U.S. DEPARTMENT OF ENERGY | Office of Science

NSTX-U

XPS characterization of PFCs in NSTX-U with MAPP

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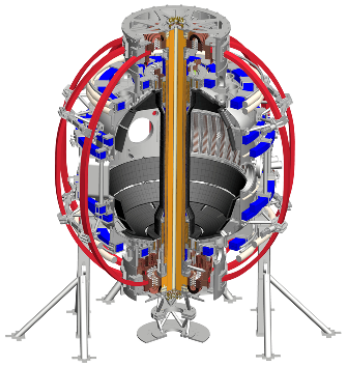
NSTX-U Results Review
PPPL
21, 22 September, 2016

RSSEL

PPPL

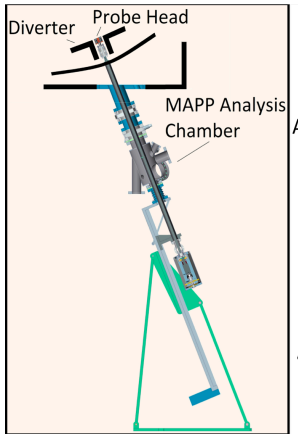


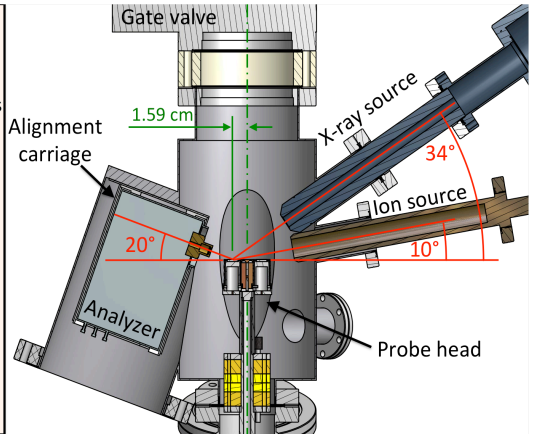
Materials Analysis Particle Probe (MAPP)

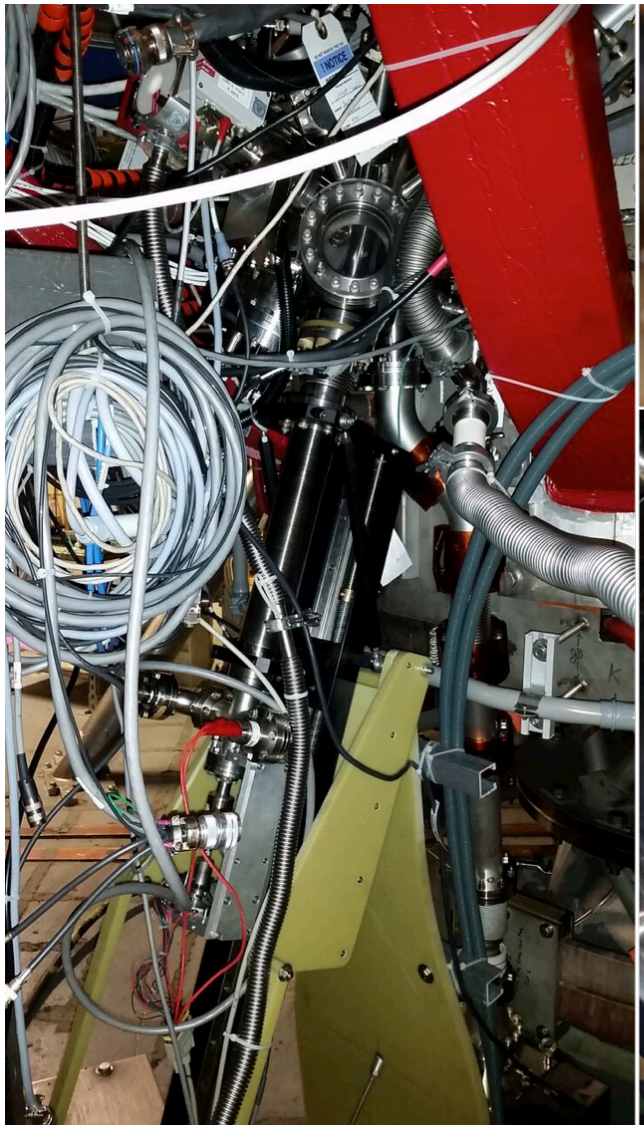
The Materials Analysis Particle Probe (MAPP) is s characterization facility for on-line diagnostic of samples exposed to fusion reactor plasmas in between discharges

