The Materials Analysis Particle Probe (MAPP) Diagnostic System in NSTX

Bryan Heim, S. Gonderman, C. N Taylor, J. P. Allain, Z. C. Yang, M. Gonzalez, E. Collins, C. H. Skinner, B. Ellis, W. Blanchard, L. Roquemore, H. W. Kugel, R. Martin, and R. Kaita

Abstract*—Lithium conditioning of plasma-facing surfaces has been implemented in National Spherical Torus Experiment (NSTX) leading to improvements in plasma performance such as reduced D recycling and a reduction in edge localized modes. Analysis of postmortem tiles and offline experiments along with atomistic modeling has identified interactions between Li-O-D and Li-C-D as chemical channels for deuterium retention in ATJ graphite. However, previous surface chemistry analysis of NSTX tiles were conducted *postmortem* (i.e., after a completed annual campaign), and it was not possible to correlate the performance of particular discharges with the state of the material surface at the time. Materials Analysis Particle Probe (MAPP) is the first *in-vacuo* surface analysis diagnostic directly integrated into a tokamak and capable of chemical surface analysis of plasma facing samples retrieved from the vessel in between discharges. It uses X-ray photoelectron spectroscopy, direct recoil spectroscopy, low energy ion surface spectroscopy, and thermal desorption spectroscopy to investigate the chemical functionalities between D and lithiated graphite at both the near surface (5–10 nm) and top surface layer (0.3–0.6 nm), respectively. MAPP will correlate plasma facing component surface chemistry with plasma performance and lead the way to improved understanding of plasma-surface interactions and their effect on global plasma performance. Remote operation and data acquisition, integrated into NSTX diagnostic and interlocks, make MAPP an advanced PMI diagnostic with stringent engineering constraints.

***Index Terms*—Lithiated graphite, plasma material interactions, plasma-surface interactions.**

I. Introduction

LITHIUM conditioning of plasma-facing surfaces (PFS) has been implemented in the National Spherical Torus Experiment (NSTX) leading to improvements in plasma performance such as reduced D recycling and a reduction in edge localized modes [1]–[4]. Nuclear reaction analysis of postmortem NSTX tiles has measured the lithium and D spatial distribution in divertor tile samples [5]. X-ray photoelectron spectroscopy (XPS) of cored NSTX diverter tile samples after removal of the passivated surface by heating and Ar sputtering has revealed unique chemical functionalities between Li-O-D and Li-C-D [6], [7] Offline experiments simulating plasma material interactions by deposition of a nominal thickness of 0.5–5 μm of lithium on ATJ graphite followed by irradiation with a 1 -keV deuterium beam has revealed similar chemical functionalities of Li, O, and D. [8], [9]. A PMI probe has also been used to expose samples to NSTX plasmas and retrieve them for later analysis.

Postmortem analysis of NSTX tile and PMI probe exposed samples has revealed distinct chemical attributes of lithium and deuterium interactions; however, it cannot link the chemical functionalities to the performance of specific plasma shots. The exposure of such samples to ambient air obscures the previous surface (1–10 nm) chemistry. In-vacuo surface analysis is needed to better identify the chemical functionalities responsible for deuterium retention in PFS. In addition, integrated plasma exposures make it very difficult to discern surface chemistry correlations to specific plasma shots and core plasma behavior, which is of interest to wall conditions strategies.

The Divertor Material Evaluation Studies diagnostic system on DIII-D [10], [11] inspired Materials Analysis Particle Probe (MAPP) design and engineering, along with the success of the installed PMI probe in the 2011 NSTX campaign. [12] This probe located in the lower outer diverter, utilized an in-vacuo thermal desorption spectroscopy (TDS) measuring deuterium retention on up to four samples post exposure to around six to eight identical plasma discharges. Extraction of such samples was then taken under argon atmosphere for further analysis a Purdue University. XPS and TDS revealed correlated data between offline experiments and measured data; however, the desire to directly compare shot to shot plasma facing chemistry and plasma performance began the engineering of a new in-vacuo material analysis probe.

