Effects of lithium on chemical sputtering, physical sputtering, and deuterium pumping on graphitic substrates

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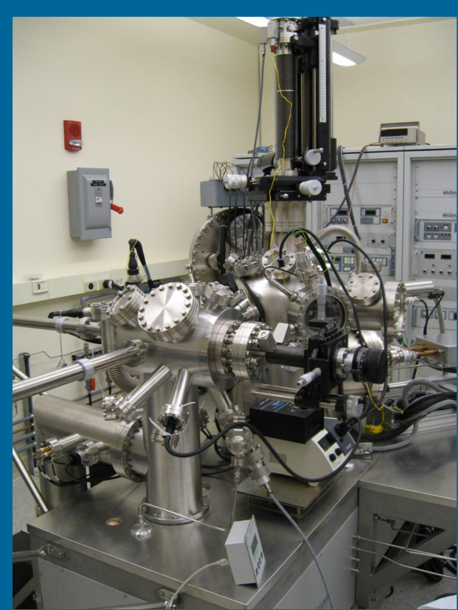
Abstract

Lithium research has been conducted in various tokamak devices such as TFTR, CDX-U, FTU, T-11M and NSTX, as a means of enhancing plasma performance. Lithium has been found to reduce hydrogen recycling and facilitate H-mode power threshold. No systematic data is available on D retention and surface chemistry properties of lithiated graphite structures. The presence of lithium in graphite reduces erosion (either physical or chemical sputtering) by factors between 10-30. For pure lithium surfaces D retention can reach levels of 1:1 and the mechanism consists of a combination of D retention in solution with Li (liquid state) and precipitation into Li-D solid crystallites based on the solubility limit of D in Li. For the case of lithiated graphite and its effect on D retention and subsequent sputtering by the plasma the mechanisms are more complex. The chemical state of Li, C and O become critical to understand the role Li plays in pumping D. This work therefore includes well-diagnosed in-situ studies of lithiated graphite exposed to controlled conditions and compared to post-exposure samples from NSTX.

Surface Characterization Methods

The Omicron system is located at Birck Nanotechnology Center at Purdue University. This system is specifically for surface analysis and uses a variety of methods to determine the surface chemistry and physical properties of sample substrates. Characterization techniques include quadrupole mass spectroscopy (QMS), thermal desorption spectroscopy (TDS), X-ray photoelectron spectroscopy (XPS), high resolution electron energy-loss spectroscopy (HR-EELS), and contains in-situ Scan Tunneling Microscopy (STM). In addition, Omicron employs several methods for surface modification including evaporation sources, ion guns, and x-ray sources.



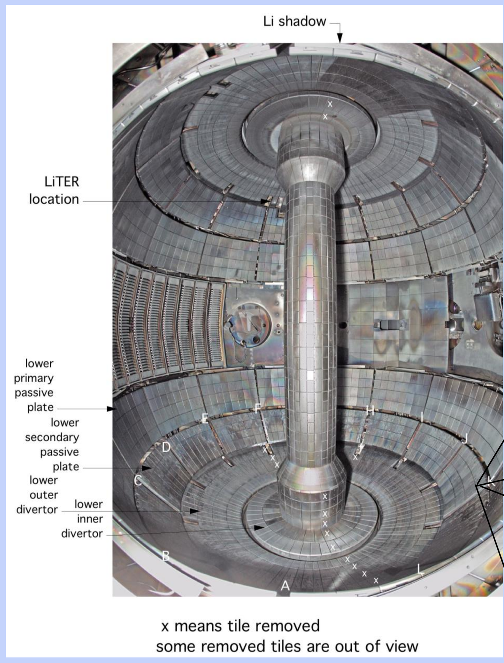
Omicron surface characterization Cluster (Birck Nanotechnology Center)

