XPS study of depth profiling induced chemistry in NSTX-U tile samples

PRINCETON PLASMA PHYSICS LABORATORY

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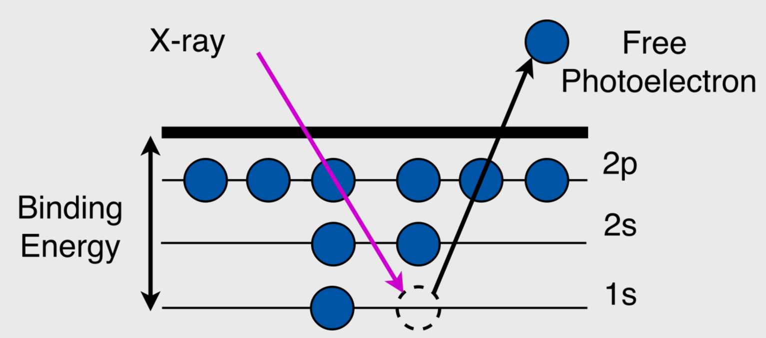
Motivation

Plasma performance is strongly influenced by the chemical state of plasma facing surfaces

X-Ray Photoelectron Spectroscopy (XPS) measures the atomic composition and chemical state of the top few nanometers of surfaces by measuring the energy of photoelectrons ejected by monochromatic X-ray radiation.

The electron binding energy shifts slightly depending on the chemical environment.



Binding Energy = X-Ray Energy − Ejected Electron Energy

Tile samples were removed from NSTX-U for investigation

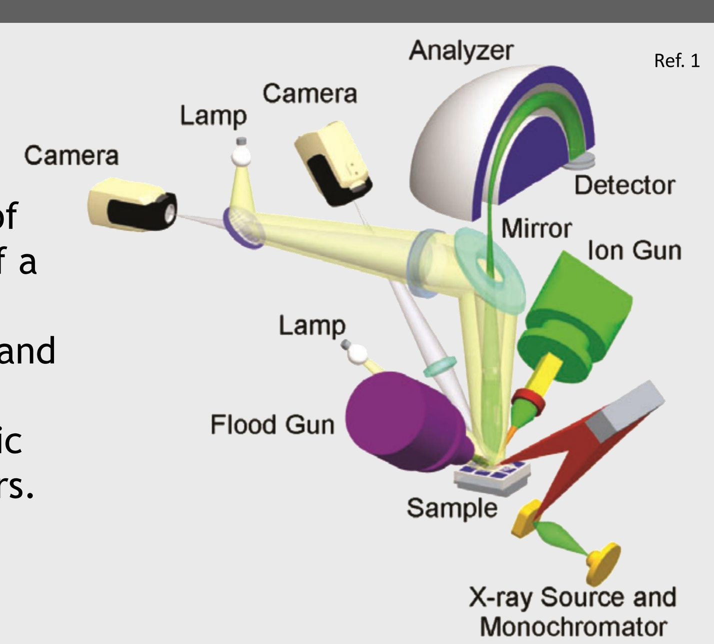
XPS beyond the first few nanometers are obtained by sputtering the surface with argon ions or gas clusters.

However, energetic argon ions can damage the sample and change the chemical state. Tantalum Pentoxide - Ta_2O_5, and a polymer (PEDOT:PSS) were used to evaluate this effect and calibrate the sputtering rate.

Experiment

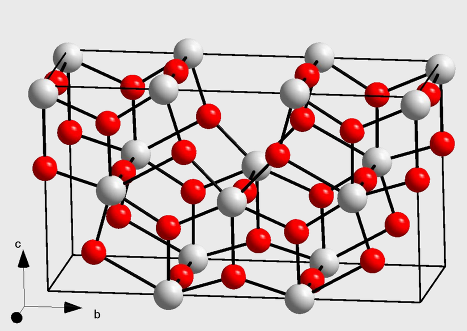
Thermo Scientific K-Alpha

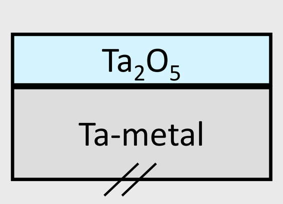
The Thermo Scientific K-Alpha XPS/UPS instrument is a state of the art XPS system consisting of a monochromatic X-Ray source, hemispherical energy analyzer and a MAGCIS dual-purpose ion gun capable of producing monatomic argon ions as well as gas clusters.