The MAPP is the first in-vacuo surface analysis diagnostic system directly attached to a tokamak, capable of shot-to-shot chemical surface analysis of PFS. MAPP allows the exposure of up to four samples (C, Mo, Li-C, liquid Li, etc.) to NSTX plasmas through an outboard diverter port, and rotary probe motion allows individual characterization of each sample at off-center focal point. MAPP will be capable of performing all diagnosis between plasma discharges (ca. 10 min, depending on analyzer setting and desired resolution). Since personnel are excluded from the tokamak area, full remote operation of a hemispherical electrostatic sector particle analyzer, X-ray and ion sources, mass spectrometer, gas manifold systems, power supplies, gauges, and valves is required. This paper will present the extensive engineering, constrains, and current progress of this novel in-vacuo, *in-situ* PMI diagnostic on NSTX.

Manuscript received August 15, 2011; revised December 26, 2011; accepted December 26, 2011. Date of publication February 10, 2012; date of current version March 9, 2012.

B. Heim, S. Gonderman, C. N Taylor, J. P Allain, Z. C. Yang, M. Gonzalez, and E. Collins are with the Purdue University, West Lafayette, IN 47907-2017 USA (e-mail: bheim@purdue.edu).

C. H. Skinner, B. Ellis, W. Blanchard, L. Roquemore, H. W. Kugel, R. Martin and R. Kaita are with the Princeton Plasma Physics Laboratory, Princeton, NJ 08543-0451 USA.

Color versions of one or more of the figures in this paper are available online at http://ieeexplore.ieee.org.

Digital Object Identifier 10.1109/TPS.2011.2182062

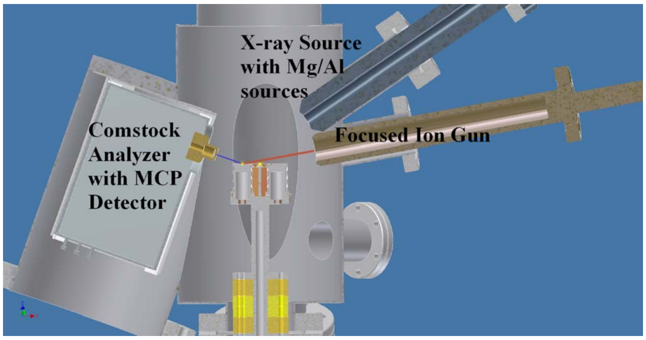

Fig. 1. Rendering of MAPP surface analysis chamber, attached to NSTX. All excitation and analysis sources are focused slightly off axis to allow characterization of each sample via rotation.

Fig. 2. Picture of MAPP analysis system attached to NSTX TIV, illustrating the tight space constraints for all equipment.

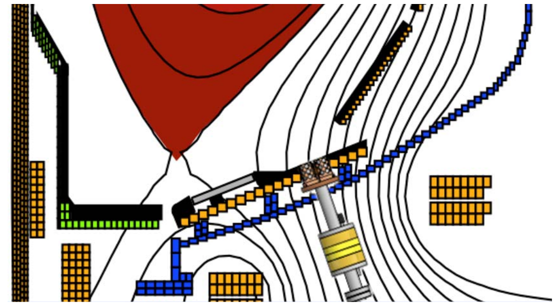

Fig. 3. MAPP head location inserted flush with outer divertor.

