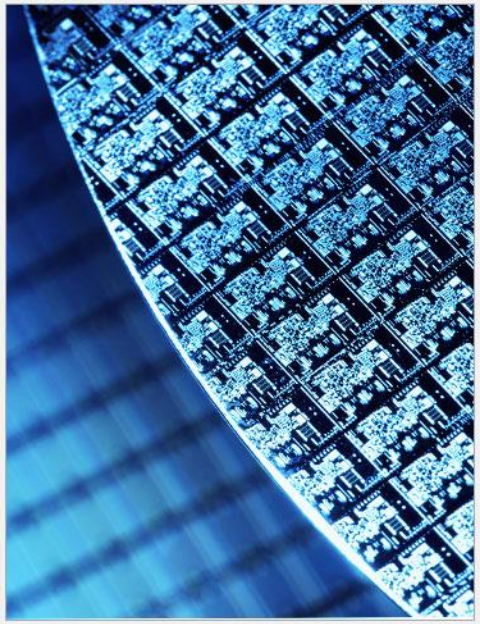
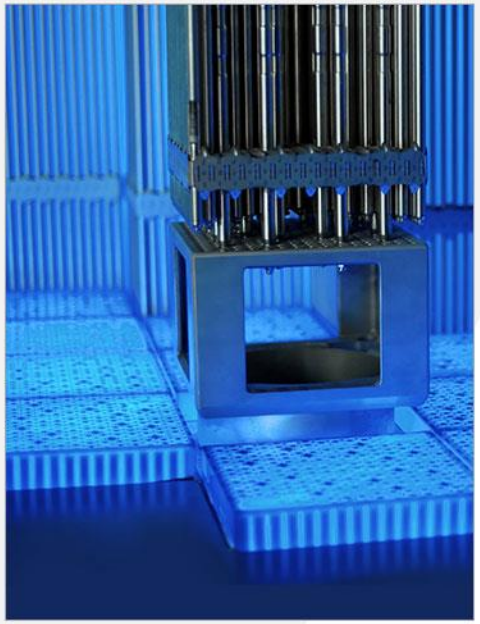
CAMECA
SCIENCE & METROLOGY SOLUTIONS
AMETEK


DEVELOPMENTS IN ATOM PROBE TOMOGRAPHY

JUNE 2022

DAVID J. LARSON

ACKNOWLEDGEMENTS

LEAP Engineering Team

- D. Lenz, J. Bunton, T. Payne, E. Oltman, B. Geiser, E. Strennen, D. Rauls, J. Shepard, J. Mandt, G. Groth

