NSTX Plasma-Material Interface (PMI) Probe and supporting experiments

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Outline: NSTX PMI Probe

- Summary of Li-based PMI work at Purdue
- Post-mortem tiles vs controlled laboratory experiments
- Chemistry of NSTX tile surfaces
- NSTX PMI Probe design
- Joule Milestone experiments
- Future *in-situ* PMI probe design: MAPP
- Support of LLD experiments
Summary of Li-based PMI work at Purdue

- At Purdue we’re investigating the role lithium coatings on ATJ graphite has on deuterium pumping and recycling of hydrogen
- We systematically study lithiated graphite surface chemistry and ion-induced desorption to elucidate plasma-material interface interactions in NSTX
- Lab experiments also look at the effect of a lithiated graphite environment on the performance of NSTX plasma with the liquid lithium divertor (LLD)
Lithiated graphite work at Purdue

- Post-mortem analysis of 2008 NSTX campaign tiles
  - Along inner divertor floor and bottom of center stack tiles
  - Tiles near LiTER port
  - Also examined Si witness samples (retrieved from various locations in NSTX)
- Controlled *in-situ* lithiated graphite studies
  - Correlation of D irradiations with graphite tiles
  - Mechanisms for D retention as function of D flux and Li dose
  - Mechanisms for surface passivation on Li-C
  - Control experiments with: Si, lithium foil, SS, Mo, W, etc…
- NSTX PMI probe design and analysis
  - Probe samples: Si, ATJ graphite, Pd
  - TDS and XPS analysis
Controlled in-situ lithium deposition on ATJ graphite followed by air exposure

- Lithium deposition yields peak at 529.5 eV ± 0.5 eV
- Exposure to air yields peak at 290 eV and 529 eV peak disappears
Control group with “pure lithium” target

- Pure ‘as received’ Li is used and transferred to the chamber in an Ar environment.
- Strong sample charging effects observed, graphitic bond at 284.5 eV is used for calibration.
- O1s peak appears at 531.5 eV which corresponds to \( \text{Li}_2\text{CO}_3 \) and/or \( \text{LiOH} \).
- \( \text{Li}_2\text{CO}_3 \) peak at 290.2 eV is found to be weak in the XPS spectra of C1s.
- After \( \text{Ne}^+ \) etching a strong O1s peak appeared at 528.5 eV and assigned to \( \text{Li}_2\text{O} \).
- The peroxide (529eV ± 0.5 eV) functionality is not observed on Li metal.

From “a” to “c” removal of surface oxide layer

S.S. Harilal and J.P. Allain, Appl. Surface Sci. in press 2009
**D irradiation yields additional peak shifts in O1s and C1s indicating additional functionalities**

On a ATJ graphite substrate…

### 532 eV ± 0.25 eV
- The O1s peak is located at 532 eV (C-O bonds)

### 529.5 eV ± 0.5 eV
- Lithium on graphite induces a second O1s peak at 529.5 eV.
- Bombarding (D2⁺) a lithiated graphite sample shifts the 529.5 eV peak slightly (<0.25 eV).

**529.5 eV = Li + O interaction only**

### 533 eV ± 0.25 eV
- Bombarding (D2⁺) a *lithiated* graphite sample shifts the O1s peak to 533 eV.
- Bombarding (D2⁺) a plain graphite sample (with no Li) *does not* shift to 533 eV (shifts the O1s peak to 531 eV).

**533 eV = Li + O + D interaction, exclusively**

Conclusion of experiments: We have established a methodology where to identify the D, O, Li interactions on NSTX tiles and link to controlled *in-situ* off-line experiments at Purdue

See tomorrow’s talk by C. Taylor for details
Exposure to air compromises study of the Li-O-D-C system

Exposure to air completely removes any information of Li-O-D functionality in ATJ graphite
“Current” qualitative hypothesis of functionality states of lithiated-graphite surfaces in NSTX

Processes

1) Fresh ATJ sample
2) Post Li deposition
3) Post D bombardment
4) Post air exposure

Penetration depth of D atoms

Legend of functionality states

1. Most dominant interaction
2. Less dominant interaction
3. Least dominant interaction

Most dominant interaction

Li-D-O dominant functionality

Li-O\textsubscript{x} dominant functionality

Bulk graphite (C-O)

O\textsubscript{1}s

532 eV

C-O

531.5 eV

Li-O-D-C

533 eV

Li-O-D

529.5 eV

Li\textsubscript{x}-O\textsubscript{1-x}

529.5 eV

Li\textsubscript{x}-O\textsubscript{1-x}

532 eV

Li-C-O

532 eV

Li\textsubscript{x}-O\textsubscript{1-x}

533 eV

Li-C-O

529.5 eV

Li-O-D-C

532 eV

Li-O-D

532 eV

Li\textsubscript{x}-O\textsubscript{1-x}

531.5 eV

Li-C-O

531.5 eV

Li-O-D-C

531.5 eV

Li-O-D

529.5 eV

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Li-C-O

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Li-O-D-C

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Li-O-D

533 eV

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Li-C-O

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Li-O-D-C

533 eV

Li-O-D
PMI Probe and Joule Milestone Experiments

- Density control via Li pumping of D is goal of multi-year Li program on NSTX.
- Retention is FY09 Joule Milestone.