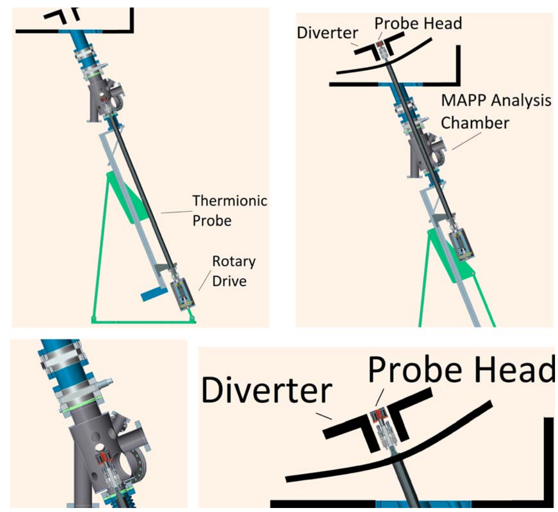

Fig. 4. MAPP analysis chamber and probe assembly shown connected to NSTX TIVs, probe extracted to analysis position (left) and insertion into the divertor (right) for exposure. Zoomed images of the analysis position (bottom left) and inserted MAPP probe (bottom right) illustrates the surface of the probe flush with NSTX divertor floor (i.e., for inserted position).

II. MAPP Surface Anlaysis System

A. Vacuum Chamber

The location of MAPP between two toroidal field coils on the outboard divertor port at bay K requires a compact and unique design for utilization of the X-ray and ion source for characterization techniques such as XPS, direct recoil spectroscopy (DRS), and forward scattering low energy ion spectroscopy (LEISS). A Comstock electrostatic analyzer with a photonics microchannel plate (MCP) allows for charged particle detection. A quadrupole mass spectrometer will be used for TDS [12]. The layout of the MAPP analysis chamber is shown in Fig. 1. An image of the MAPP chamber, shown in Fig. 2, displays the attachment of the MAPP analysis chamber to the torus interface valve (TIV). Mockups of X-ray, ion, and RGA were used to verify that the equipment protruding from the MAPP chamber had adequate clearance volume (90 000 cm^3 constrained volume), and this has been confirmed by the attachment of the MAPP chamber to the TIV.

The MAPP vacuum chamber design was conceived from two *in situ* low-energy irradiation facilities designed and constructed by Allain *et al.* (e.g., IMPACT and PRIHSM) [13]. This design incorporates the abilility to discern between two separate spatial scales in a single facility. In particular, the active spatial scales of PMI in fusion dictate which spatial scales to probe. For example, in fusion PMI, the ion-induced erosion and D recycling are of interest. The former is a mechanism dominant in the first few (0.3–0.6 nm) ML of the surface and the latter 10 nm or deeper. Using LEISS and XPS, respectively, in one facility facilitates measurement of these mechanisms.

B. Sample Holder

The previous PMI probe sample holder was composed of four different samples, which could be simultaneously exposed to the plasma [12]. The design of the MAPP sample holder consists of a linear translation system along with a rotational allowing for surface analysis of any desired sample between or after plasma shots. A wide range of desired samples (ATJ graphite, porous molybdenum, solid Li, W, etc.) can be inserted flush to the divertor sample surface, extracted, and analyzed with a range of characterization techniques. Button heaters under each sample enable the samples to be heated to 800 Deg C for TDS measurements. The approximate location of the probe sample location near the outer strike point (OSP) on the LLD is conveyed in Fig. 3. Plasma control will allow OSP to be swept toward and away from MAPP probe to control deuterium flux on sample surface and study D pumping characteristics.