Tantalum Pentoxide

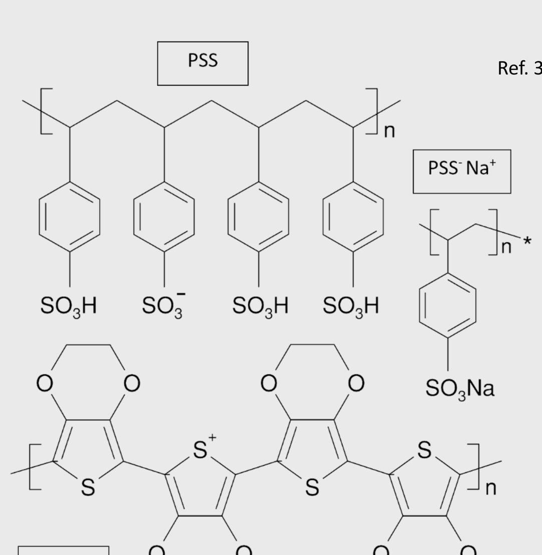
Tantalum pentoxide, or Ta_2O_5 is a sputtering standard. Commonly studied due to its optical and dielectric properties.

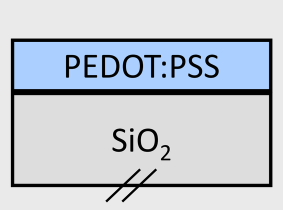




PEDOT:PSS

poly(3,4-ethylenedioxythiophene) polystyrene sulfonate is a conducting polymer. It was selected to mimic amorphous carbon-hydrogen co-deposits which occur in plasma-materials interactions.