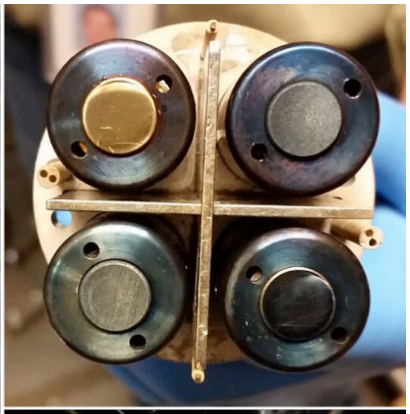
Analysis tools:

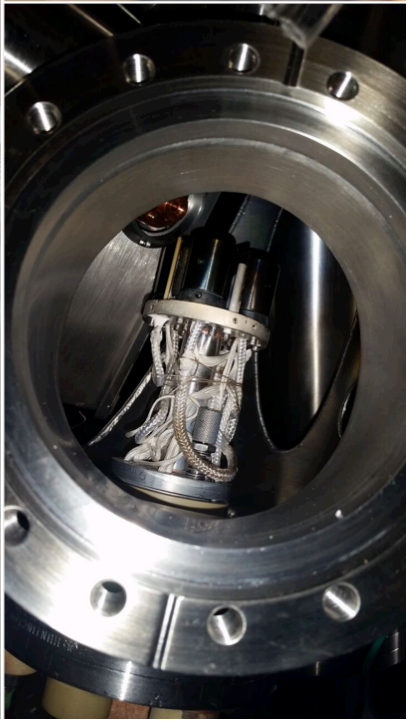
- Custom design UHV chamber
- Compact small diameter hemi-spherical energy analyzer
- Dual anode X- ray source 1 keV ion source 200^{o}C UHV heaters
- Residual Gas Analyzer
- Langmuir probes