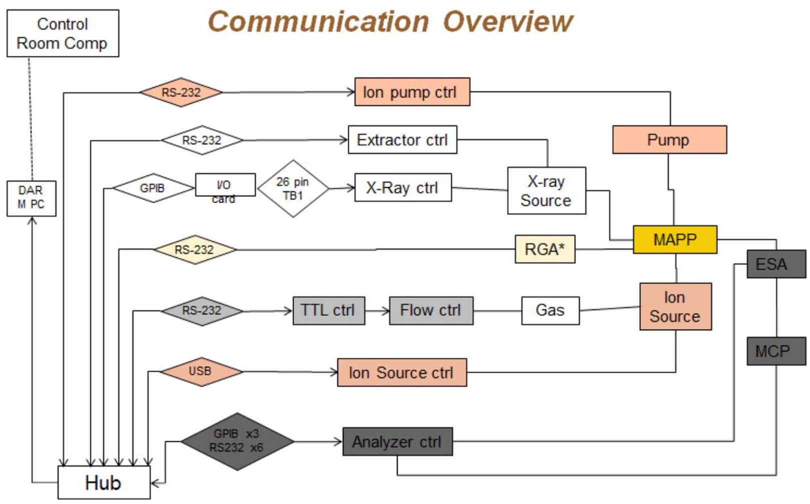

Fig. 5. This is an outline of the communication design for all the MAPP equipment. The Hub is the onsite router for the communication coming from the control room computer. Information is sent to the hub, and the hub routes that information to the proper equipment.

C. MAPP Assembly

MAPP consists of the integration of the remote control Thermionic linear and rotation drives with the analysis chamber and characterization equipment. The probe assembly will allow for precise linear and rotational alignment of samples from the analysis position, Fig. 4 (left), and to the diverter position, Fig. 4 (right). Extensive engineering at PPPL has been carried out for designing and manufacturing of probe system. For example, the MAPP probe insertion protocol had to be integrated into the safety protocol of NSTX diagnostic operational logistics. A remote station for operation of the MAPP probe was established in the NSTX control room. In principle, fully remote-controlled MAPP diagnostic system can be operated (with proper permission) from any physical location and thus also opens the door for multi-user usage in the future.

III. MAPP Remote and Data Acquisition Sytems

A. Remote System

Access to MAPP equipment and equipment controls is typically not possible between plasma shots. This requires a robust and reliable remote control system for both the physical manipulation of the sample probe, as well as, the surface characterization equipment. To accomplish this, a physical communication system was developed in conjunction with a software interface generated using Igor Pro [14]. The use of RS-232 and GPIB protocols provide the communication interface between MAPP control room computer and the mounted equipment, while Igor Pro GUI provides the communication interface between the MAPP operator and the computer. This allows for the MAPP operator to have complete control over the surface characterization equipment and data acquisition at all times. A flow chart of the communication configuration is shown in Fig. 5.

The insertion and extraction of the sample probe are an important interface between MAPP and NSTX. There are remote controlled hard and soft interlocks to protect NSTX from MAPP and vice versa. The use of both hard (no computer control) and soft (computer controlled) interlocks provides safe operation of both MAPP and NSTX during operation and prohibit unwanted equipment operation during restricted times. A 24-V signal sent from NSTX control room signals safe operation for MAPPs equipment. Ten seconds prior and after an incoming plasma shot, the 24-V signal will terminate. Termination of the 24-V signal results in safe, automatic shutdown of all MAPP equipment (terminating power to desired power supplies, inhibiting HV or ramping current). In addition to the 24-V permissive signal, the Igor Pro GUI has several key soft interlocks, which prohibit the misuse of the MAPP system. These interlocks include preventing power supplies from exceeding the safe operating voltage of the equipment, prevention of operating equipment when the chamber pressure is not in the correct range, and prevention of standard MAPP operation when the 24-V permissive signal is terminated. The combined use of both hard and soft interlocks is designed to protect both MAPP and NSTX systems.

B. Data Acquisition

Data acquired by the MAPP system come from the Comstock electrostatic energy analyzer model AC-900B. This is a relatively small analyzer with the entirety of the analyzer being able to sit in a 4×6 in mu metal box. The small size of the analyzer is mandated by the spatial constraints under NSTX and has several important effects on its operation.

Biasing the Comstock analyzer in a fixed transmission energy mode allows for the detection of charged particles via an analog MCP. The MCP output provides an energy spectrum for XPS, LEISS, and DRS characterization techniques. Remote control of all power supplies and applied bias voltage allows for remote switching of pass energy of electrons between 25, 50, and 75 ev. These pass energies dictate the energy resolution and peak to noise ratio of the spectrum and will overall dictate the time needed for a scan to be completed (5–15 min).

