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Focused Ion Beam Time of Flight Mass Spectrometry

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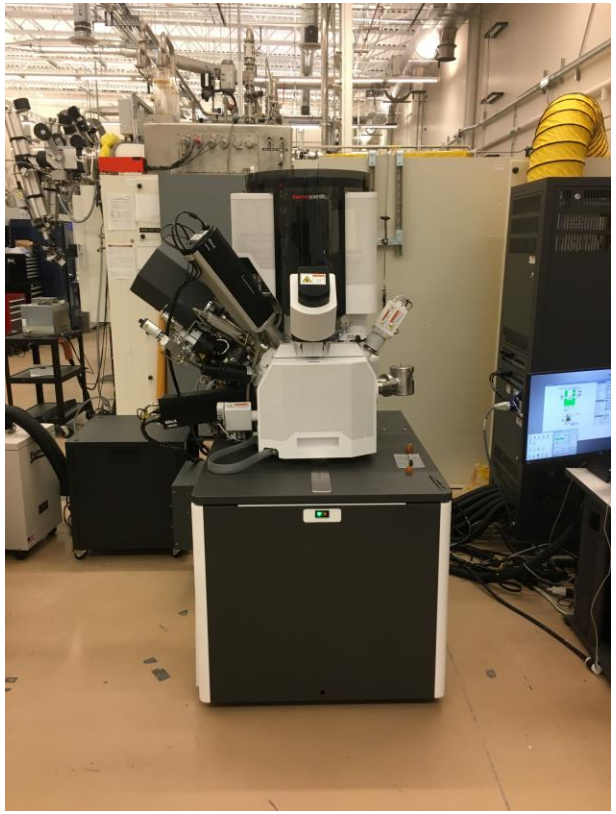
Battelle Energy Alliance manages INL for the
U.S. Department of Energy's Office of Nuclear Energy

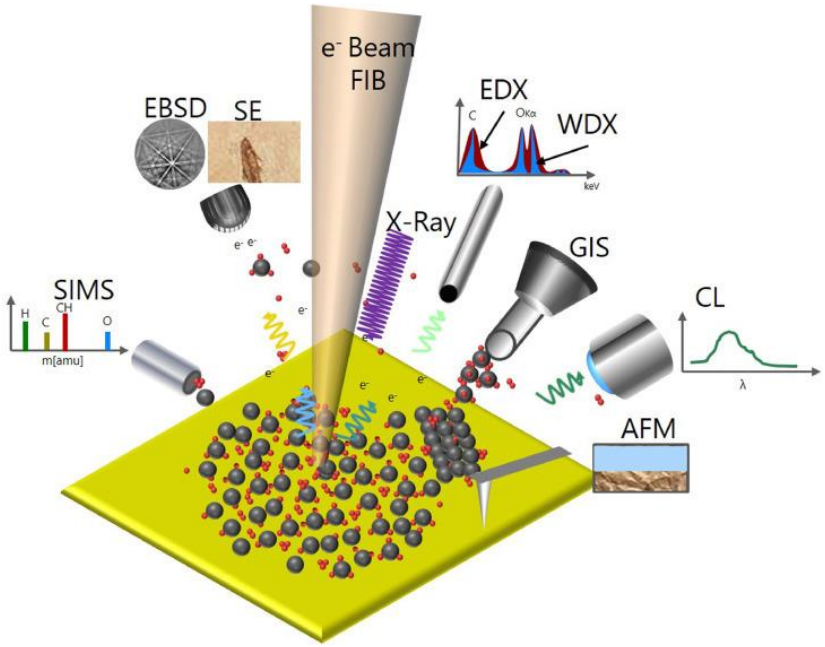
Idaho National Laboratory

Multi-ion Dual-Beam FIB/SEM



Available ions
Xe, Ar, N_2, O_2
Ion switching in >10 min

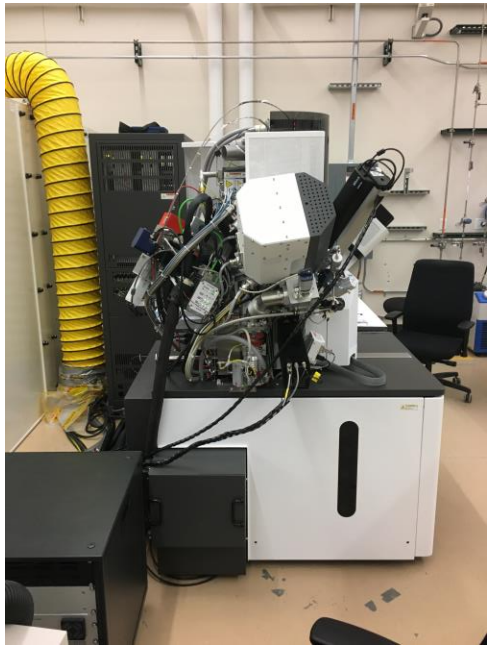
- Imaging
- Microanalysis
- Sample preparation

