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Unraveling wall conditioning effects on plasma facing components in NSTX-U with the Materials Analysis Particle Probe (MAPP)

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A novel Plasma Facing Components (PFCs) diagnostic, the Materials Analysis Particle Probe (MAPP), has been recently commissioned in the National Spherical Torus Experiment Upgrade (NSTX-U). MAPP is currently monitoring the chemical evolution of the PFCs in the NSTX-U lower divertor at 107 cm from the tokamak axis on a day-to-day basis. In this work, we summarize the methodology that was adopted to obtain qualitative and quantitative descriptions of the samples chemistry. Using this methodology, we were able to describe all the features in all our spectra to within a standard deviation of ±0.22 eV in position and ±248 s^{-1} eV in area. Additionally, we provide an example of this methodology with data of boronized ATJ graphite exposed to NSTX-U plasmas. *Published by AIP Publishing.* [http://dx.doi.org/10.1063/1.4955276]

I. INTRODUCTION

Plasma-material interactions (PMIs) can have critical effects on plasma performance including changes in plasma current, discharge length, energy confinement time, and Edge Localized Mode frequency.[1] Conditioning techniques of Plasma Facing Components (PFCs) are used in the National Spherical Torus Experiment Upgrade (NSTX-U) to provide hydrogen particle control and enhanced PFC performance.[2] The use of Li coatings has reduced hydrogen recycling by as much as 70% in CDX-U and NSTX leading to improved plasma performance.[3,4] The formation of Li–O–D complexes was identified as the main retention mechanism in graphite PFCs in NSTX.[5] Similarly, results obtained in Lithium Tokamak Experiment (LTX) indicated Li_2O bind hydrogen and improve plasma performance.[6] The ability to identify these important mechanisms requites controlled *in vacuo* and *in situ* experiments (both in-vessel and ex-vessel, respectively) combined with advanced computational modeling.

Decoupling the cause-effect relationships in the multivariable dependence of plasma performance to surface chemistry is one of the biggest challenges in PMI today. The Materials Analysis Particle Probe (MAPP)[7,8] is an *in vacuo* characterization facility for the analysis of materials in-between plasma discharges in NSTX-U.[7,9] The result is an increase in three orders of magnitude in time resolution compared to post-mortem PFC characterization conducted after one-year NSTX campaigns. The time resolution is an average of 1-2 plasma shots with depth resolutions from 0.3 to 10 nm. The time and depth resolutions are critical for correlating surface chemistry changes and edge plasma-to-core plasma behavior.

MAPP is collecting XPS data during the 2015-2016 campaign of NSTX-U. The analysis of XPS data obtained from MAPP imposes additional challenges compared with commercial XPS systems. Those are primarily dictated by size constraints of the energy analyzer that must be integrated in a custom-designed chamber under the NSTX-U tokamak divertor access port.

This work summarizes the methodology adopted to fit and analyze the XPS data collected by MAPP. Our goal is to establish standardized data analysis routines that would set better scientific foundations to our conclusions, which at the end will ease the task of correlating the PFCs conditions with plasma performance.

II. METHODS AND MATERIALS

The NSTX[10] has recently concluded an upgrade period that lead to a substantial increase in the plasma current (2 MA), toroidal magnetic field (1 T), NBI available power, and maximum pulse duration (5-8 s).[11]

MAPP is a PFC diagnosis especially designed for NSTX-U, however its design is flexible enough to allow its use in other tokamak machines. In the present, XPS and TDS are fully operational while the remaining techniques are under development. XPS is MAPP's main analysis tool, due to time and logistic constraints in NSTX-U during plasma operations; the information that the technique provides is obtained non-destructively.

The facility uses a dual anode X-ray source (PSP Vacuum TX400) in combination with a Concentric Hemispherical Analyzer (CHA) model Comstock AC-901. The final photoelectron signal is collected with two Micro-Channel Plates (MCPs) installed chevron configuration. The system operates

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based on a LabVIEW® interface that synchronizes the voltage applied to the sectors of the analyzer with the total counts read from the MCPs to create a spectrum.[9] The analyzer operates at 50 eV constant transmission mode, which sets the minimum resolution.