During the calibration phase of the MAPP system, it was discovered the use of several pass energies proved useful in analyzing the surface of the calibration sample. Specifically, the use of higher pass energy between 80 and 100 eV induces

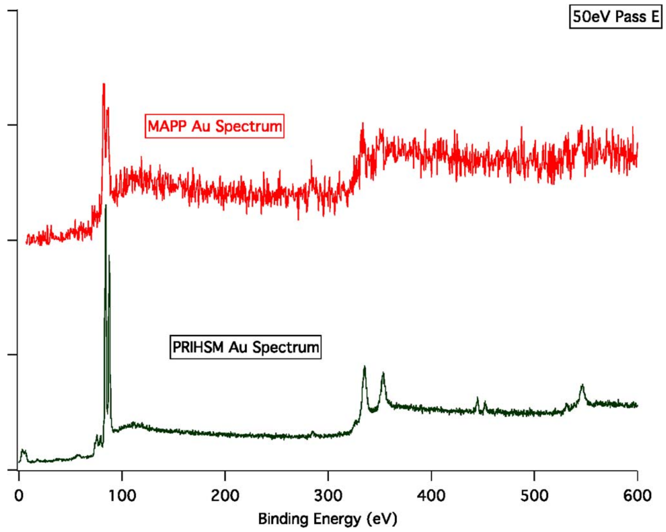

Fig. 6. Wide XPS spectrum comparing MAPP spectrum(top) and PRIHSM (bottom) for Au sample using a 50-eV pass energy.

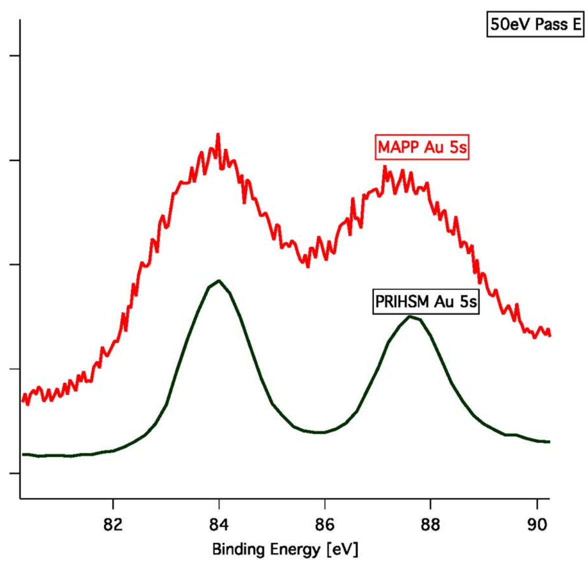

Fig. 7. Wide XPS spectrum comparing MAPP spectrum(top) and PRIHSM (bottom) for Au sample using a 50-eV pass energy.

the higher intensity. However, these settings lower the energy resolution, which is necessary for elemental identification across a wide energy spectrum. Alternatively, when conducting a specific scan on a particular peak or set of peaks, energy resolution becomes increasingly important. Energy resolution of <0.1 eV was achieved with 25-eV pass energy. Peak-to-noise ratio varied with analyzer setting such as dwell time and energy step size.

MAPP initial calibration took place off-site at Purdue University. Use of noble metals (Au, Ag) provided known XPS excitation peaks for comparison with MAPP data. The PRIHSM facility, a neighboring surface analysis facility at Purdue, provided calibrated data for comparison of standard transition metal samples [15].