METHODS

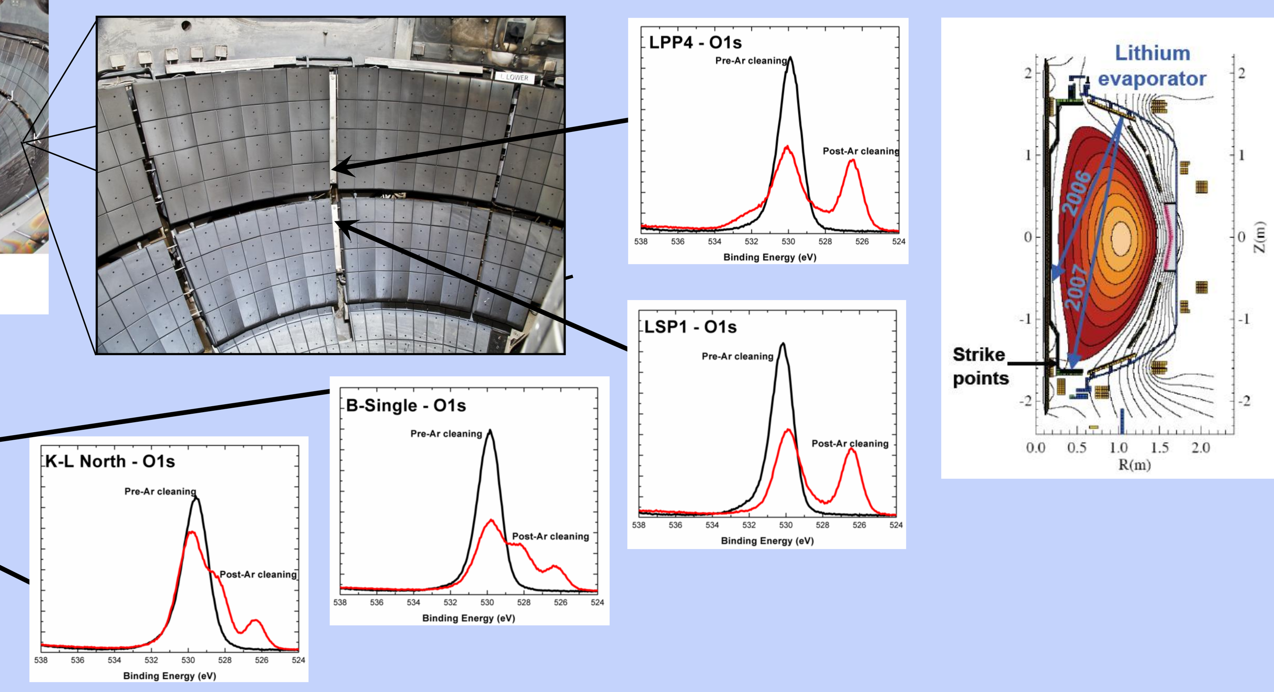
- XPS scans are performed on each sample before surface modification, after surface modification, and after any process (ie. TDS) (dwell=0.8ms, step=0.05eV)
- Lithium is deposited via a lithium evaporator. Typically, 2000 nm are deposited.
- Samples are bombarded (irradiated) with 1 keV (500 ev/amu) deuterium, 25 min.
- Samples are heated for TDS (570 °C for 20 minutes
- NSTX tiles are bombarded (irradiated) with Ar^+ to clean the passivated surface.

