Development progress of the Materials Analysis and Particle Probe[a)]

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The Materials Analysis and Particle Probe (MAPP) is a compact *in vacuo* surface science diagnostic, designed to provide *in situ* surface characterization of plasma facing components in a tokamak environment. MAPP has been implemented for operation on the Lithium Tokamak Experiment at PPPL, where all control and analysis systems are currently under development for full remote operation. Control systems include vacuum management, instrument power, and translational/rotational probe drive. Analysis systems include onboard Langmuir probes and all components required for x-ray photoelectron spectroscopy, low-energy ion scattering spectroscopy, direct recoil spectroscopy, and thermal desorption spectroscopy surface analysis techniques. Once completed, MAPP will be integrated into the National Spherical Torus Experiment-Upgrade at PPPL for its first run campaign.

I. INTRODUCTION

The plasma-material interface in tokamaks is a region of complex coupled relationships, with the state of both plasma and surface strongly influencing each other. The average interaction region of energetic hydrogen ions incident on the material surface can range between 1-100 nm. The average distribution of emitted (e.g. by sputtering) particles generally emanate from the top few monolayers of the surface. Surfaces at plasma boundaries of tokamaks are intrinsically sensitive to compositional changes through the course of a single plasma shot dictating the behavior of the plasma core via the plasma edge. Reactive boundaries, such as lithium coatings, are sensitive to chemical changes as well as compositional changes.

Understanding plasma-surface interactions in this region is critical to the continued advancement of magnetic fusion research; to this end, surface analysis is a powerful quantitative tool that can help achieve this goal via characterization of plasma facing component (PFC) surfaces. Ideally, surfaces should be interrogated promptly before and after individual plasma exposures, eliminating as many confounding variables as possible. However, past experiments have relied on *ex situ* post-analysis of the PFCs, attempting to recover surface conditions assumed to exist during plasma exposure.[1] Significant air exposure and time lapse are serious challenges to this type of analysis.

In contrast, the Materials Analysis and Particle Probe (MAPP) diagnostic—a collaborative effort between PPPL and UIUC—is a compact *in vacuo* surface science diagnostic, designed to provide *in situ* surface characterization of PFCs in a tokamak environment.[2] MAPP provides a sample analysis chamber within the vacuum envelope of the tokamak, so that its interchangeable sample disks can be exposed to tokamak plasma discharges and then promptly investigated with a variety of surface analysis diagnostic systems: x-ray photoelectron spectroscopy (XPS), low-energy ion scattering spectroscopy (LEISS), direct recoil spectroscopy (DRS), and thermal desorption spectroscopy (TDS).[3,4]

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II. MAPP EXPERIMENTAL DESIGN

A. Vacuum hardware and manipulation

The MAPP features a private vacuum vessel for sample analysis, which can be sealed and independently pumped. Following proper vacuum conditioning and baking, an ultimate base pressure of 3×10^{-8} Torr has been reached with an 80 L/s turbomolecular pump backed by a dry scroll pump. A 200 L/s Gamma Vacuum ion pump is available as a third stage pump once the pressure is below 10^{-5} Torr.

The MAPP sample holder head carries four small (d = 0.94 cm) interchangeable sample disks, and it is mounted on a bellows drive for translational motion. The 0.96 m bellows stroke allows the samples to be inserted into a plasma exposure chamber (i.e. a tokamak) and then retracted into the private analysis chamber. MAPP samples are oriented on the head such that one of them is at the so-called “supercenter”. The supercenter is defined by the intersection volume of all solid angles from MAPP’s sources and detectors; a sample at the supercenter can thus be investigated with any of MAPP’s surface analysis techniques. A custom Geneva drive allows for precise indexing of each sample at the supercenter.

B. Surface analysis components

For XPS analysis, MAPP uses a PSP Vacuum TX400 x-ray source to irradiate the sample at the supercenter with x-rays. Some of the ejected photoelectrons and Auger electrons are then scattered into a Comstock AC-901 electrostatic energy analyzer, where they are selected for energy. A pair of microchannel plate detectors on the outlet of the analyzer provides the required gain for counting individual electrons. The x-ray source has dual magnesium and aluminum filaments, so that either Mg K_α (1253.6 eV) or Al K_α (1486.6 eV) x-rays are available for sample analysis. The x-ray source is oriented so that the beam strikes 56° from the sample normal.