Figs. 6 and 7 illustrate a comparison between the wide and narrow XPS regions of Au 5s energy spectra between MAPP and PRIHSM. Noise in the MAPP spectrum is apparent and

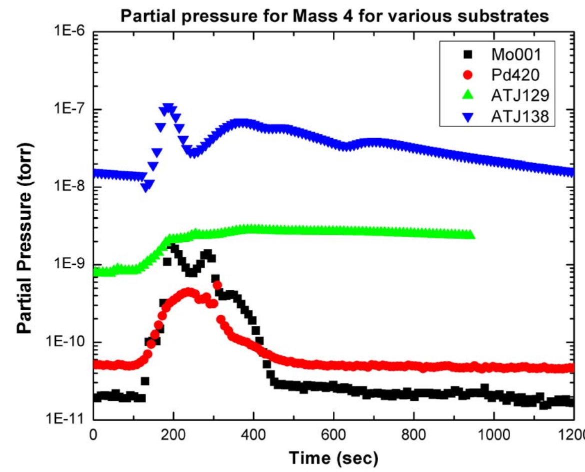

Fig. 8. TDS spectrum of D2 particle pressure after comparing various samples. Mo001 (porous molybdenum sample, TDS post 2 kμm lithium deposition and 30 min D2), Pd 420 (palladium thin film TDS post 2 kμm lithium deposition and 30 min D2 irradiation), ATJ 129 (polycrystalline graphite, TDS post 2 kμm lithium deposition and 30 min D2 irradiation), ATJ 138 (polycrystalline graphite, TDS post exposure to LITER evaporation and plasma shot via PMI probe in NSTX 2011 campaign [12]).

partially due to high secondary electron interaction with the detector. However, the peaks are clearly defined and with a signal-to-noise ratio less than 10% as indicated by identification of each excitation peak, this is sufficient resolution for measurements in the NSTX divertor region.

For the LEISS and DRS measurements ion species of He^+ and Ne^+ will be used between 1 keV and 5 keV at a 30° scattering angle and ion current around 100 nA to minimize the sputtered particles. Knowing the target mass energy and using the binary collision theory, the elastic scattered particles from the first 1–2 monolayers can be analyzed. LEISS measures the surface concentration of Li, C, and O species. DRS measures the hydrogen content at the surface and since measurements are taken between shots can be correlated to D alpha light measurements at the plasma edge.

TDS in MAPP has been calibrated in offline tests and on NSTX. Extensive data have been collected for a variety of materials on both the installed PMI probe [12], and offline experiments. Fig. 8 shows a TDS spectrum for D2 mass species (4 amu) for a variety of different substrates. A temperature ramp of 1 deg C/s up to 550 °C thermally desorbed the implanted D2. Sharp peaks in the spectrum show the different bonding states of D2 and can elucidate on deuterium surface retention for various substrates. For example, a low-energy peak component is found on almost every spectrum. This is currently attributed to the role oxygen plays on retention of deuterium when the presence of lithium brings a large amount of oxygen to the surface.

Further calibrations will consist of detailed DRS and LEISS measurements both offline, and plans are being discussed for installation of the MAPP probe on the Lithium Tokamak Experiment (LTX) at PPPL. The importance of correlating surface chemistry to the behavior of the core plasma with various wall conditioning techniques is motivating the use of MAPP in devices outside NSTX.

The ambient 6–8 Gauss magnetic field at the MAPP location on NSTX could potentially interfere with photoelectron and ion spectroscopy-based techniques. To test the effect of this magnetic field on the energy spectrum, a Helmholtz coil was designed and constructed to surround map in an 8–10 gauss field. No effect to either peak intensity or energy resolution was noticed during high field test at both high- and low-pass energies. This will be further checked by recalibrating MAPP after attachment and integration on NSTX.