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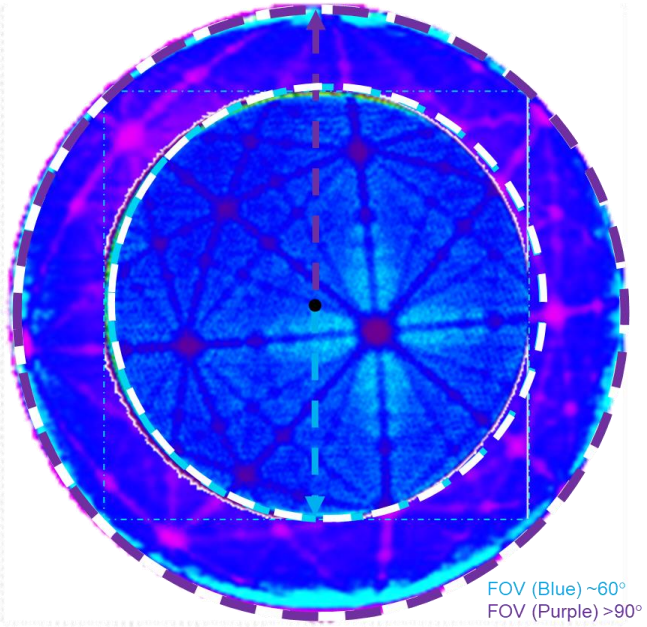
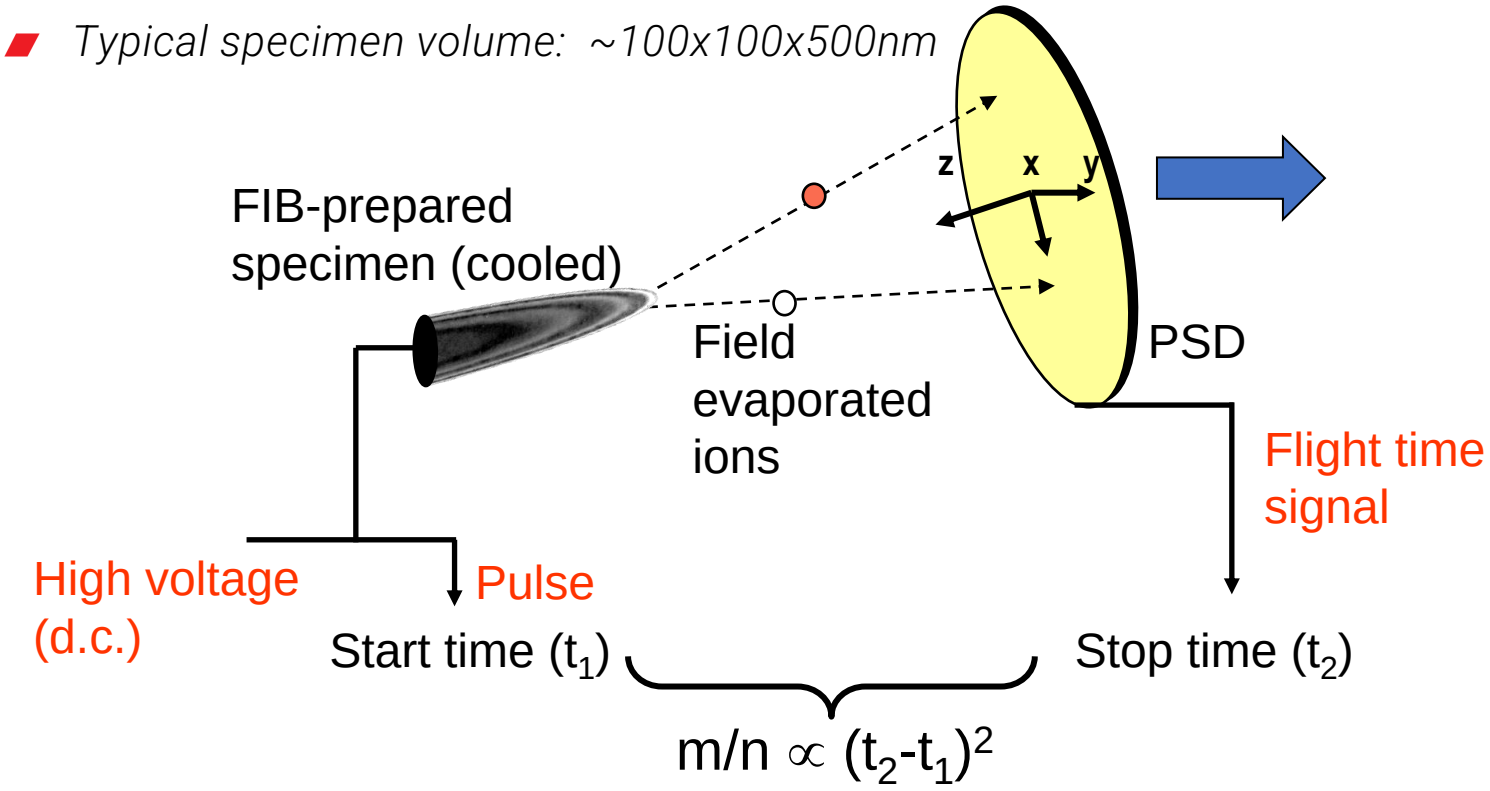