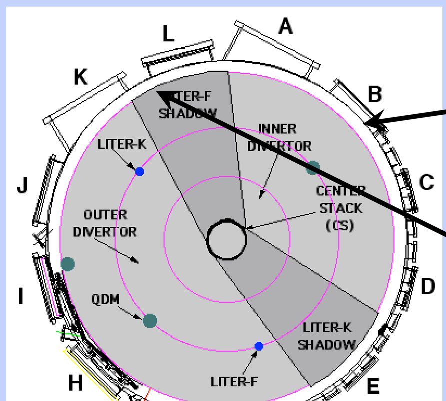
NSTX PFCs post-exposure analysis

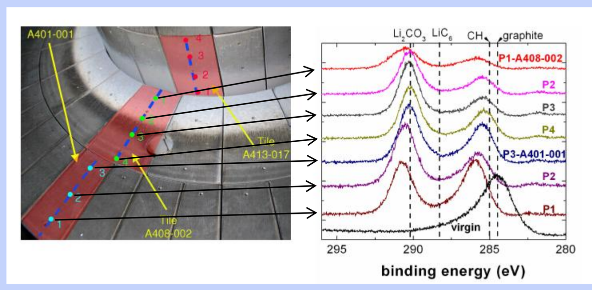
Sample locations in NSTX

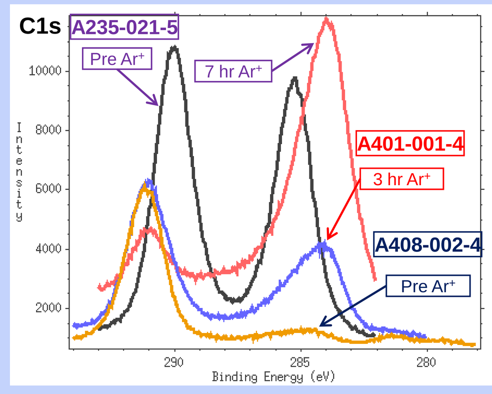


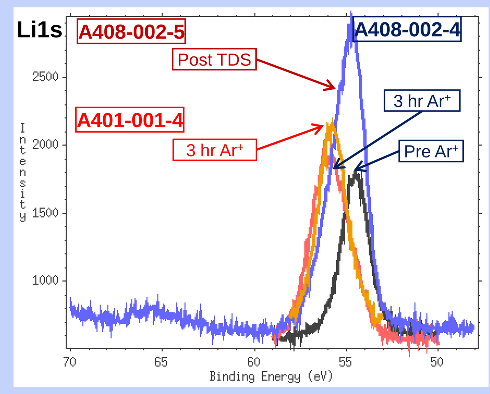
PPPL removed 23 Si witness samples (ws) from NSTX. After the samples were exposed to atmospheric conditions, a passive oxidized layer began to form on the surface. In addition, three tiles were cored yielding about 5 samples per tile. Analysis of these samples at Purdue University indicated the need of conducting control experiments with *in-situ* analysis to avoid ambient air exposure effects. These control experiments will provide complementary data to post-exposure analysis at Purdue.