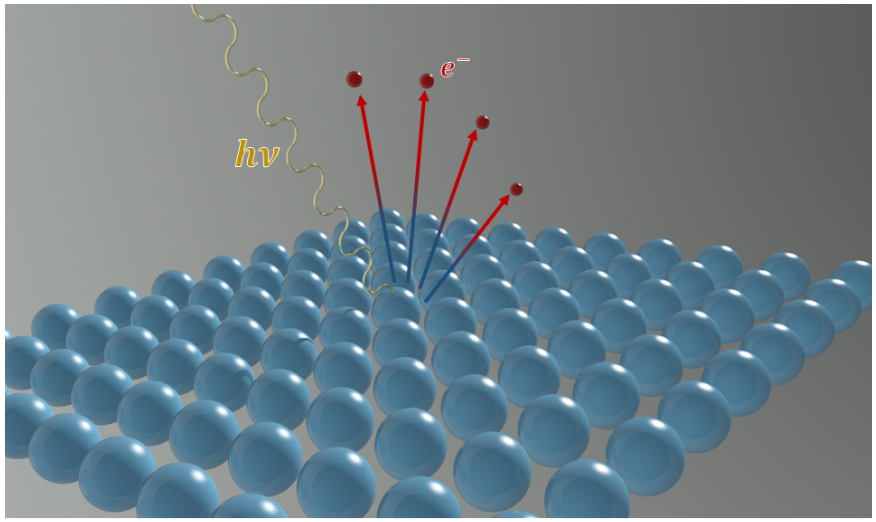


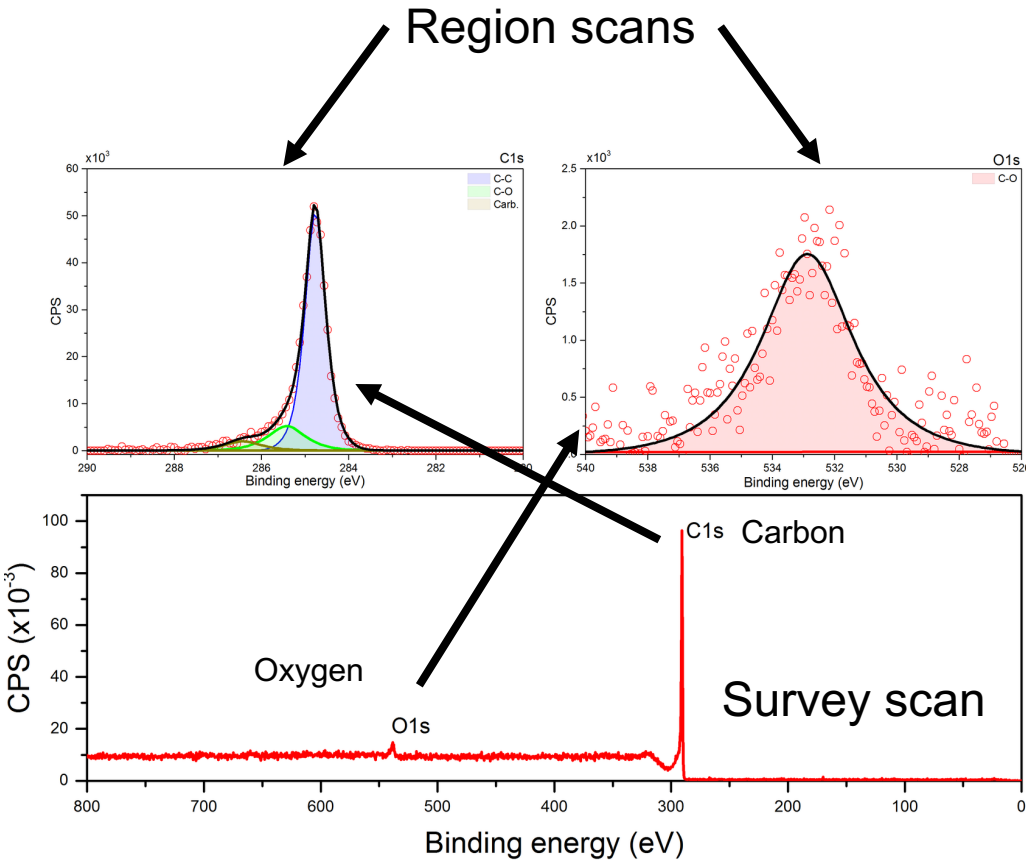




X-ray Photoelectron Spectroscopy (XPS)

- Emission of photoelectrons under incident soft X-ray beam.
- Probe top 10 – 20 nm of sample
- Elemental identification in survey scans
- Region scans allow quantitative analysis (atomic percentage)





XPS data acquisition methodology

- **#1_ATJ:**
  - Survey (when time allows)
  - B1s
  - O1s
  - C1s
- **#2_TZM:**
  - Survey (when time allows)
  - B1s
  - O1s
  - C1s
  - Mo3ds
- **#4_Au:**
  - Survey (when time allows)
  - B1s
  - O1s
  - C1s
  - Au4f

M	T	W	R	F	S	S
1/28	29	30	31	2/1	2	3
4	5	6	7	8	9	10
11	12	13	14	15	16	17
18	19	20	21	22	23	24
25	26	27	28	29	3/1	2

M	T	W	R	F	S	S
2/29	3/1	2	3	4	5	6
7	8	9	10	11	12	13
14	15	16	17	18	19	20
21	22	23	24	25	26	27
28	29	30	31	4/1	2	3

M	T	W	R	F	S	S
3/28	29	30	31	4/1	2	3
4	5	6	7	8	9	10
11	12	13	14	15	16	17
18	19	20	21	22	23	24
25	26	27	28	29	30	5/1

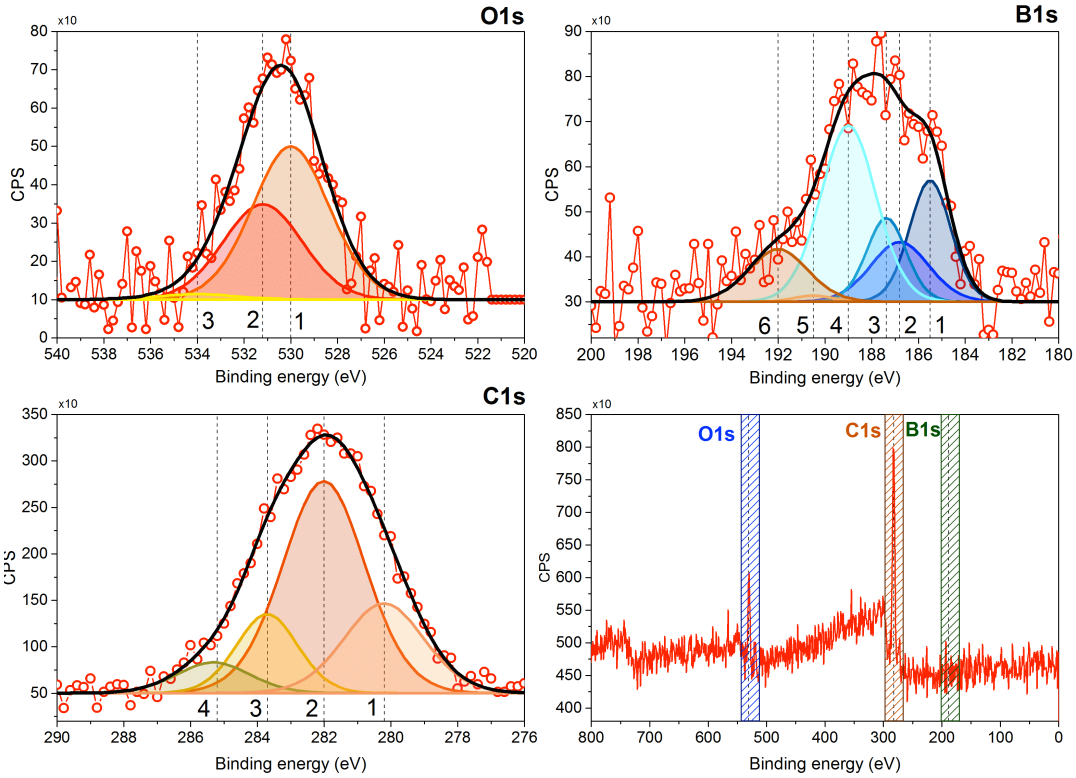
Table 2.6: Calendar of MAPP's samples activities between February/2016 and April/2016. Conventions: • Boronization, • Mini boronization, • GDC, • Maintenance day, • Vent, • New samples installed, • XPS, • Plasma exposure