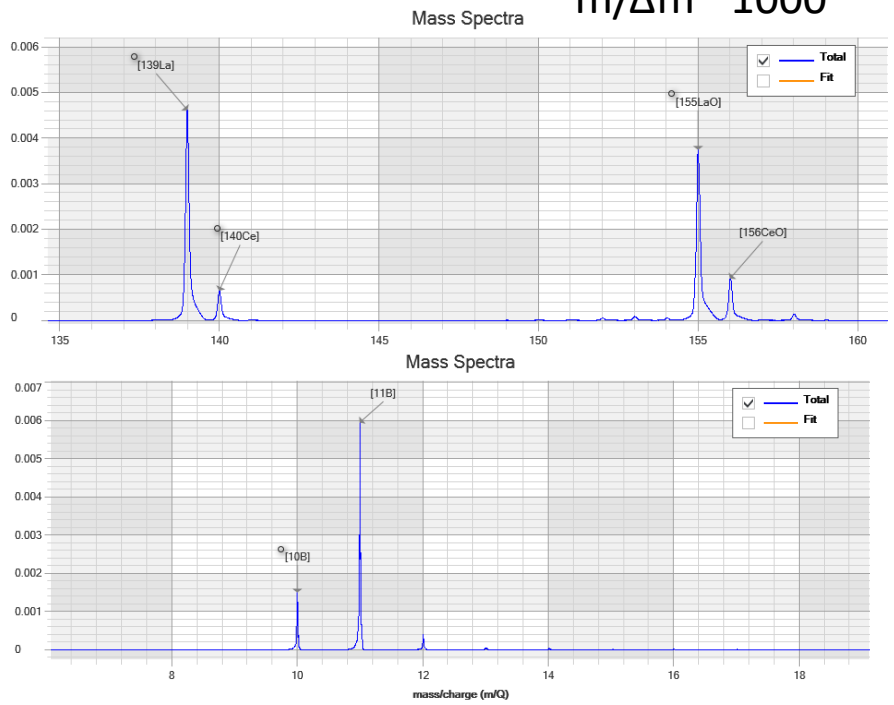


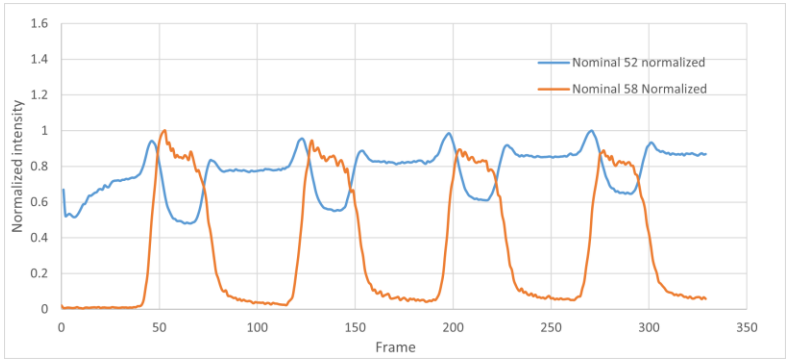
Progress in Crystal Growth and Characterization of Materials, 65, 1, 2019,1-19