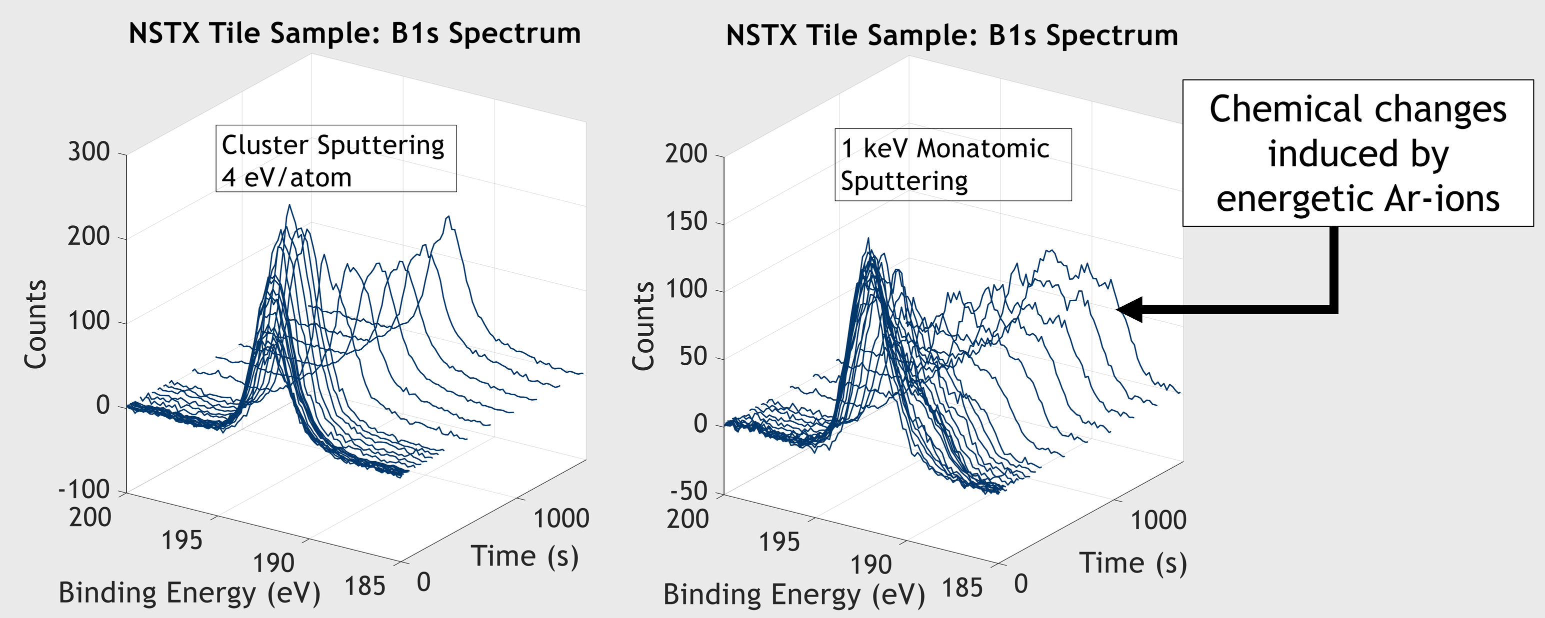


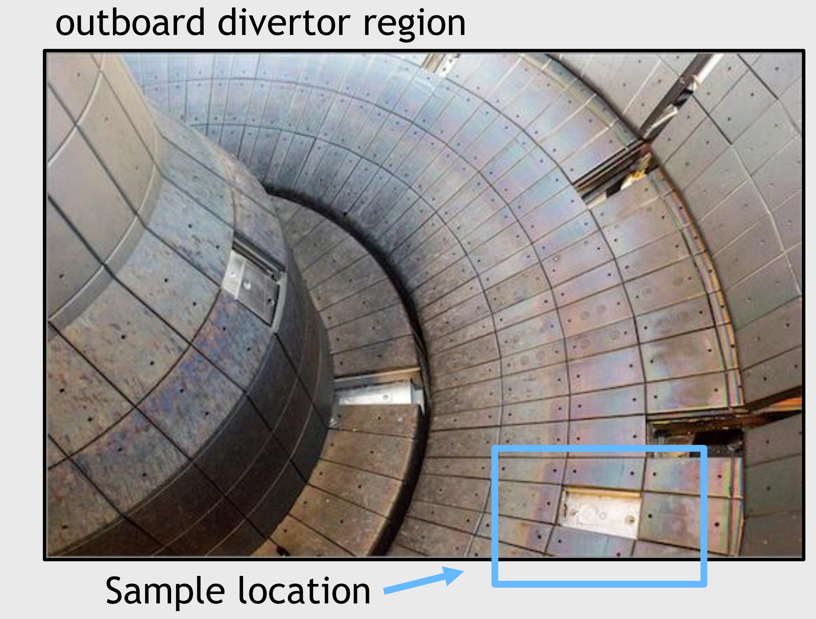
Results

Cluster Sputtering Tokamak Wall Sample Avoids Chemical Changes

During the 2015-2016 experimental campaign, boronization was used to condition PFCs (made of ATJ graphite).

Several tiles were removed from the NSTX-U tokamak to be analyzed with XPS and depth profiling to compare Ar ions with Ar clusters.

