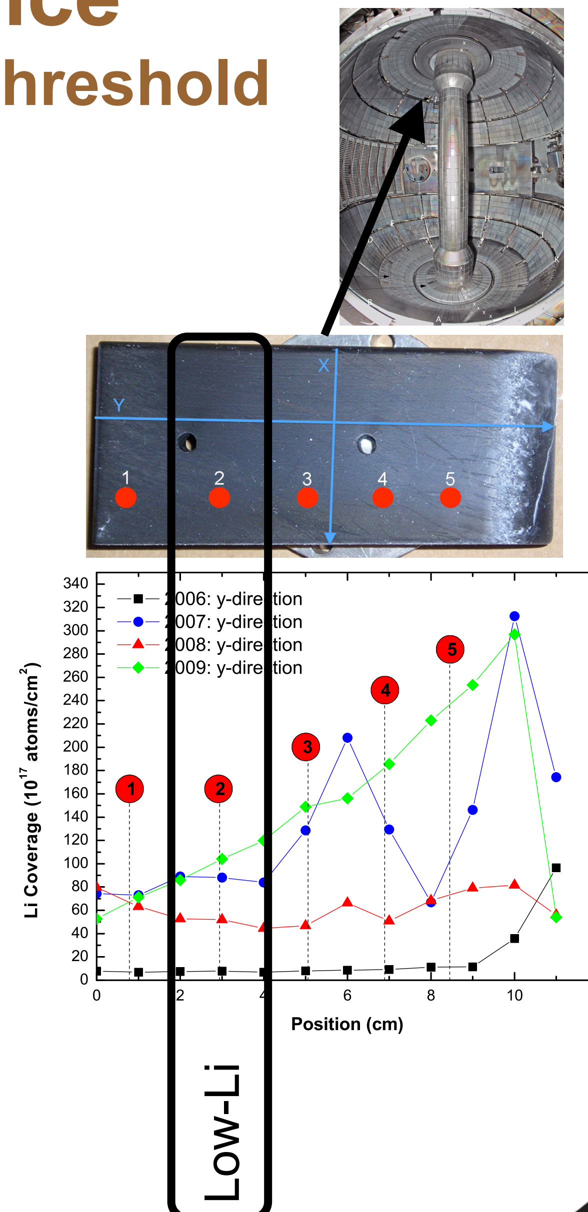

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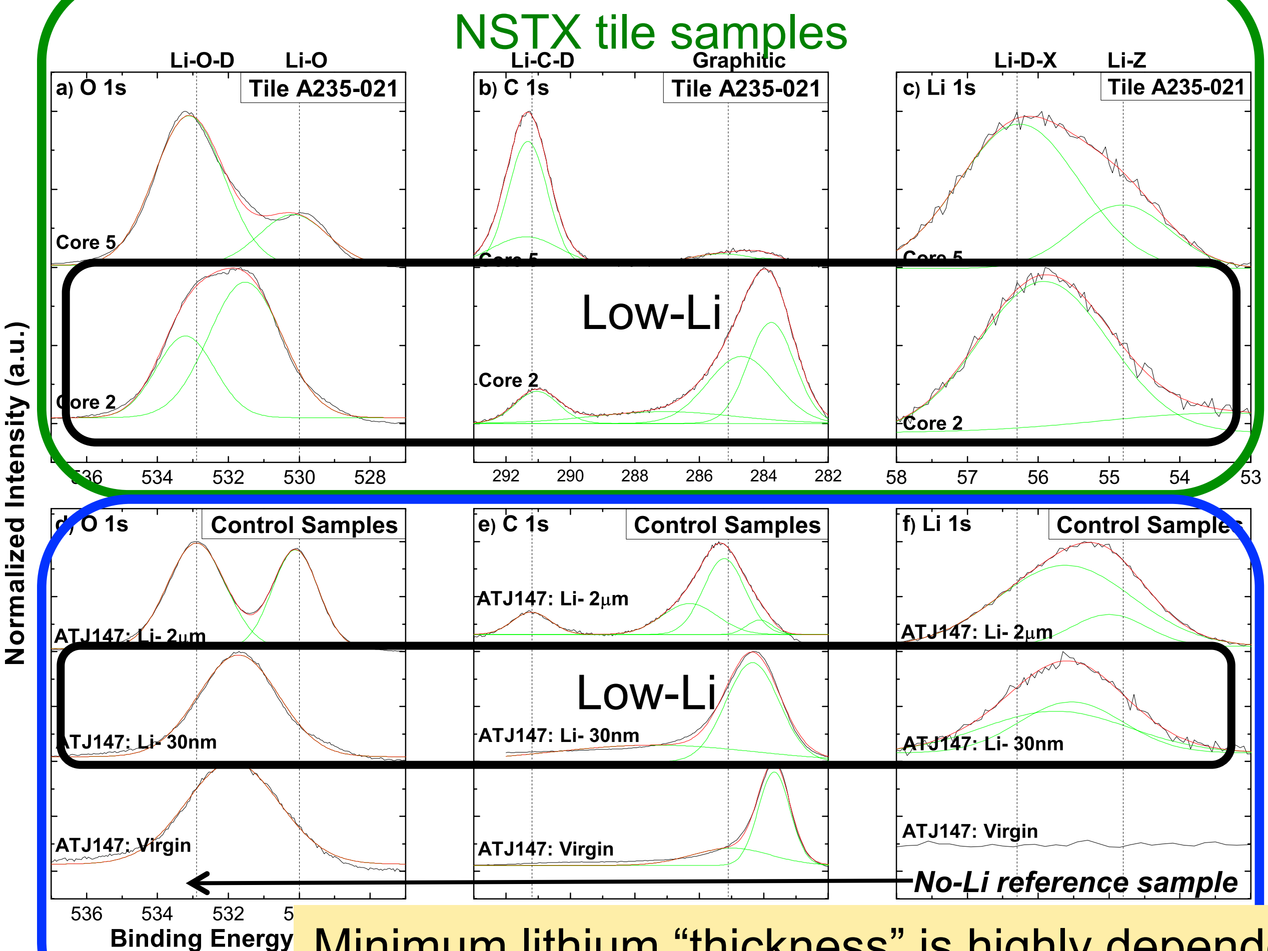
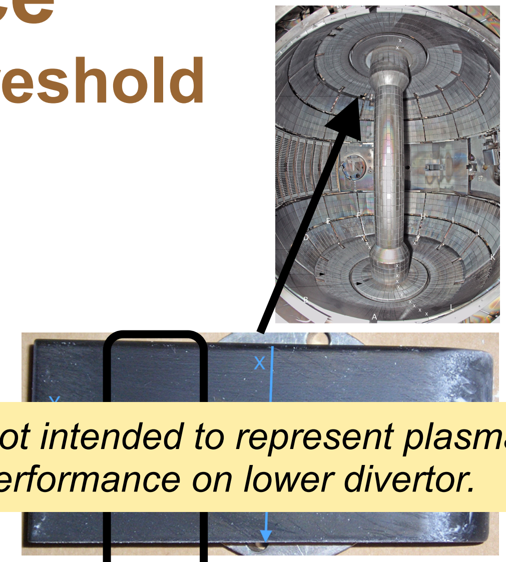
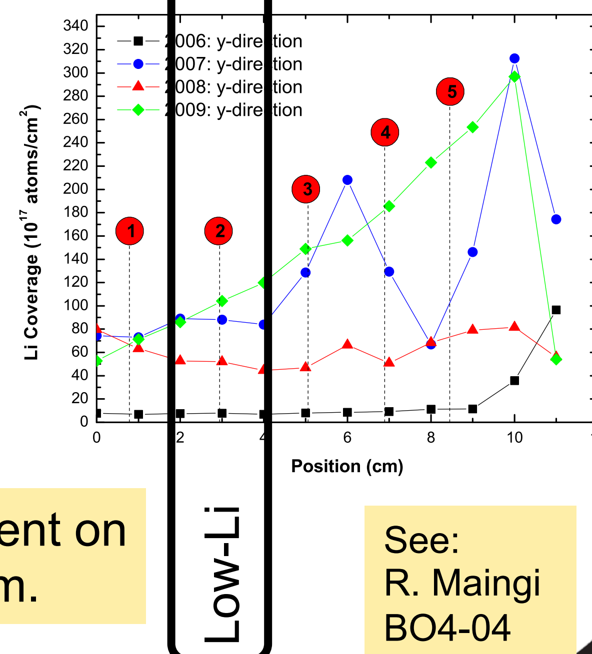




Not intended to represent plasma performance on lower divertor.



Minimum lithium “thickness” is highly dependent on morphology and on range between 50-500 nm.

See:
R. Maingi
BO4-04

C.N. Taylor, et al., Nucl. Fusion, submitted 2011.

Conclusions

- Procedures for recovery of pre-passivated surface show that Ar sputter cleaning and heating is capable of uncovering peaks associated with deuterium retention.
  - To avoid excessive difficulty in resuming plasma operations after a mid-campaign vent (where mild passivation may occur), perform glow discharge *and* heating.

- Regions closest to lower divertor corner have unused deuterium pumping capability remaining while other locations are saturated with deuterium and become deuterium recycling sources.
  - Lithium conditioning should focus on high recycling regions.

- Excessive heating (~850ºC) effectively depletes sample of deuterium related interactions observed in O 1s and C 1s regions.
  - Thermal desorption experiments with deuterated Li-graphite should heat to ~850ºC to ensure thorough desorption.

- A minimum lithium threshold is *highly sensitive to surface morphology* and is found between 50-500 nm.

See also:
J.P. Allain: PI2.06
Wednesday, 4:30 pm