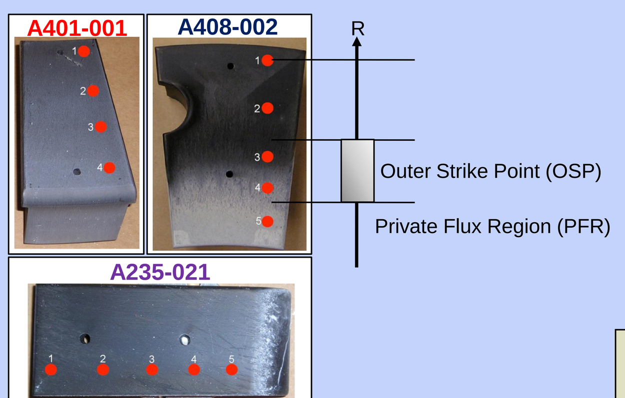


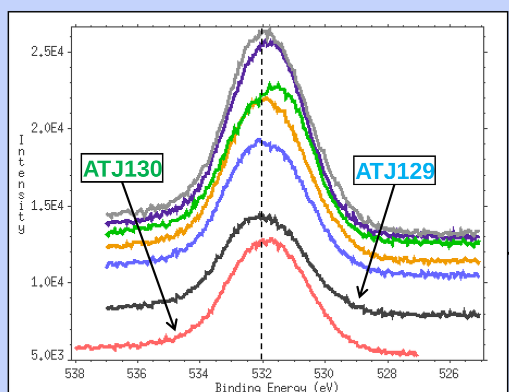




Lithium Surface Chemistry

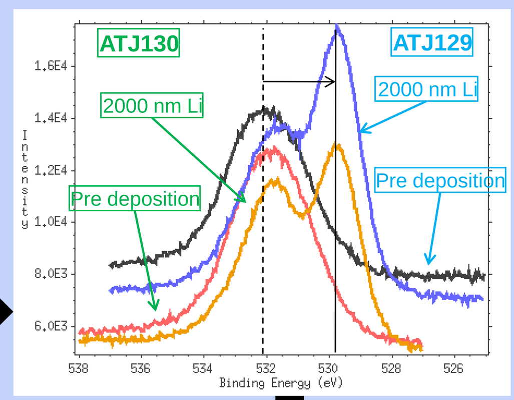
Lithiated control samples are compared to the NSTX tiles to determine the mechanism by which lithium pumps deuterium. Experiments show a strong correlation between controlled lab experiments and NSTX tile cores.





Control Samples

Peaks shown are: **O1s**

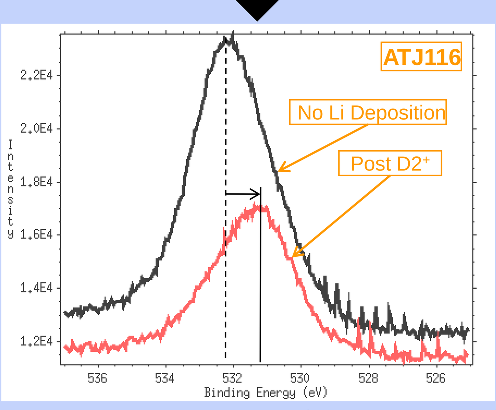


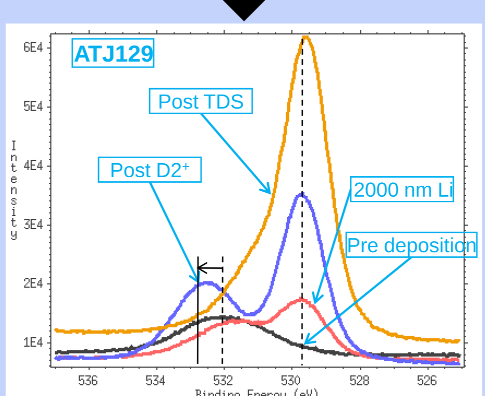
Lithium Deposition — NO / YES

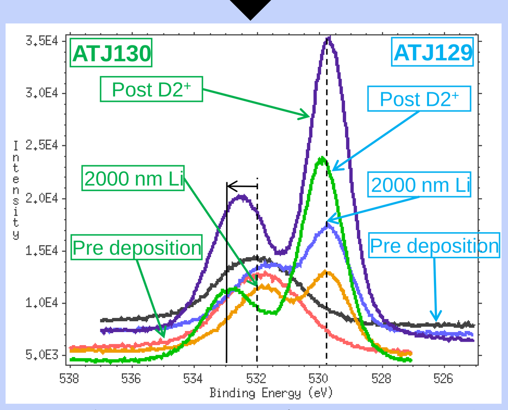
$D2^+$ Bombardment — YES

Plus TDS

$D2^+$ Bombardment — YES







Comparing control ATJ graphite samples to NSTX tile cores shows correlation in O1s

ATJ Reference Tiles

532 eV
- The O1s peak is located at 532 eV.

529.5 eV
- Lithium on graphite induces a second O1s peak at 529.5 eV.
- Bombarding ($D2^+$) a lithiated graphite sample *does not* shift the 529.5 eV peak.