XPS data analysis

Deconvolution of peaks allows identification of chemical bonds

ID	Bond	Position (eV)	FWMH (eV)	Region
1	B-C	187.2	2.1	B1s
2	B-B	188.1	2.9	
3	B-D	189.1	2.0	
4	B-OD	190.2	2.8	
5	NSO	191.7	2.0	
6	B_2O_3	193.2	3.0	
1	B-C	282.7	2.9	C1s
2	C-C	284.5	2.2	
3	C-O, C-OD	288.9	2.6	
4	Carb.	285.2	2.8	
1	B-O	531.5	4.0	O1s
2	C-O, C-OD	532.7	3.9	
3	B-OD	533.9	4.0	

Deconvolution of XPS spectra of boronized ATJ sample (MAPP data in NSTX-U)[1]



Source:

- Extensive literature review
- Baseline and reference measurements in laboratory (HR-XPS) collaboration with Princeton University

[1] F. Bedoya, et al., Review of Scientific Instruments **87**, 11D403 (2016).

Effect of boronization on PFCs in NSTX-U

- **Boron oxidation due to plasma exposure measured with MAPP via XPS**

High oxygen %
Low % of B in metallic state

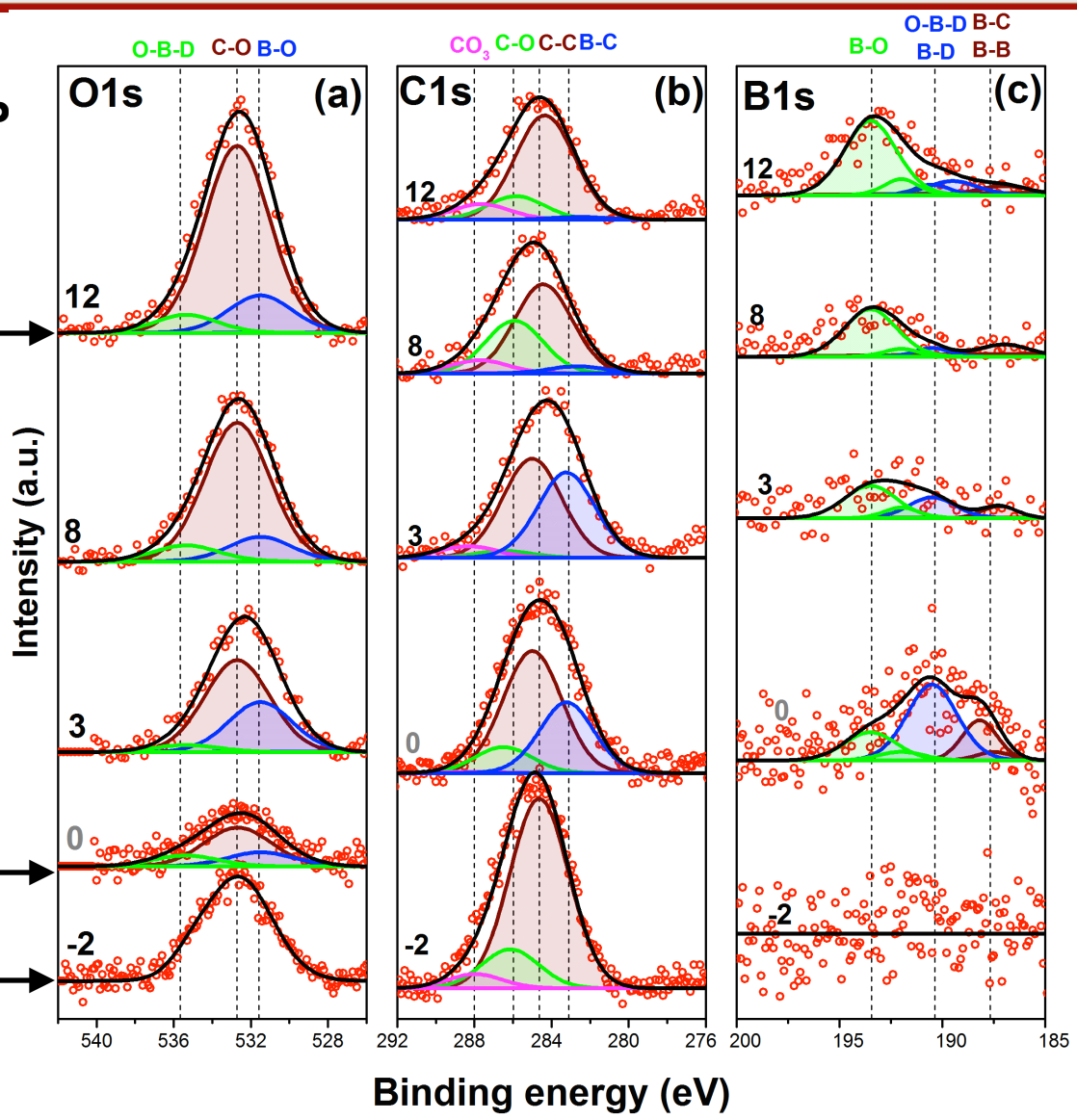
3. ATJ post boronization post plasma exposure