III. DATA ANALYSIS METHODOLOGY

A. XPS data collection optimization

Due to the size limitations imposed by the installation of a surface facility in an environment as complex as NSTX-U, MAPP's hemispherical energy analyzer is considerably smaller than those used by commercial systems.[8] In general, the resolution of Concentric Hemispherical Analyzers (CHAs) is a function of their mean radius, the entrance and exit slits, and the angle subtended by the hemispherical sectors[12] in addition to the pass or transmission energy. In addition, the signal intensity is smaller as a consequence of the size of the CHA. Thus, in order to obtain good resolution in combination with counts sufficient for good statistics, the acquisition time has to be optimized. To reduce the effect of noise in the collected signal, an approach with small gate time (time to read the pulses from the MCP) and an increased number of scans of the particular XPS region was adopted for the collection of data. The noise is, in general, random in contrast with the signal and must be the same over multiple times; taking the average of multiple scans reduces the effect of noise and smoothens the signal. Figure 1 shows the effect of increasing the number of scans.

B. Binding energy calibration

Having established a proper number of scans per region, to provide traces smooth enough to fit with XPS dedicated software, the next step is to use such fittings to calibrate the system and characterize its accuracy and precision. It is a common practice in all XPS facilities to do a regular check of a known photoelectric peak and compare the value obtained in the machine with that reported in the literature. In MAPP, this is done with a gold sample. Gold's most intense XPS peaks are located at 88.0 eV and 84.3 eV. Those are the energies of the $4f_{5/2}$ and $4f_{7/2}$ core electrons, and according to the orbit splitting of the f-subshell, the ratio of the area of the peaks is 3:4.[13]

The fittings of the peaks are shown in Figure 1. In addition to the constraints listed above, the FWHM of the peaks has been set to be equal. According to the results, the $4f_{5/2}$ peak measured by MAPP is at 86.5 eV, giving a 1.5 eV offset to calibrate MAPP's measurements.

C. Estimation of experimental uncertainty

To assess the accuracy of the system, fittings of 12 different scans on the 4f doublet region were performed. The traces were fitted to analyze the variation on the peaks' features. With this information, the values of uncertainty in position and area (which translates into the uncertainty in the atomic fractions) can be computed. The error in the peak position and peak area is taken as the standard deviation computed in the twelve different fittings. This is summarized in Figure 2.

D. Methodology for species identification and quantification of XPS data

The methodology applied to the XPS data in order to identify the chemical species present in the samples can be described as follows:

- Identification of all potential bonds types and compounds on the samples based on the data reported in the literature. These species can be also identified in controlled laboratory experiments performed on the same samples studied in MAPP.
- First iteration of fittings with first set of constraints:
  - Set position of peaks on the binding energy scale as a constant.
  - Limit the range of FWHM of peaks. Since the FWHM of XPS peaks is a measure of the system's

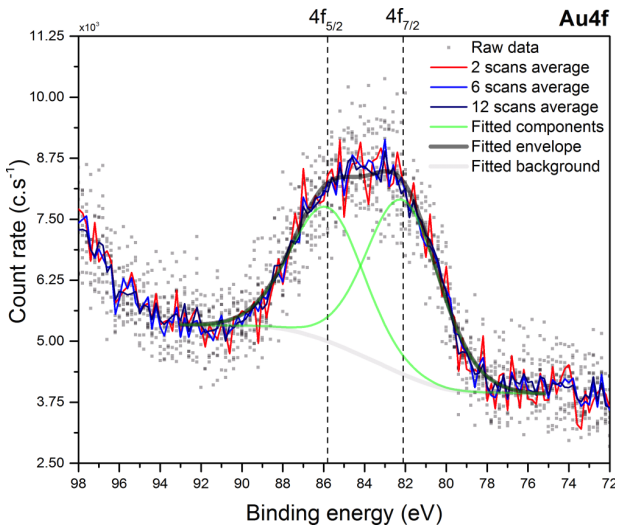

FIG. 1. Au4f region scan showing the progressive reduction of the effect of noise in the signal when taking the average of multiple scans.

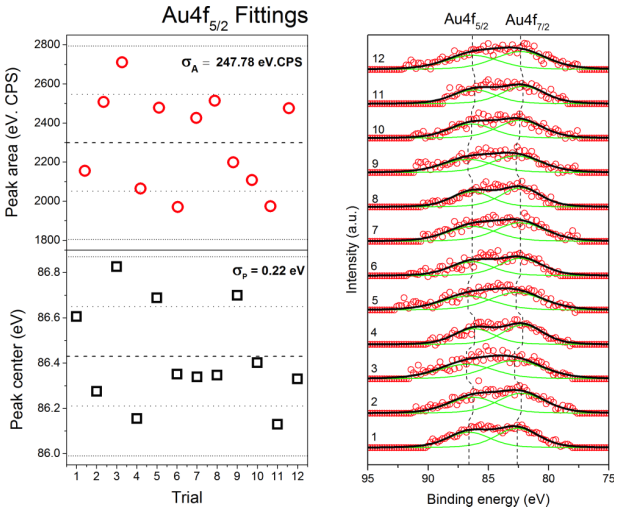

FIG. 2. Estimation of error in MAPP's data and fittings using 12 Au4f traces. The standard deviations in fitted areas and peaks' positions are shown. The right panel shows the analyzed data.