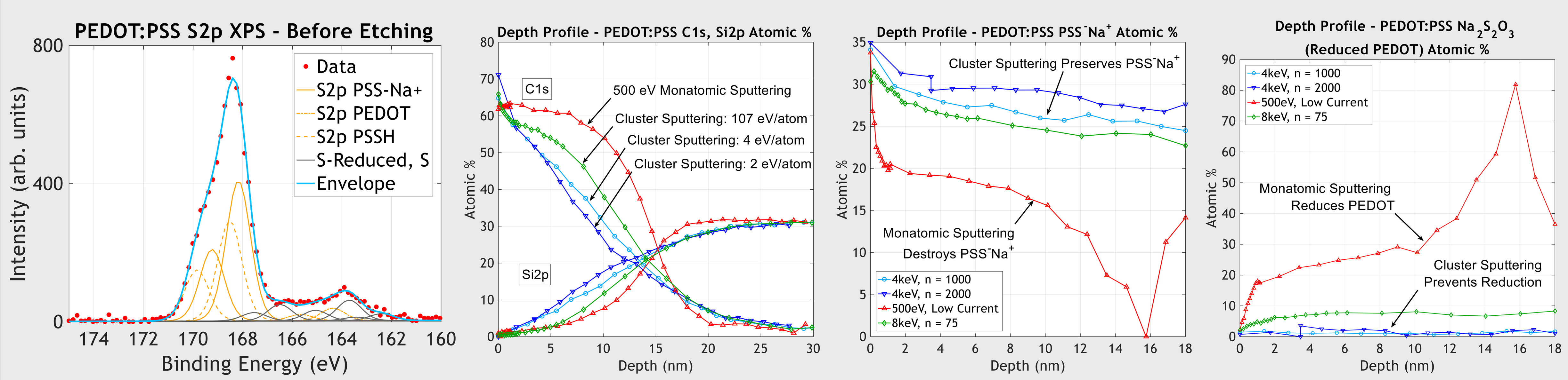




Monatomic Ar ion sputtering caused significant chemical changes in the B1s spectrum and created a significant shift of the XPS peak to lower binding energy. Argon cluster sputtering mitigates this artifact.

Sputtering Causes Chemical Modifications in a Polymer

The polymer PEDOT:PSS experiences major chemical modifications during monatomic argon ion sputtering, and less severe, although still present, modifications using an argon gas cluster beam.



XPS captures the evolution of the chemical components of the polymer. Argon ion have a higher sputtering rate than gas clusters but degrade the polymer's chemistry. Typical chemical modifications include reducing several functional groups to lower binding energy configurations.

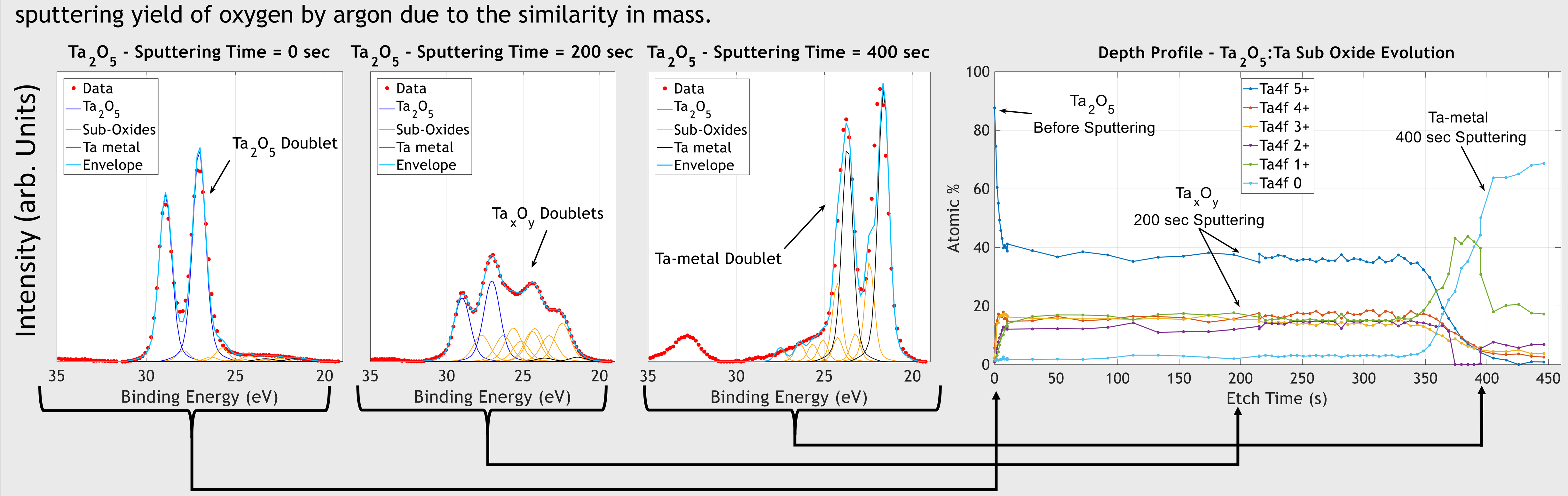
Cluster Energy (eV)	Cluster Size	Sample Current (nA)	eV/atom	Sputter Rate (nm/sec)
8000	2000	10.14	4.0	0.016*
8000	500	25.19	16.0	0.150
8000	75	40.15	106.7	0.198
4000	2000	7.56	2.0	0.017
4000	1000	20.43	4.0	0.027

*By rotating the sample while sputtering, 8keV / 2000 atom clusters sputtered at a rate of 0.019 nm/sec

Sputtering with rotation also improves apparent interface 'sharpness'.