Days after boronization
D^+ fluence increasing

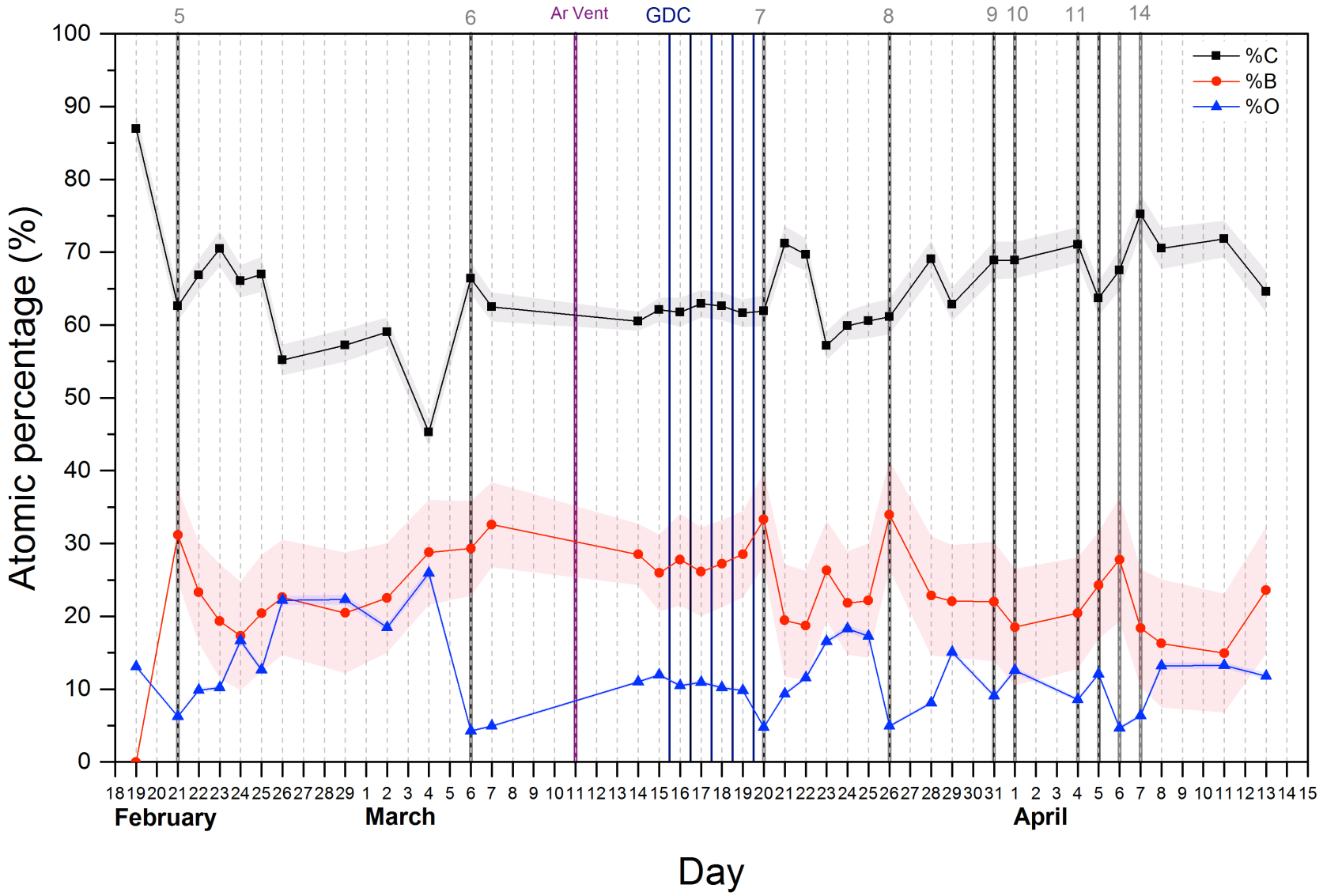
Low oxygen %
High % of B in metallic state