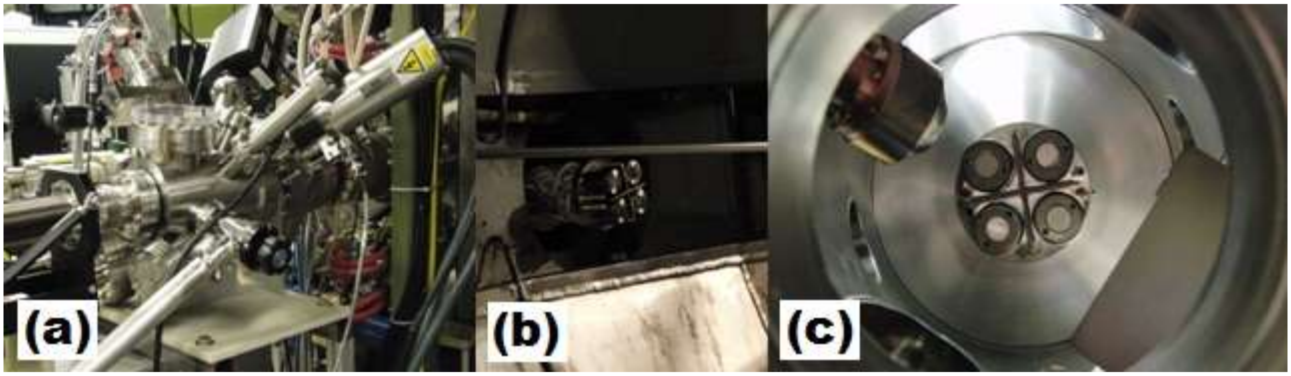

Fig. 1 – (a) MAPP attached to LTX; (b) MAPP head in extended exposure position; (c) MAPP head in retracted analysis position

The analyzer is of hemispherical sector design, operated in constant pass energy mode. The two hemispherical sectors are currently biased so that 50 eV ions/electrons reach the detector. This pass energy was determined to have the best compromise between energy resolution and statistical count rate.[5] The entire analyzer is then separately biased such that an ion/electron of a given energy is slowed to 50 eV before entering the analyzer. The analyzer is oriented so that its acceptance solid angle is oriented 70° from the sample normal, in a forward scattering configuration from the x-ray and ion sources.

For LEISS and DRS analysis, MAPP uses an NTI 1404 ion source to irradiate the sample at the supercenter with He^+, Ne^+, or Ar^+ ions. Depth profiling is also possible with the ion source, as it can provide enough current to operate as an effective sputter gun. The ion source is oriented such that the beam strikes 80° from the sample normal, a grazing incidence that amplifies ion scattering sensitivity to the top surface monolayer. The same analyzer is used with the ion source, but biased oppositely to slow ions to 50 eV pass energy.

For TDS analysis, MAPP uses integral 60 W HeatWave 101137 button heaters. These are rated for 1200 °C operation under high vacuum and are held in thermal contact against the back side of the samples with inconel wave-springs. Heat shielding baffles between samples help prevent cross-heating of other samples. A residual gas analyzer (RGA) is oriented with a direct view of the sample face. During a TDS scan, the RGA monitors the evolution of volatiles from the surface as the temperature increases. For high temporal resolution, eight pre-selected mass peaks are monitored, rather than the full spectrum.

C. Plasma diagnosis

The MAPP head also carries two cylindrical (r_p = 0.8 mm, adjustable length) Langmuir probe tips for local plasma diagnosis around the samples. These provide a measure of plasma ion flux to the samples during an exposure. When biased sufficiently negative, Langmuir probes collect a fixed electrical current proportional to the ion flux. This allows comparison of observed surface changes against measurements of plasma flux and total fluence to the samples. By sweeping the bias in and out of this region, local plasma electron temperature and ion density measurements are also possible.[6] Probes are biased with a Kepco 50-8M BOP power supply.

III. DIAGNOSTIC DEVELOPMENT RESULTS

A. MAPP redesign for LTX

The MAPP has been designed to operate in the experimental environment of the National Spherical Torus Experiment Upgrade (NSTX-U) at PPPL, which requires that all control and analysis systems are capable of full remote operation. To provide the capability for testing MAPP prior to NSTX-U installation, the system was redesigned to attach to the Lithium Tokamak Experiment (LTX) at PPPL [Fig. 1(a)].[7] On NSTX-U, MAPP will enter the vessel through the lower divertor in a near-vertical configuration, but on LTX, MAPP must enter the vessel horizontally through the outboard midplane shell gap [Fig. 1(b)].

B. Remote operation system upgrade

MAPP's remote control system is based in a suite of interlinked LabVIEW® Virtual Instruments (VIs), one for each piece of equipment. The interaction among the VIs is managed by two central VIs. One controls a local interlock system based on the pressure gauge installed in the chamber. It generates a safety signal that is distributed to other drivers, acting to ensure a safe operational pressure for the other devices—the x-ray source, microchannel plate detector, ion pump, *etc.*

The other controls a global interlock system, critical for MAPP's operation in NSTX-U. Various MAPP components must be de-energized during NSTX-U plasma discharges due to the Tesla-scale magnetic fields present. Between discharges, these components may be safely energized for surface analysis. A minimum of 12 min must elapse between discharges, providing a window for MAPP operation and analysis. The global interlock system interrupts power to the instruments before a discharge, while a custom circuit prevents overloading of the instruments.

In this environment, analysis system VIs are running to acquire XPS, TDS, and ISS data. These VIs control the relevant voltages for each system, performing the required parameter scans, and then aggregate the data to generate spectra.