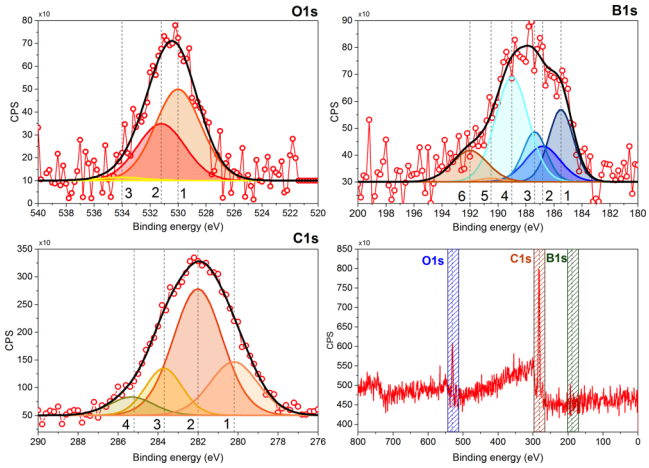

FIG. 3. Example of data fitting using the methodology described in this work. The sample is boronized ATJ previous to plasma exposure in NSTX-U. Further information on the peaks can be found in Table I.

resolution, the minimum FWHM that the detector can identify will be set by the resolution.

- Fit the set of peaks and record FWHM, this value should be constant for each peak.

- Impose the same set of constraints, i.e., peaks' position and defined FWHM (from last step) to the next set of scans.

Since in its routine operation MAPP acquires data from the same set of samples on a day-to-day basis, the peaks observed in a particular day should keep the same properties, i.e., position and FWHM in the upcoming days. This sets the constraints for the data set (data from one sample in different days) and creates an analysis guide for the new fittings. In this way, the final outputs of this method are the areas of the peaks which can be used to monitor the chemical state and the composition of the samples.

IV. EXAMPLE RESULTS

Figure 3 shows a set of data fitted with the methodology described in Section III D. MAPP is currently being used in NSTX-U to study the chemical evolution of boronized ATJ graphite exposed to D^+ irradiation. Details about the boronization procedures in NSTX-U can be found somewhere else.[14,15] The data shown in Figure 3 were taken 30 minutes after a boronization in the tokamak.

The set of constraints used to obtain the fittings shown in Figure 3 are listed in Table I. All the fittings reported in this worked were performed using CASAXPS software.

The fittings in Figure 3 reveal the existence of boron on the ATJ samples, most likely in the form of boron carbide (B_4C).

TABLE I. Summary of the constraints used as inputs for the fittings in Figure 3.

ID	Bond	Position[16] (eV)	FWHM (eV)	Region
1	B–C	185.5	2.1	B1s
2	B–B	186.8	2.9	
3	B–H	187.4	2.0	
4	B–OH	189.0	2.8	
5	NSO	190.5	2.0	
6	B_2O_3	192.0	3.0	
1	B–C	280.2	2.9	C1s
2	C–C	282.0	2.2	
3	C–O, C–OH	283.7	2.6	
4	Carb	285.2	2.8	
1	B–O	530.0	4.0	O1s
2	C–O, C–OH	531.2	3.9	
3	B–OH	534.0	4.0	

V. CONCLUSIONS

We have successfully established an analysis routine for XPS data collected during *in vacuo* experiments with MAPP in NSTX-U. Our approach is based on adopting well known surface science analysis methods to a less controlled environment. Despite the noisy appearance of our XPS spectra, our error analysis indicates that a set of fits of the Au4f doublet only varies ±0.22 eV in position and 247.78 s^{-1} eV in area. This encouraging result guarantees the repeatability of our measurements. It is worth mentioning that MAPP's approach is part of a bigger enterprise that involves validation of its measurements with results from computer simulations and controlled laboratory studies. The data and work described here encompass the initial results of the *in vacuo* component of the bigger effort.

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