2. ATJ post boronization

1. ATJ graphite



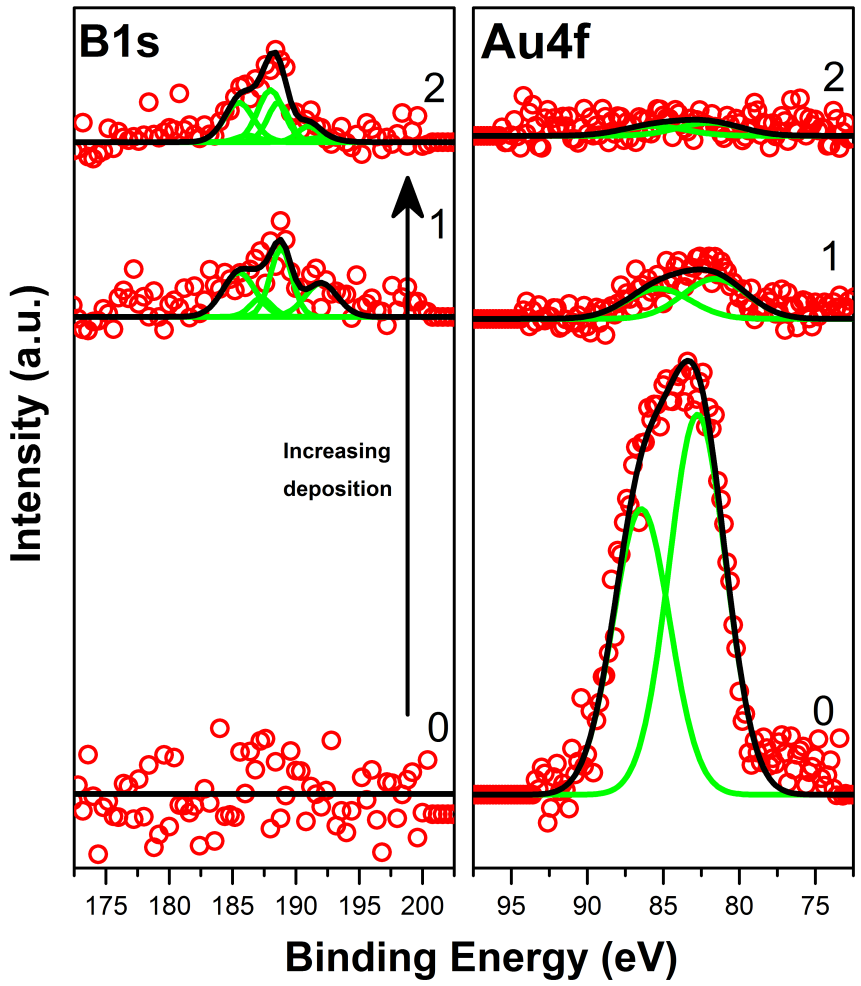
Evolution of atomic concentrations

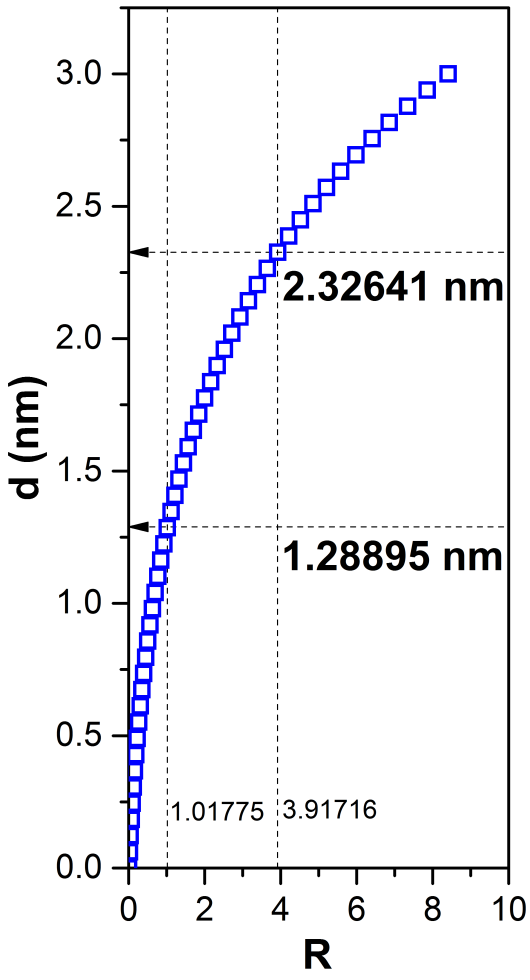


Deposition in weekly vs. daily boronizations

Boron thickness on Au sample

- Weekly d-TMB saturates probed depth of XPS thus the coatings are >20nm
- Daily d-TMB deposit ~1.5 nm on MAPP's position LD bay K