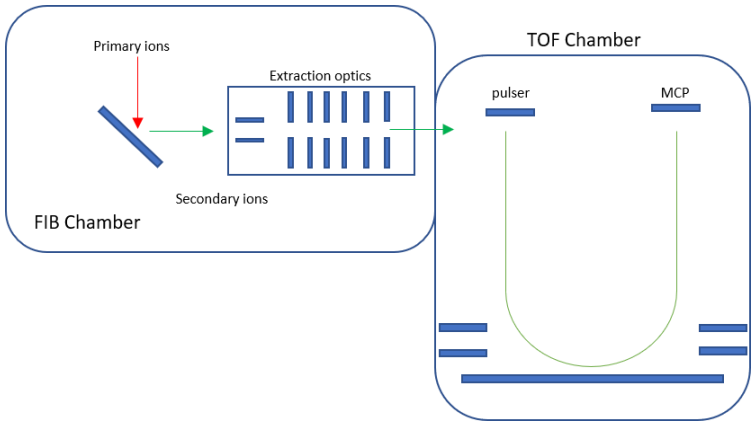
Time-of-Flight FIB-SIMS

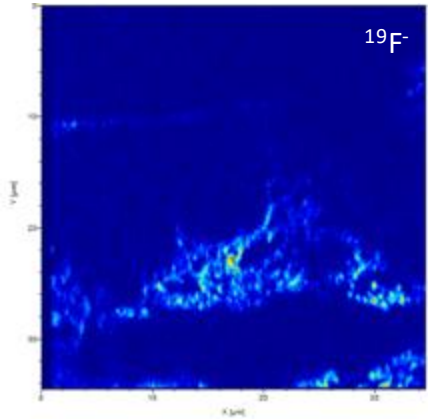
m/Δm ~1000







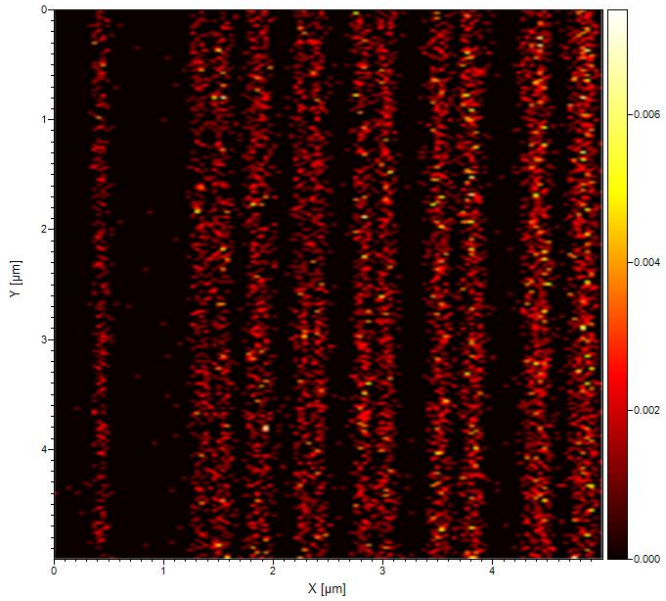


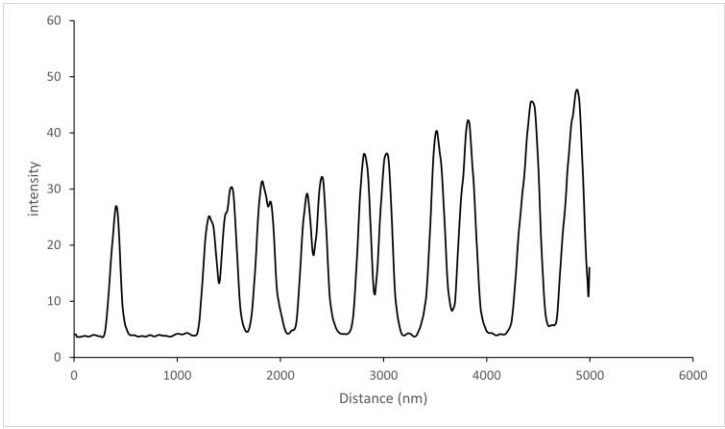


Ion flight path of 0.5 m

Comparison to EDS and EPMA

	EDS	WDS	SIMS
range of detectable elements	boron or heavier	boron or heavier	all elements
detection of isotopes	no	no	✔
detection limit	0.1-1 at.%	100-500 ppm	1 ppm (or lower)
quantification	✔ (semi)	✔	limited
lateral resolution	500-1000 nm	500-1000 nm	50 nm
depth resolution	1000 nm	1000 nm	>5 nm