529.5 eV = Li + O interaction only

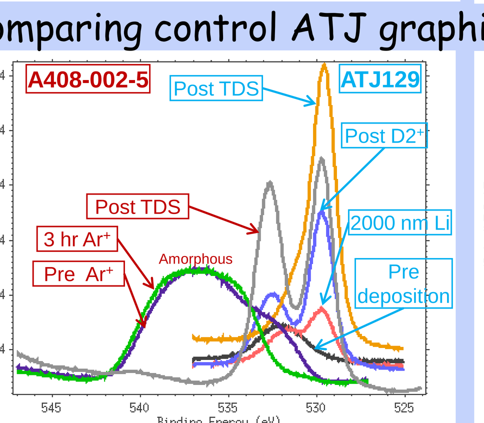
533 eV
- Bombarding ($D2^+$) a *lithiated* graphite sample shifts the O1s peak to 533 eV.
- Bombarding ($D2^+$) a plain graphite sample (no Li) *does not* shift to 533 eV (shifts the O1s peak to 531 eV).

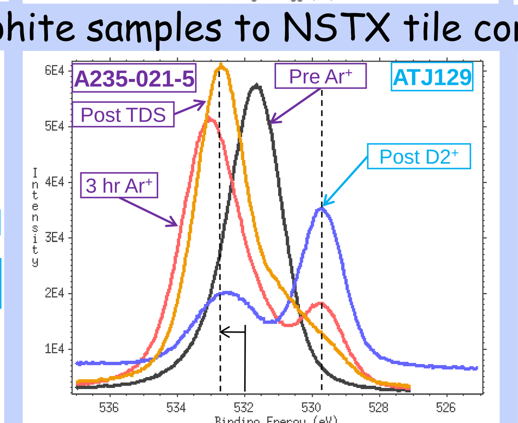
533 eV = Li + O + D interaction, exclusively

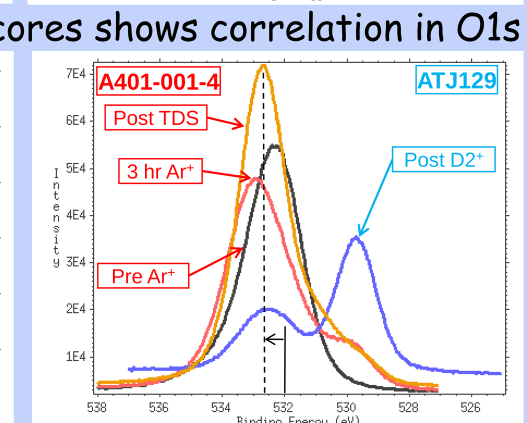
NSTX Tiles

- NSTX tiles have been exposed to air, which induces a passivated layer to form.
- Tiles cleaned 3-7 hours with Ar ion gun.
- Tiles heated for thermal desorption spectroscopy (TDS).

- After TDS, two peaks are seen:
  - **529.5 eV = Li + O interaction**
  - **533 eV = Li + O + D interaction**







- It can be seen that the 529.5 eV peak for A235-002-5 is significantly smaller than A408-002-5.
- This corresponds directly to the amount of lithium per tile since the 529.5 eV peak is induced by a Li + O interaction.
- A408-002-5 Li coverage = 120 E17 Li/cm²
- A235-002-5 Li coverage = 35 E17 Li/cm²

- A408-002-5 lies closer to the center stack than A408-002-4
- A408-002-5 lies within the private flux region (PFR) = amorphous surface
- A408-002-4 is exposed to the outer strike point (OSP) = structured surface

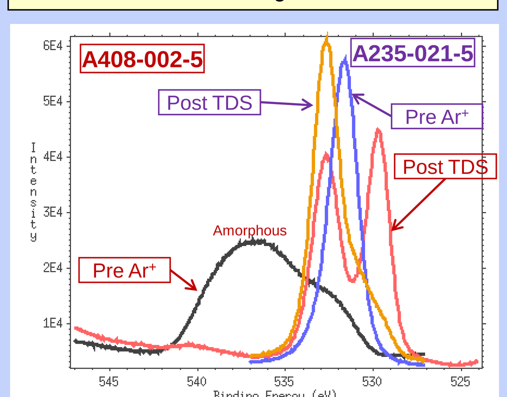
- C1s spectra from reference sample ATJ 129 shows distinct absence of a carbonate peak at binding energies above 290 eV.
- In-situ laboratory experiments have shown that large amounts of water and/or oxygen will induce carbonate peak.