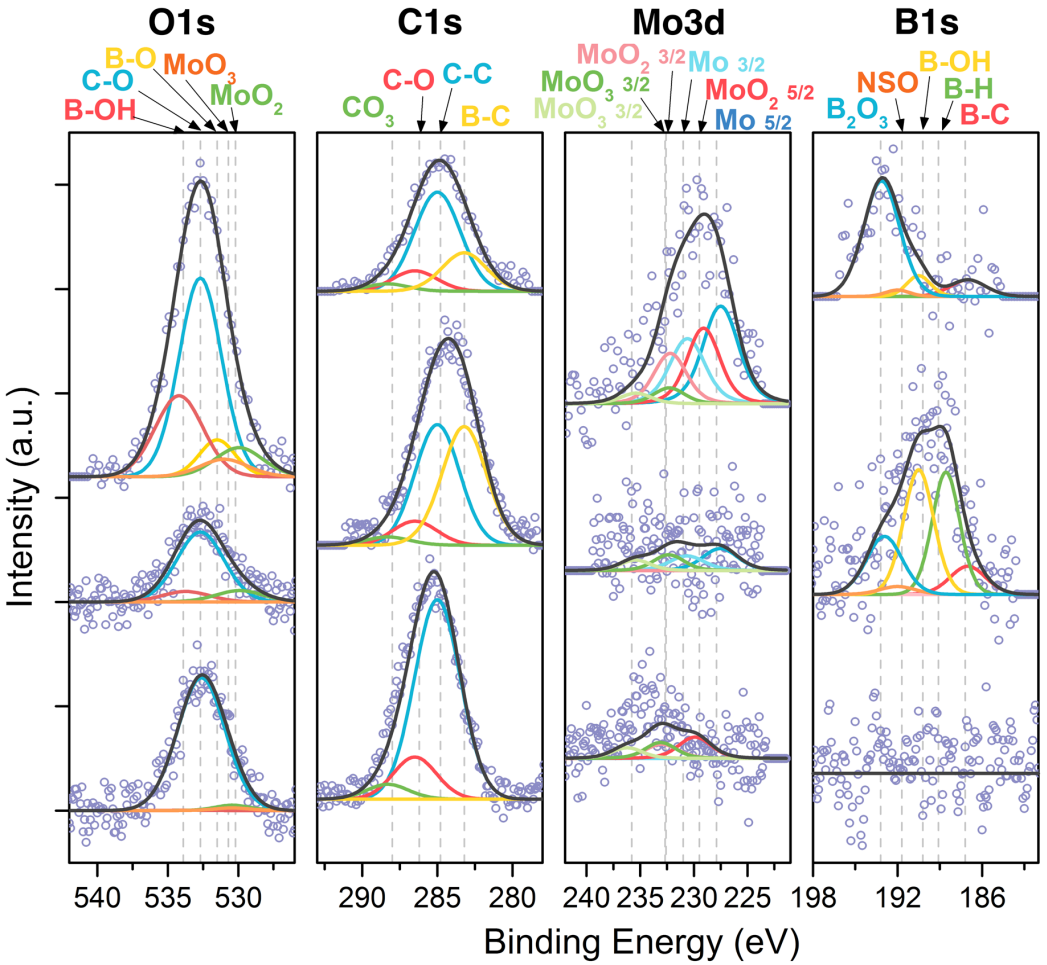


Coating (Boron): **o**
Bulk material (Gold): **B**

$$\frac{I_o}{s_o} = \frac{I_B}{s_B}\left[\exp\left(\frac{d}{\lambda_B cos\theta}\right) - \exp\left(\frac{d}{\lambda_B cos\theta}(\lambda_o - \lambda_B)\right)\right]$$

TZM analysis results

- XPS of TZM sample boronized and exposed to plasmas.
- Evidence of effective covering after boronization.
- Oxidation as in the ATJ case is also evident.
- Sputtering of B coatings is very likely.