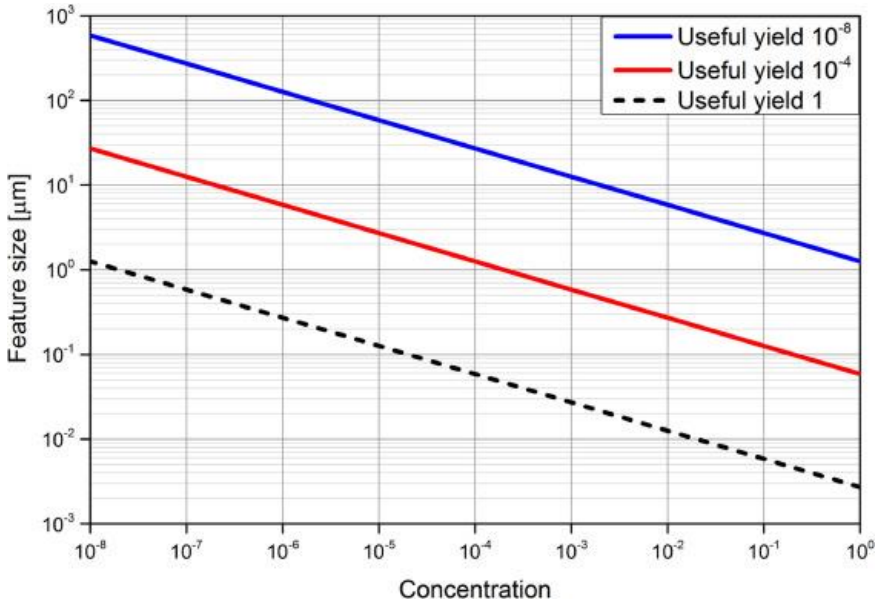


Lateral resolution
~50 nm

Useful Ion Yield

$$UY = \frac{Number\ of\ M\ ions\ detected}{Number\ of\ sputtered\ M\ atoms}$$

$$p_M = \frac{I(M^+)}{UY * V}$$



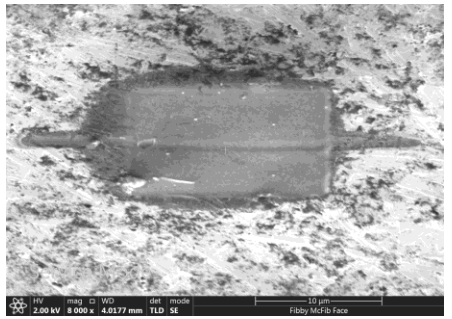
100*100*100nm sputter volume, a UY of 10^{-3}, and a minimum of 10 ions per pixel

$$p_m = 10^{19}\ \mathrm{at/cm^3}$$

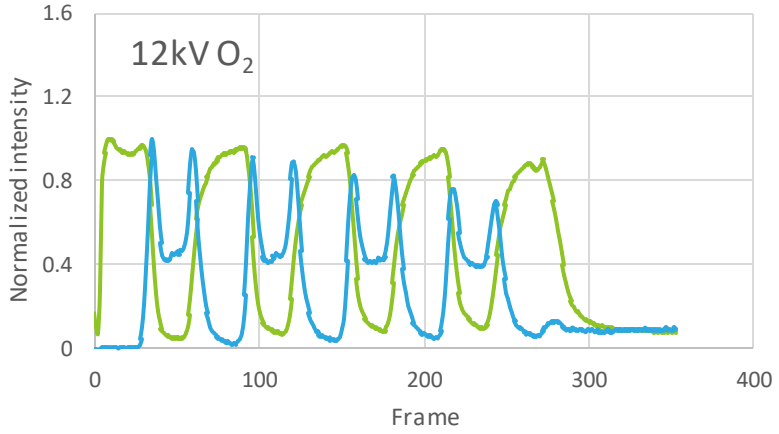
Benefits of Multi-ion plasma Column Ion- yield study for Al

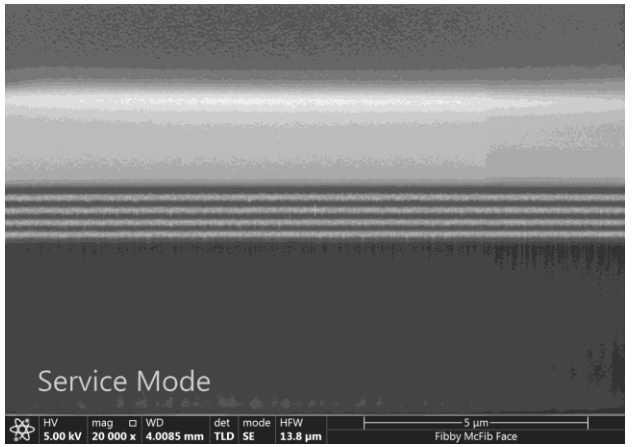
$$UY = \frac{total\ ion\ count}{total\ number\ of\ removed\ atoms}$$

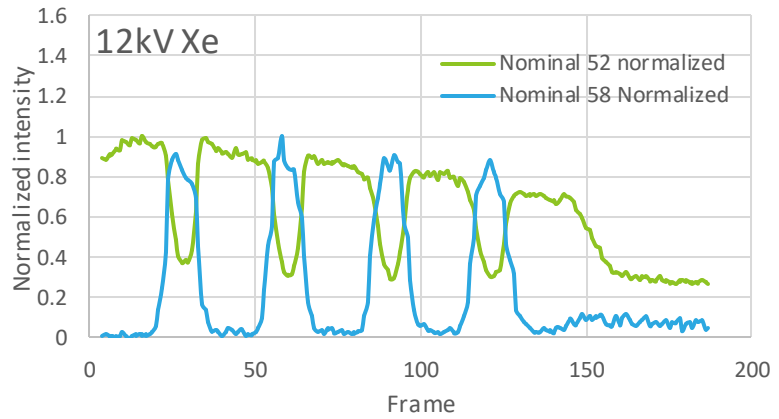
Primary ion	Useful yield
O_2^+	1.5E-04
N_2^+	5.3E-05
Xe^+	8.4E-06
Ar^+	3.6E-06