IV. Linking PMI Diagnosis With Laboratory Experiments

A. Linking MAPP to Laboratory in situ Experiments

The success of the MAPP system relies in part on the strategy to link fundamental *in situ* experimental data from well-controlled laboratory experiments to data acquired by MAPP during NSTX shots. The primary reason is that in order to elucidate on PMI mechanisms in the complex tokamak plasma edge environment, one must decouple the various irradiation-driven mechanisms with controlled offline experiments. This has been accomplished using the PRIHSM experimental facility at Purdue University. Offline experiments conducted by Taylor *et al.* [6] showed the complex surface chemistry that leads to the retention of deuterium in the lithiated graphite. This was primarily demonstrated by looking carefully at D interactions with C and O in the presence of Li leading to characteristic XPS spectra. Those results led to a series of measurements both ex situ (postmortem) and *in situ* with a early PMI probe version of MAPP in NSTX that helped link the complex surface chemistry in NSTX tile surfaces and XPS data. Furthermore, TDS data compared with gas balance data by Skinner *et al.* helped confirm that the chemical complexes on the surface was primarily due to two primary states of D bonding on the lithiated graphite surface. One consisting of the strong covalent bonding of D atoms with C or O atoms. The second consisting of the Coulombic attraction of D to C or O in the presence of Li. Atomistic modeling recently by Krstic *et al.* is helping elucidate on the fundamental interactions [16].

Another key goal of the MAPP design is to decipher the key measurement variables to link MAPP data to laboratory experiments. Based on the data described above, the MAPP diagnostic system includes XPS and DRS to ensure comparison with well-controlled experiments.

V. Conclusion

MAPP will, for the first time, enable prompt and sophisticated surface analysis of materials exposed on an operating tokamak. The development of complex remote and data acquisition systems interlinked with the intricate challenge of placing MAPP beneath a tokamak represents an engineering milestone for application of surface analysis. MAPP will allow the study of chemical interactions and D retention on a variety of PFS, and for the first time to able to correlate PMI interactions to plasma performance.

References

[1] H. W. Kugel, M. G. Bell, H. Schneider, J. P. Allain, R. E. Bell, R. Kaita, J. Kallman, S. Kaye, B. P. LeBlanc, D. Mansfield, R. E. Nygren, R. Maingi, J. Menard, D. Mueller, M. Ono, S. Paul, S. Gerhardt, R. Raman, S. Sabbagh, C. H. Skinner, V. Soukhanovskii, J. Timberlake, and L. E. Zakharov, "Lithium coatings on NSTX plasma facing components and its effects on boundary control, core plasma performance, and operation," *Fusion Eng. Des.*, vol. 85, no. 6, pp. 865–873, Nov. 2010. DOI:10.1016/j.fusen.2010.04.004.

[2] M. Ono, M. G. Bell, R. E. Bell, R. Kaita, H. W. Kugel, B. P. LeBlanc, J. M. Canik, S. Diem, S. P. Gerhardt, J. Hosea, S. Kaye, D. Mansfield, R. Maingi, J. Menard, S. F. Paul, R. Raman, S. A. Sabbagh, C. H. Skinner, V. Soukhanovskii, and G. Taylor, "Implecations of NSTX lithium results for magnetic fusion research," *Fusion Eng. Des.*, vol. 85, no. 6, pp. 882–889, Nov. 2010. DOI: 10.1016/j.fusengdes.2010.03.045.

[3] R. Maingi, R. E. Bell, J. M. Canik, S. P. Gerhardt, S. M. Kaye, B. P. LeBlanc, T. H. Osborne, M. G. Bell, E. D. Fredrickson, K. C. Lee, J. E. Menard, J.-K. Park, and S. A. Sabbagh, "Triggered confinement enhancement and pedestal expansion in high-confinement-mode discharges in the national spherical torus experiment," *Phys. Rev. Lett.*, vol. 105, no. 13, pp. 135 004-1–135 004-4, Sep. 2010. DOI: 10.1103/PhysRevLett.105.135004.

[4] H. W. Kugel, D. Mansfielda, R. Maingib, M. G. Bella, R. E. Bella, J. P. Allainc, D. Gatesa, S. Gerhardta, R. Kaitaa, J. Kallmana, S. Kayea, B. LeBlanca, R. Majeskia, J. Menarda, D. Muellera, M. Onoa, S. Paula, R. Ramand, A. L. Roquemorea, P. W. Rossa, S. Sabbaghe, H. Schneidera, C. H. Skinnera, V. Soukhanovskiif, T. Stevensona, J. Timberlakea, W. R. Wamplerg, J. Wilgrenb, and L. Zakharova, "Evaporated lithium surface coatings in NSTX," *J. Nucl. Mater.*, vol. 390/391, pp. 1000–1004, Jun. 2009. DOI:10.1016/j.jnucmat.2009.01.262.