C. Rotational sample manipulation

As with other MAPP systems, the Geneva drive controlling the rotational sample manipulation must be automated for remote operation. However, the nature of this mechanism requires special consideration: Cables leading from vacuum feedthrough to MAPP head are fixed on one end and rotating on the other. They are long enough for a full rotation, but continued rotation in

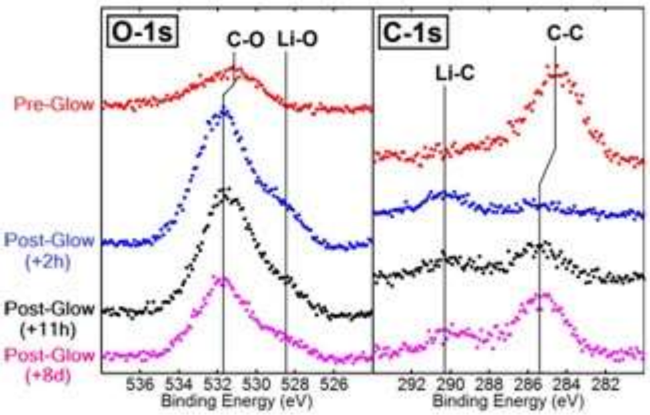

Fig. 2 – XPS scans of C-1s and O-1s photoelectron regions before and after MAPP sample exposure to an Ar glow in LTX, showing the appearance of chemical shifts following the glow

a single direction would break the electrical connections. The remote operation system must be thus able to prevent accidental over-rotation in a given direction.

To accomplish this, another VI control interface is under development. The system will take inputs from micro-switches located at seven of the eight Geneva drive positions. By software linkage with the main control system, the operator will be able to determine which sample is at the supercenter and safely switch among them. With proper positioning of the micro-switches, the system can even report if any mechanical problems arise in the Geneva drive.

D. LTX *in situ* XPS

The XPS capabilities on MAPP are fully operational and have been successfully tested in dedicated experiments on LTX.[8] Photoelectron and Auger electron peaks are easily distinguishable, and quantitative analysis of peak areas has been used to successfully determine elemental composition fractions. Signal levels are high enough to quantify even the low-cross section Li-1s photoelectron peak with uniform Li coatings just a single monolayer thick. Intrinsic energy resolution, as determined by the FWHM of the Au-4f photoelectron peak in a gold reference sample, has been measured at 2.5eV. This is sufficient to resolve some of the chemical shifts; as seen in Fig. 2, *in situ* argon glow discharge exposure experiments have led to appearance of peaks in the C-1s and O-1s photoelectron spectra corresponding to Li-C and Li-O bonding.

Energy calibration has been preferentially performed using the Au-4f photoelectron peak of a gold reference standard inserted in one of the four MAPP sample positions. However, this method not only occupied a sample holder location, but the Au-4f signal was easily attenuated by a thin overlayer coating. This led to the design of a gold reference that can be inserted from one of the other supercenter-facing ports when the MAPP head is retracted behind the supercenter. Initially behind a gate valve, the sample is extended into the supercenter via a differentially-pumped translation feedthrough.

E. LTX *in situ* TDS

The TDS capabilities on MAPP have been tested in a "manual" mode, where RGA and temperature data are recorded separately while heater power is increased by an autotransformer. These tests have demonstrated that the MAPP hardware will allow satisfactory TDS analysis, so that the system should work as designed once the software interface is completed. MAPP now has heaters located beneath all four of the samples. Thermocouples placed inside the heater stems are used to obtain the sample temperature after proper calibration. Good thermal isolation between the samples has been demonstrated, as each sample can reach its maximum temperature of over 700 °C during a TDS run before any other samples exceed 100 °C.

F. Onboard Langmuir probes

MAPP's onboard Langmuir probes are fully operational and providing local measurements of plasma electron temperature and ion density. Probes have been swept at 2 kHz, so plasma flux to the probe can be calculated with 500 μs time resolution during plasma discharges. Temporal integration then provides total ion fluence over the entire discharge.

From these results, it is likely that LTX plasmas are not intense enough to rapidly alter the sample surfaces, at least at MAPP's outboard midplane location. In the highest-power LTX discharges, MAPP's onboard swept Langmuir probes have measured an ion fluence of 1×10^{19} m^{-2}, only a single monolayer equivalent of hydrogen. By contrast, the centerstack triple probes and visible fast camera suggest that LTX plasmas have been limiting mainly on the centerstack. The triple probes have measured a much larger ion fluence of 6×10^{20} m^{-2}, which corresponds to a regime where plasma-induced surface alterations have been observed.[9]

IV. CONCLUSIONS

The MAPP's attachment to LTX has allowed for extensive testing of all control and analysis systems. The XPS analysis capability and the onboard Langmuir probes are fully operational. TDS hardware has been successfully tested in a non-automated mode and will be complete when controllable by the software interface. The ion source remains under development but should be ready for the start of *in situ* testing within the next few months. Control systems for translational and rotational motion are also in the final stages of development. From the completed and ongoing testing, MAPP will be prepared for full operation on NSTX-U in time for the start of its first run campaign.

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