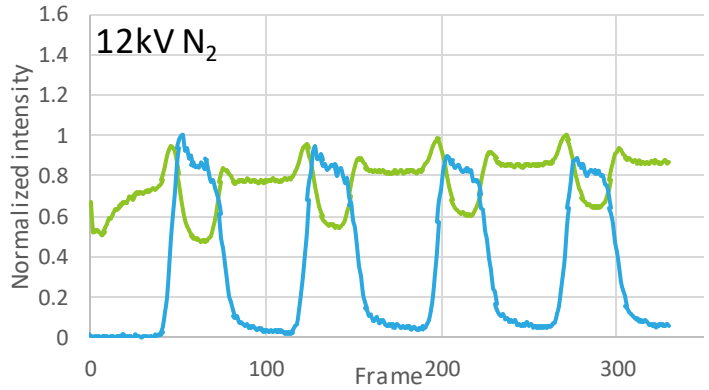


Examples – Depth profiling



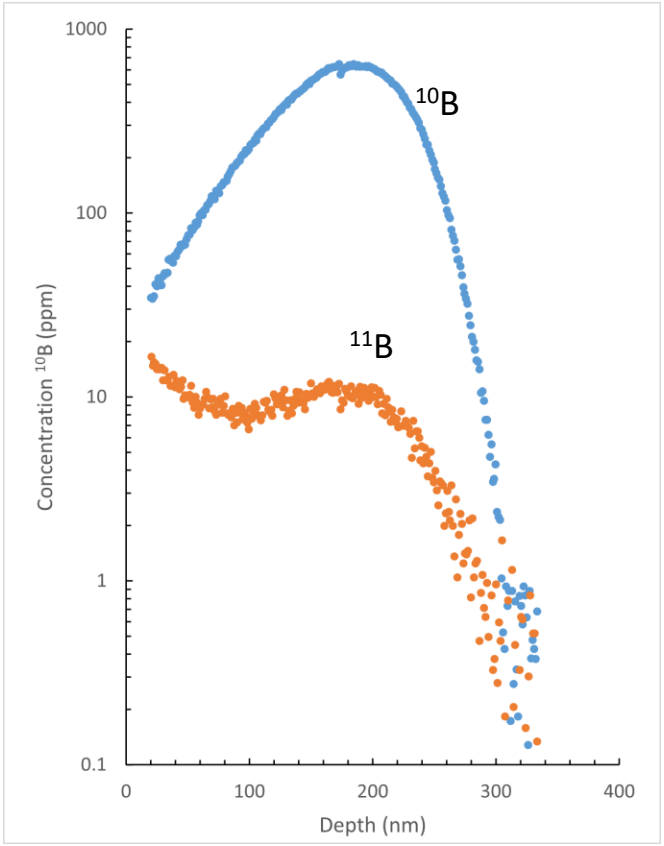






Depth profiling Ion implanted Materials

O_2^+ primary ion, 5 kV,
52° angle of incidence

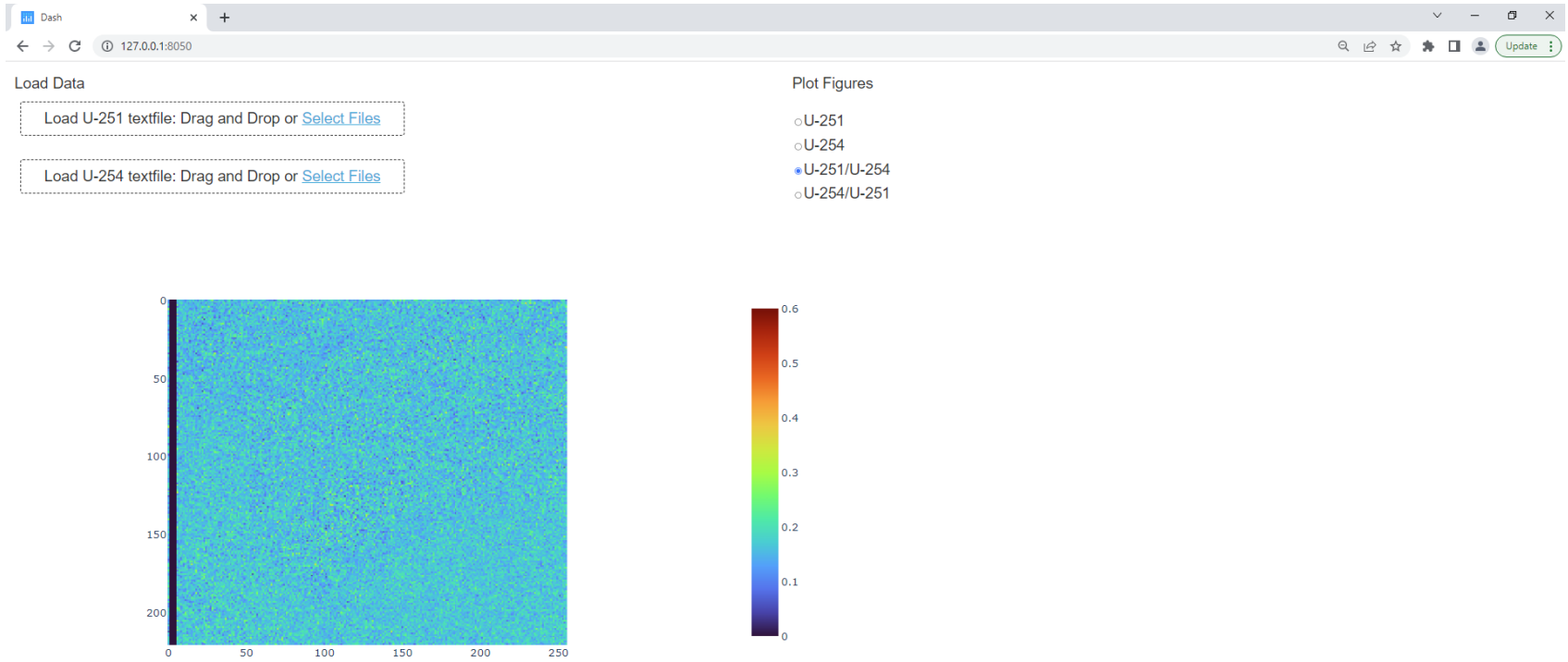


- Indicates sensitivity is ~1 ppm under these conditions