[5] W. R. Wampler, C. H. Skinner, H. W. Kugel, and A. L. Roquemore, "Measurement of lithium and deuterium on NSTX carbon tiles," *J. Nucl. Mater.*, vol. 390/391, pp. 1009–1012, Jun. 2009. DOI:10.1016/j.jnucmat.2009.01.262.

[6] C. N. Taylor, B. Heim, and J. P. Allain, "Chemical response of lithiated graphite with deuterium irradiation," *J. Appl. Phys.*, vol. 109, no. 5, pp. 053 306-1–053 306-6, Mar. 2011. DOI:10.1063/1.3555097.

[7] C. N. Taylor, B. Heim, and J. P. Allain, "Post-Mortem NSTX tile surface analysis of plasma interactions with conditioned surfaces," *Nuclear Fusion*, 2011, to be published.

[8] J. P. Allain, D. L. Rokusek, S. S. Harilal, M. Nieto-Perez, C. H. Skinner, H. W. Kugel, B. Heim, R. Kaita, and R. Majeski, "Experimental studies of lithium-based surface chemistry for fusion plasma-facing materials applications," *J. Nucl. Mater.*, vol. 390/391, pp. 942–946, Jun. 2009. DOI:10.1016/j.jnucmat.2009.01.242.

[9] C. N. Taylor, J. P. Allain, B. Heim, P. S. Krstic, C. H. Skinner, and H. W. Kugel, "Surface chemistry and physics of deuterium retention in lithiated graphite," *J. Nucl. Mater.*, vol. 415, no. 1, pp. S777–S780, Aug. 2011. DOI:10.1016/j.jnucmat.2010.09.049.

[10] J. N. Brooks, C. P. C. Wong, and D. G. Whyte, "DiMES divertor erosion experiments on DIII-D," Sandia National Labs., Albuquerque, NM, Tech. Rep. SAND96-1281C, 1996.

[11] C. P. C. Wong, D. G. Whyte, R. J. Bastasz, J. Brooks, W. P. West, and W. R. Wampler, "Divertor materials evaluation system (DiMES)," *J. Nucl. Mater.*, vol. 258–263, pp. 433–439, Oct. 1998.

[12] C. H. Skinner, J. P. Allain, W. Blanchard, H. W. Kugel, R. Maingi, L. Roquemore, V. Soukhanovskii, and C. N. Taylor, "Deuterium retention in NSTX with lithium conditioning," *J. Nucl. Mater.*, vol. 415, no. 1, pp. S773–S776, Aug. 2010. DOI:10.1016/j.jnucmat.2010.08.063.

[13] J. P. Allain, M. Nieto, M. R. Hendricks, P. Plotkin, S. S. Harilal, and A. Hassanein, "IMPACT: A facility to study the interaction of low-energy intense particle beams with dynamic heterogeneous surfaces," *Rev. Sci. Instrum.*, vol. 78, no. 11, pp. 113 105-1–113 105-14, Nov. 2007.

[14] IGOR Pro, Wavemetrics Inc.

[15] O. El-Atwani, J. P. Allain, and S. Ortoleva, "In-situ probing of near and below sputter-threshold ion-induced nanopatterning on GaSb(1 0 0)," *Nucl. Instrum. Methods Phys. Res. B, Beam Interact. Mater. At.*, vol. 272, pp. 210–213, Feb. 2012. DOI:10.1016/j.nimb.2011.01.067.

[16] P. Krstic, J. P. Allain, A. Allouche, J. Jakowski, E. Yang, J. Dadras, and C. N. Taylor, *Fusion Eng. Des.*, 2011, to be published.

Author photographs and biographies not available at the time of publication.