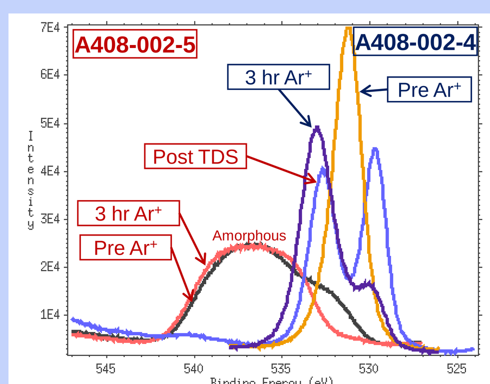
D Coverage[1]
(E17 D atoms/cm²)

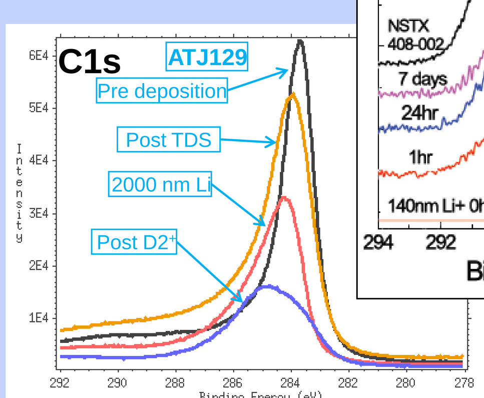
Tile /Position	1	2	3	4	5
A-235-021	5	4	4	5	5
A-408-002	45	50	90	100	90
A-401-001	25	40	25	15	-

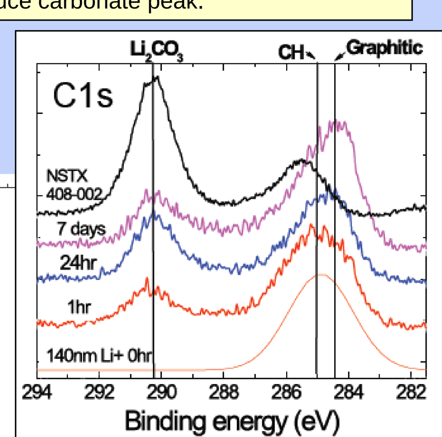
Li Coverage[2]
(E17 Li atoms/cm²)

Tile /Position	1	2	3	4	5
A-235-021	10	10	10	10	35
A-408-002	60	100	100	110	120
A-401-001	50	65	55	40	-









Discussion and Conclusions

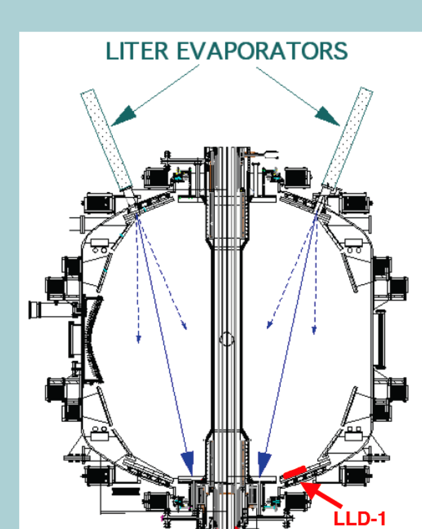
- XPS Analysis of NSTX tiles and controlled reference samples from laboratory experiments show discernable changes in surface chemistry induced by presence of lithium.
- From the reference cases the peak corresponding to 529.5 eV ± 0.8 eV is independent of $D2^+$ exposure and heat. This peak therefore consists strictly of a Li and oxygen interaction (binding).
- $D2^+$ bombardment in the lab experiments lead to similar surface chemical state compared to NSTX tile surfaces (after Ar cleaning) corresponding to a peak at 533 eV. Therefore this peaks consists strictly of a D interaction with lithiated graphite.
- Post-exposure NSTX tile core samples show a complex chemistry dependent on location along the divertor in NSTX (e.g. amorphous state in PFR sample).
- Results also provide a means to conduct well-diagnosed experiments under *similar* surface conditions as those found after multiple exposure campaigns in NSTX. A new in-situ probe in NSTX will help correlate effect of specific shots with surface chemical state and couple to lab experiments.