Isotope ratio Imaging

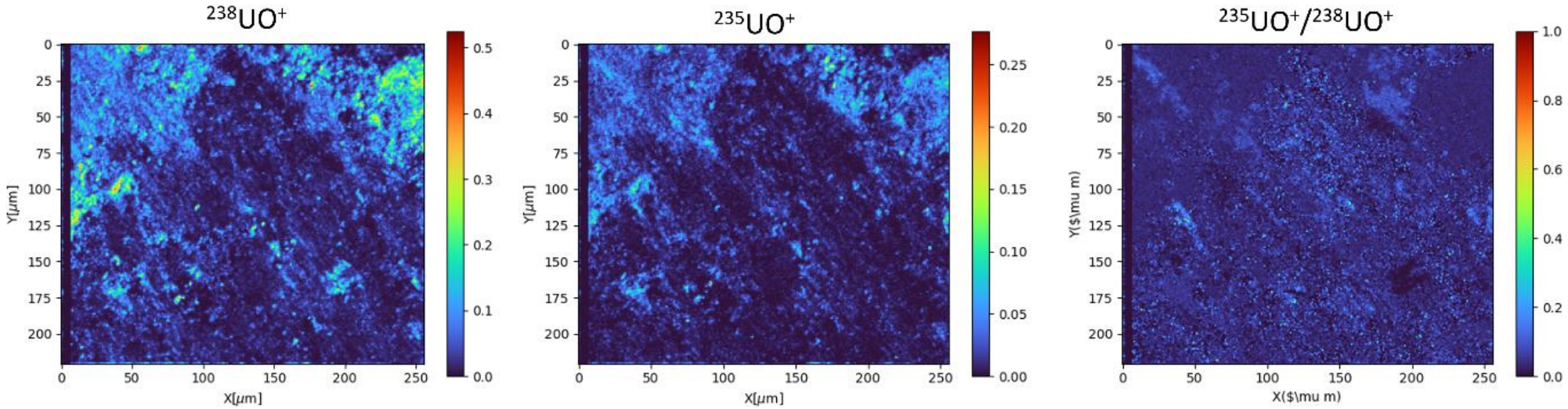
- Nuclear applications of SIMS often benefit from isotope ratio determination
  - Burn up
  - Fission product analysis and distribution
- Typical isotope ratio determinations in irradiated nuclear fuel are bulk methods