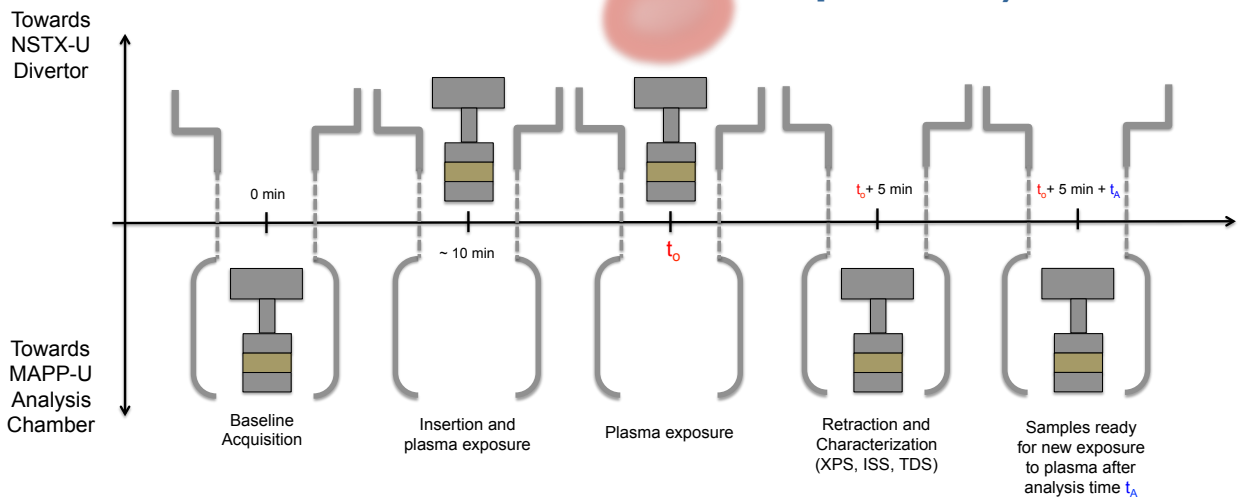


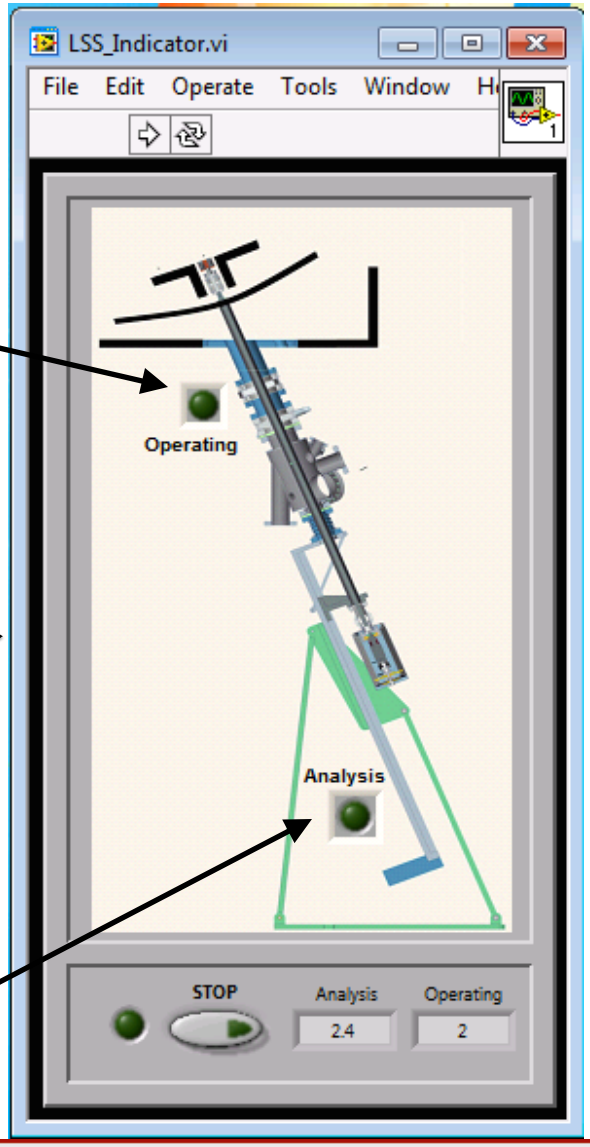
MAPP plans for FY2017 campaign

- Full remote operation (EPICS and LabView® Interface) now possible for in between plasma discharges measurements

Flush with LD tiles at bay J (boronization, lithiumization, D^+ plasma exposures)

Sequence of operation





XPS, ISS, DRS measurements

Conclusions

- Successfully able to characterize the chemistry of PFC in NSTX-U and their evolution.
- Oxidation has a relevant role behind the modifications on the PFC chemistry according to XPS.
- Results indicate that B coatings act as good gettering which could lead to a decrease in presence of impurities.
- B coatings ”quickly” saturate with oxygen (depending on power and exposure), this in addition to erosion might be the reason of temporal improvements
- TZM sample effectively boronized, plasmas were able to remove the coating in weeks time ~ hundreds of discharges
- According to XPS data, daily boronized deposited coatings at least 10 thinner than weekly boronizations