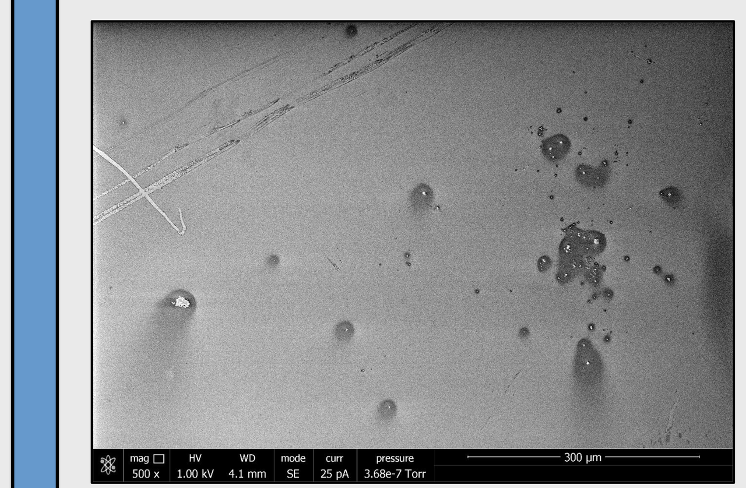
Sputtering Creates Reduced Oxidation States in Metal-Oxides

When sputtering tantalum pentoxide *preferential sputtering* of oxygen creates reduced oxidation states. This effect is attributed to a higher sputtering yield of oxygen by argon due to the similarity in mass.



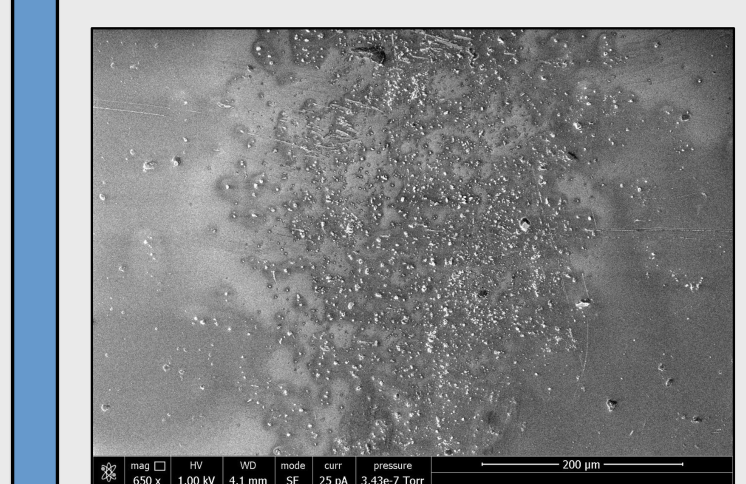
Surface Morphology

Different sputtering conditions appear to affect the sample surface differently



Cluster sputtering using settings of 8keV / 2000 atoms.

SEM image of the bottom of a crater while **rotating** the polymer sample azimuthally.



Cluster sputtering using settings of 8keV / 2000 atoms.

SEM image of the bottom of a crater without rotating the polymer sample.

Images captured with Verios 460 XHR SEM, 1keV electrons with 25 pA of current. Image levels adjusted to clarify features.

Conclusion

- Sputtering with monatomic argon ion beams strongly modified the surface chemistry of NSTX-U samples.
- Sputtering with argon gas clusters greatly reduced the changes.
- Gas cluster sputtering of polymer films provided sputter time to depth calibration.
- The transition from polymer film to substrate showed a reduction of interface 'sharpness' with clusters.
- SEM revealed surface morphology is likely affected by sputtering conditions.

Acknowledgements

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