Four thrusts on NSTX:
1. Particle balance of ohmic/RF shots (Static: valves closed, no pumping)
   a) Before and with Li, LLD.
   b) Time dependent data (sec - hours)
   c) Measure D retained / D ion flux
   d) Quartz Microbalance data.
2. Particle balance of NBI shots (dynamic)
3. Surface analysis of ‘fresh’ samples using sample probe (Purdue collab.).
4. Modeling, including WallPSI code of Pigarov and REDEP by Jeff Brooks.

Expose 4 samples for ~ 5 ohmic, then 5 NBI shots. Withdraw same evening. Retrieve under argon. Ship to Purdue for analysis using PHRISM, Omicron and KRATOS. Piggy-back commissioning when opportunities available.

Charles Skinner (PPPL), Rajesh Maingi (ORNL), Vlad Soukhanovskii (LLNL), JP Allain, (Purdue U) Chase Taylor (Purdue U) and NSTX team.
**NSTX PMI Probe Design**

**Design Goals**

- Simultaneously expose 4 each, 12.5 mm diameter samples (1-3 mm thick) to NSTX plasma.

- After exposure, retract the samples into the test station where they will be individually heated to 1000 °C and perform thermal desorption spectroscopy. Initially, only one sample will be heated.

- While heating one sample, adjacent samples must be kept at ~100 °C.

- Samples must be heated to 1000 °C and cooled to <200°C between plasma discharges before being reinserted into a subsequent discharge. (not required for initial operation).

- Initially, only one sample will be heated and the others will be sent to Purdue. The design must provide for provisions to heat all samples.

- When heating, simultaneously provide temperature measurement of top surface of all 4 samples along with temperature measurement of energized heater.

- Probe head carrying all samples must fit through 2.5” diameter aperture.

- Some samples are to be shipped to Purdue for further surface analysis without heating, so probe head must be removable from probe shaft to retrieve samples.

L. Roquemore, M. Denault, T. Provost
Summary of PMI Probe experiments

With no lithium conditioning

Neutral Beam Plasmas
- ATJ132 – TDS at NSTX
- ATJ133 – TDS at Purdue
- Pd425 – XPS
- Si105

Ohmic Heated Plasmas
- ATJ134 – TDS at NSTX
- ATJ135 – TDS at Purdue
- Rh sample
- Si112

With lithium conditioning

Neutral Beam Plasmas
- ATJ138 – TDS at NSTX
- ATJ139 – TDS at Purdue
- Pd431 – XPS
- Si109

Ohmic Heated Plasmas
- ATJ136 – TDS at Purdue
- ATJ137 – TDS at Purdue
- Pd422 – XPS
- Si108
**ATJ133 – Exposed to NB Plasma**

**Brief DoE**
- No lithium conditioning
- 6 NSTX NB plasma shots
- Shipped to Purdue under Ar
- TDS at Purdue

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**Graphs**

- **O1s**
  - 1. After NB Plasma
  - 2. Post TDS (Purdue)

- **C1s**

- **Li1s**
Brief DoE
- Lithium conditioning
- 6 NSTX NB plasma shots
- Ar cleaning
- TDS performed at Purdue
- XPS at Purdue

Note: 30 min Ar cleaning XPS scan was also taken
**AJT139 vs. post-mortem tile A235-002-2**

**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?
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
Lithium dose affects Li-D-O-C functionality

**O1s**

1) Post 30nm Li deposition
2) Post D irradiation

**O1s**

1) Post 2knm Li deposition
2) Post D irradiation

Li-30nm post deposition, post D irradiation

Li-2000 nm post deposition, post D irradiation
NSTX PMI Probe location and lithium deposition

Simulation by Leonid Zakharov
Current PMI Probe Analysis: TDS spectra

ATJ Probe Samples: HDO – 19 amu

- TDS spectra from both in-vacuo system at NSTX and at Purdue. Current analysis includes: accounting for different pumping speeds, spectra fragmentation
- Temperature ramps are slightly different at NSTX and Purdue, this is also being analyzed.
Summary and Future Work

- Controlled particle-surface interaction experiments at Purdue show importance of \textit{in-situ, in-vacuo} characterization to study the role of Li-based systems on hydrogen retention and recycling.

- We’re finding that D retention in lithiated graphite is dictated by more than simple interactions between Li and D alone. The presence of carbon and oxygen plays a major role in dictating how D is bound in the lithiated graphite system.

- Key Results from preliminary PMI Probe data
  - Although we measure lithium on samples, not enough to presence Li-D-O functionality that are observed in post-mortem NSTX tiles and control experiments.
  - Preliminary TDS data will determine the amount of retention for this amount of Li.
  - Pd and Si samples will also shed light on amount of Li deposited on PMI probe and quantify amount of D implanted in region (cross-calibrate with nearby probes).
  - PMI Probe location will be valuable with LLD (see modeling by J. Brooks).
Future Work (partial list)

- Study effect of serial lithium depositions on Li intercalation and diffusion in ATJ graphite irradiated with D
- Study lithium surface chemistry and evolution on Li-coated porous Mo structure used in LLD
- Study low-energy He interactions with lithiated graphite surfaces (evaluating He-glow discharge exposures in NSTX)

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