Development of Isotope Ratio imaging for FIB-SIMS



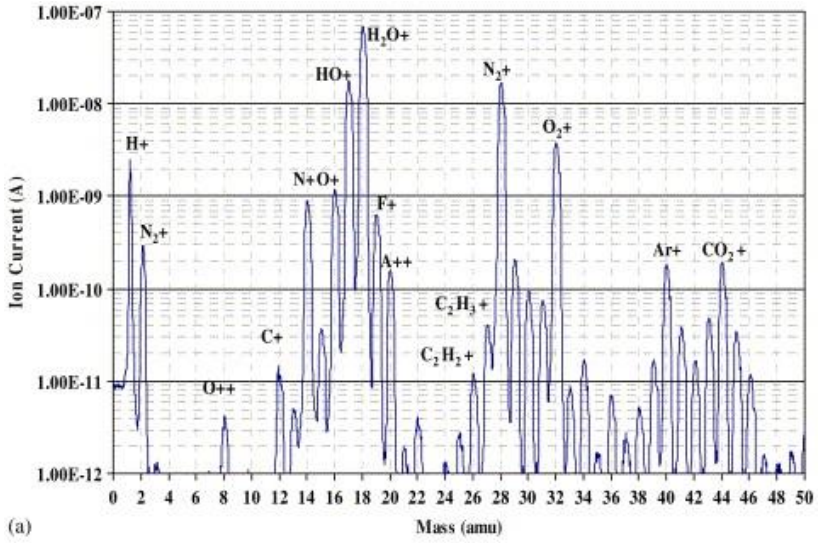
- Developed an easy to use GUI tool for generating isotope ratio images
- Available to all users of the TOF-SIMS
- Have added pre and post processing functions to enhance usefulness of the tool

Development of Isotope Ratio imaging



Reduction of Molecular Hydride Formation

- Formation of molecular hydrides occurs during the sputtering process
- Molecular hydrides complicate the mass spectrum and present isobaric interferences.
- Make it so extremely high Mass resolving power is necessary.
  - $^{238}UH^{+}$ from $^{239}Pu^{+}$ is **<u>26385</u>** M/ΔM