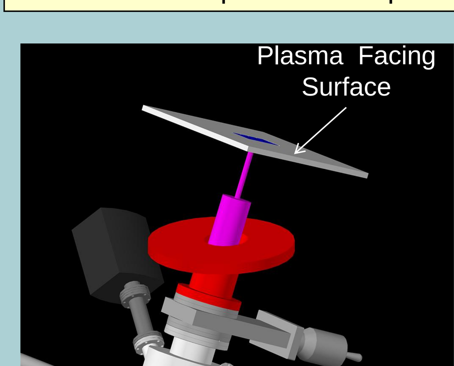
Future Work

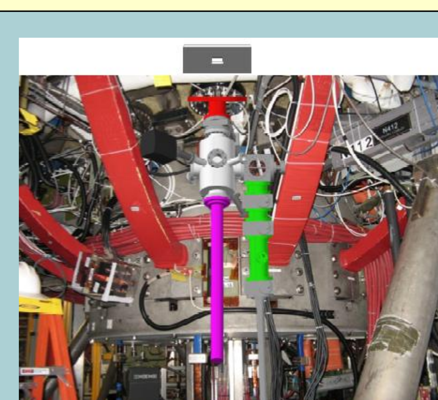
- As detailed analysis has indicated laboratory experiments can help elucidate on some underlying mechanisms for lithium graphite interaction with hydrogen isotopes.
- However, correlation to specific NSTX shots must be done with surface analysis in-situ *in addition to* post-exposure measurements in lab experiments at Purdue
- This effort has motivated the use of an in-situ surface analysis probe integrated near the inner divertor of NSTX to work in close proximity with the LLD

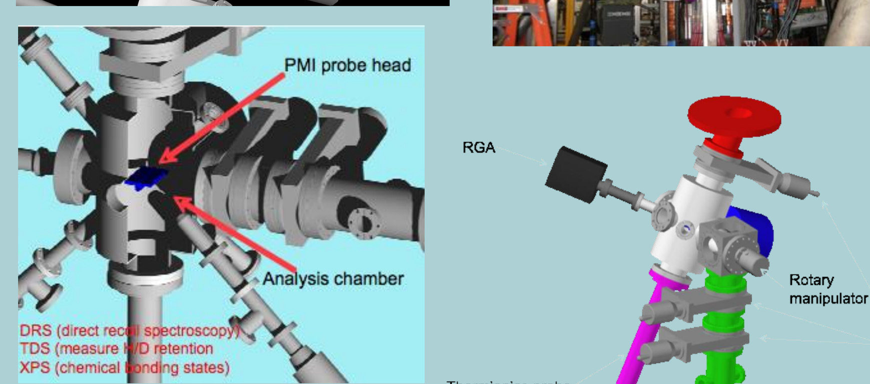
NSTX Surface Analysis Probe

The primary purpose of a surface analysis probe is to perform an *in-situ* analysis of plasma facing surfaces to determine the role of lithium and the mechanism by which lithium pumps deuterium. Samples attached to a witness plate will be brought to the plasma facing surface to be exposed for a specified number of shots.









The surface analysis probe will employ a simple design to insert a sample witness plate to the plasma facing surface. Subsequent versions of the surface analysis probe will include additional analysis equipment including x-ray photoelectron spectroscopy (XPS) and thermal desorption spectroscopy (TDS).

Acknowledgements

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[1] W.L. Wampler, "Ion Beam Analysis of Lithium on Tiles from NSTX" SNL, 2007.
[2] W.L. Wampler, "Ion Beam Analysis of Deuterium on Tiles from NSTX" SNL, 2007.

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