Residual gas analysis in the TOF vacuum system, 78, 1, 83-89

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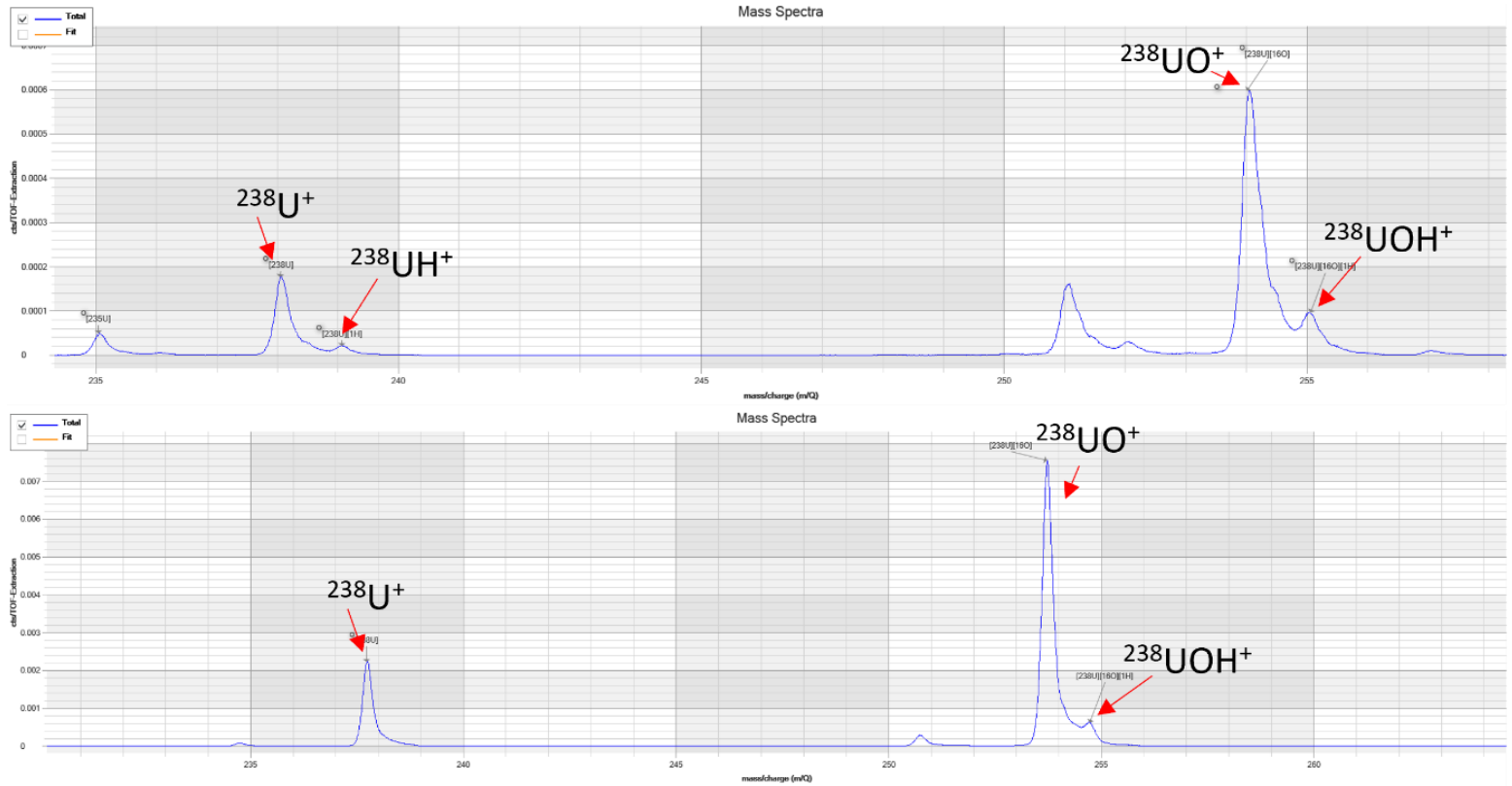
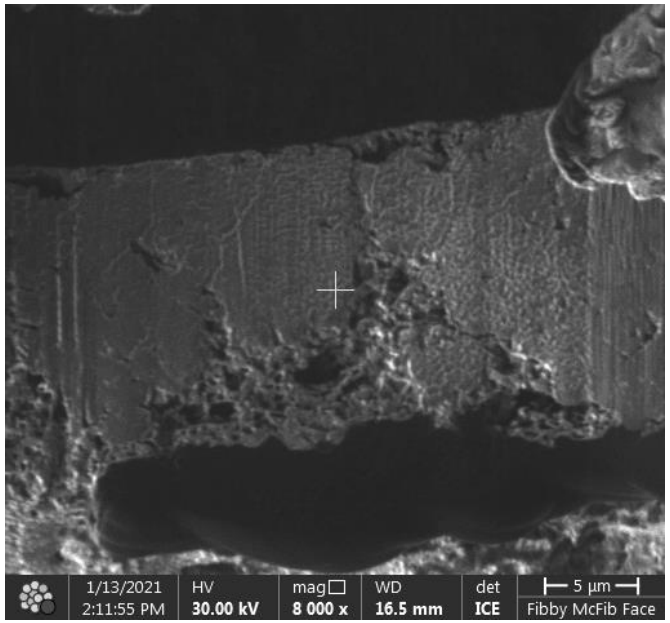
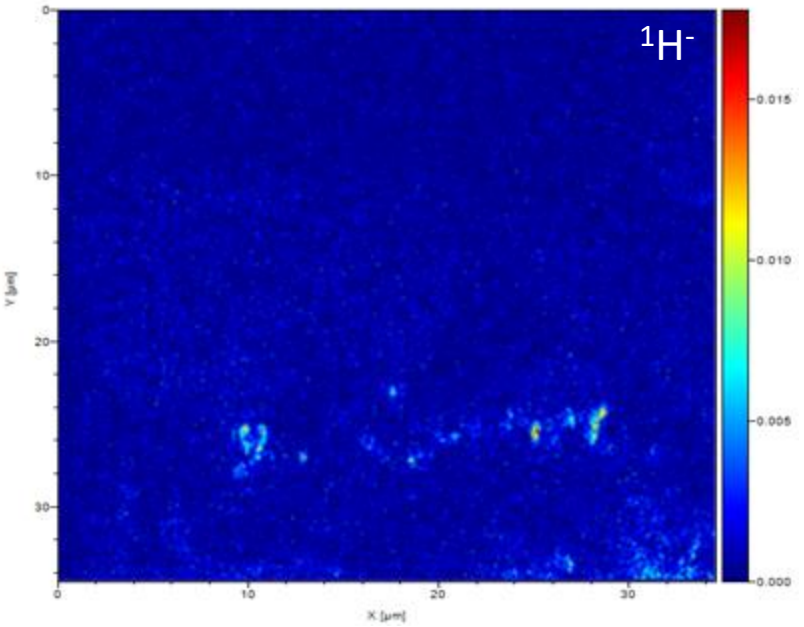


Hydrogen Imaging

30 kV Xe primary ions





- Limited chamber vacuum (10^{-5} pa) make hydrogen detection difficult

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