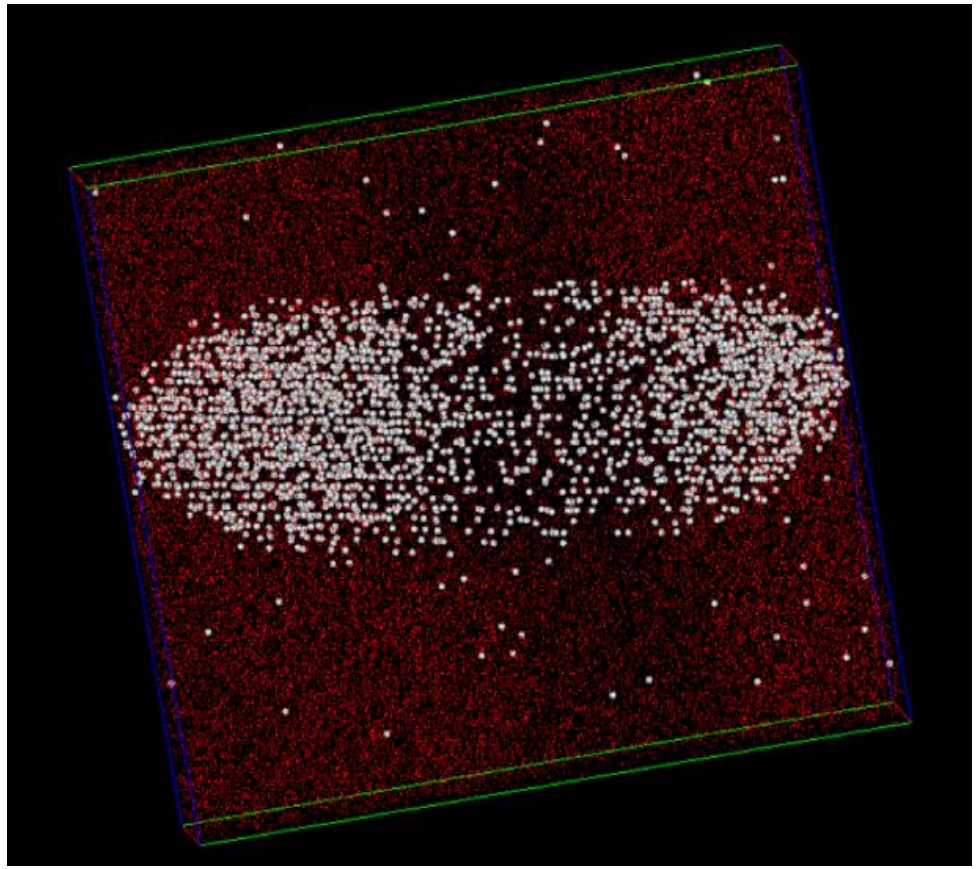
OUTLINE

- Brief introduction to atom probe…
- CAMECA has recently launched two new atom probes in the 6000 product line
- Invizo 6000 features and benefits
- LEAP 6000 XR features and benefits
- External research collaborations

ATOM PROBE TOMOGRAPHY PRINCIPLES OF OPERATION

- *Uses electrostatic field evaporation*
- *Position-sensitive single-ion detector*
- *Time-of-flight mass spectrometer*
  - ***Voltage (Field) pulsing [e-conductive materials only]***
  - ***Laser (thermal) pulsing***
- *Typical specimen volume: ~100x100x500nm*





Schematic courtesy A. Cerezo (Univ. Oxford)

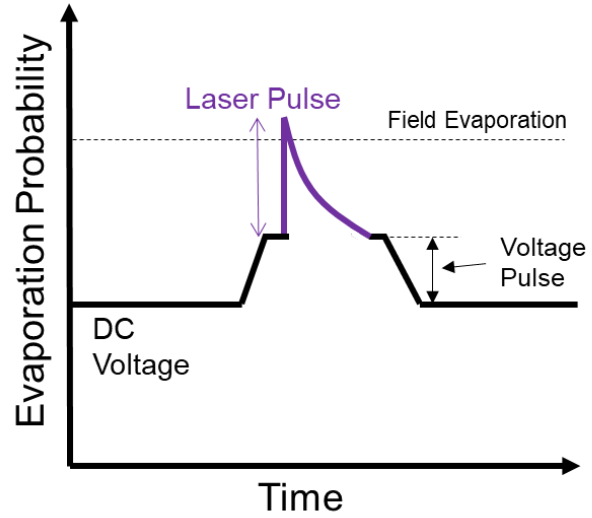
INTRODUCTION TO THE LEAP 6000 XR™

Goals of the design 6000 XR:

- High-performance reflectron design
- Continues local electrode compatible LEAP platform in research and near-FAB applications
- Improving the throughput and ease-of-use through fully automated laser-specimen-electrode alignment
- Improve detection sensitivity
- Improve both specimen yield and data quality

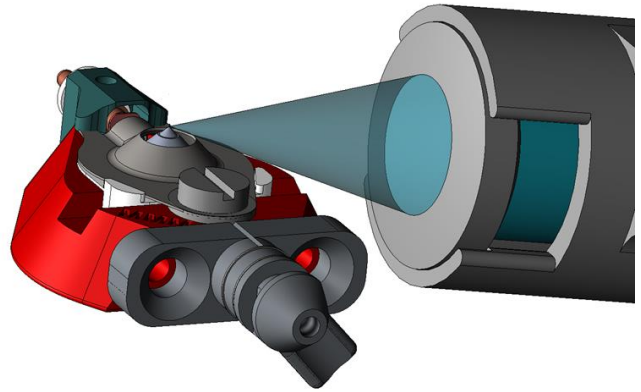
LEAP 6000 XR technology to achieve goals:

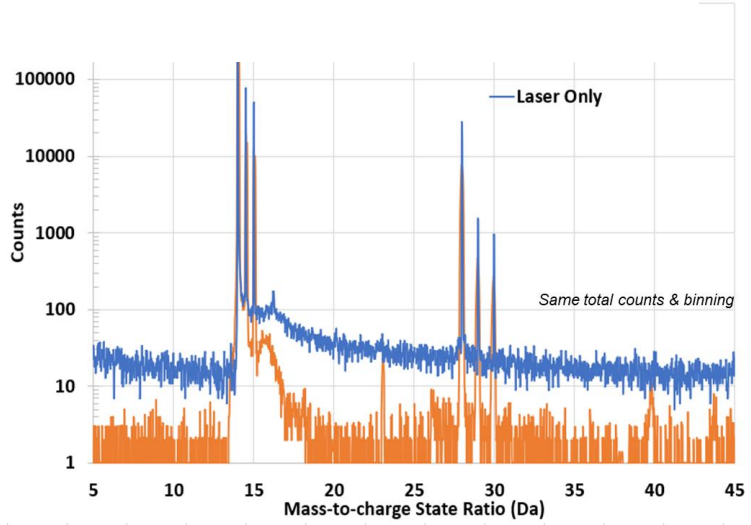
- Redesign the optical system incorporating new laser source
- Multi-mode pulsing operation (simultaneous voltage plus laser pulse)
- Deep UV (257.5 nm) laser with ~1µm spot size



KEY BENEFITS OF THE LEAP 6000 XR™

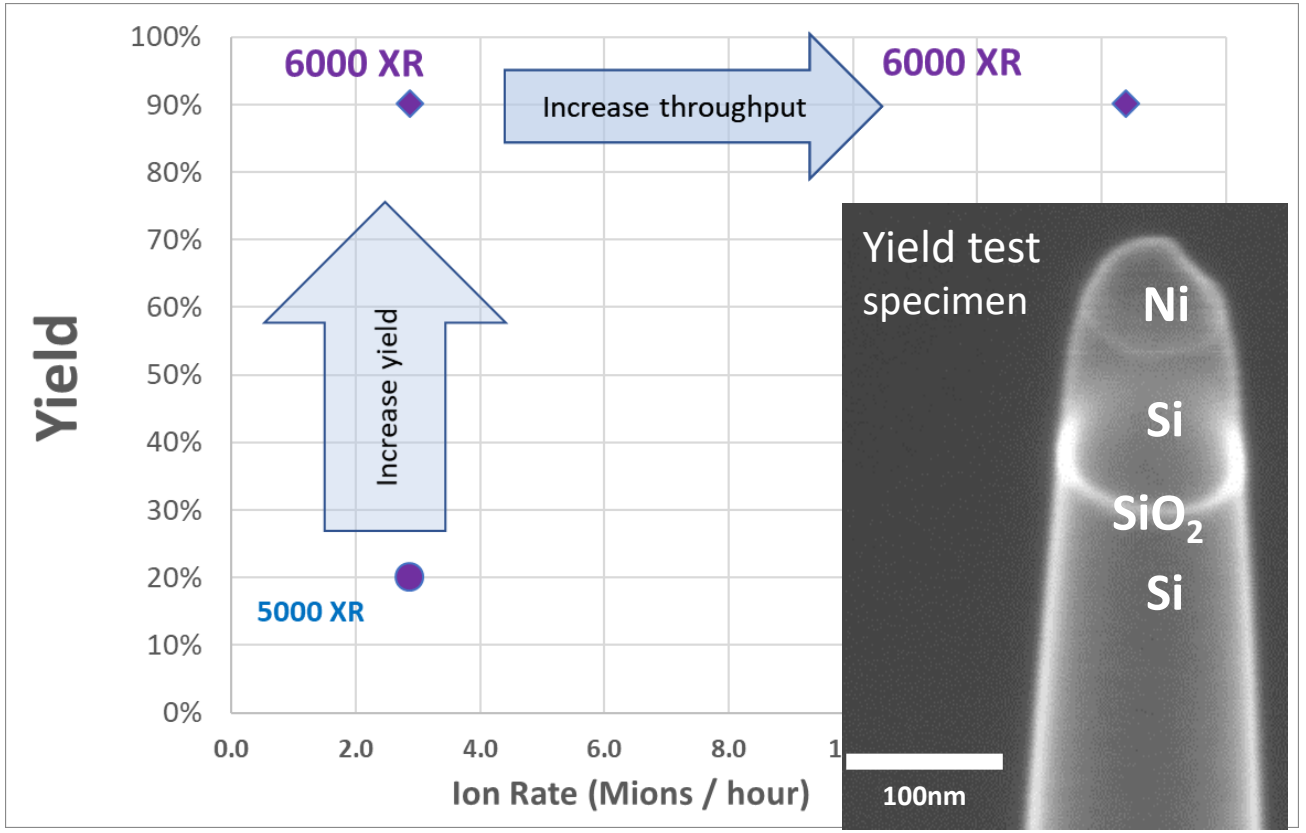
- Continuity in the platform including local electrodes and microtips
- Increased automation provides complete laser-specimen-electrode alignments, no manual operation required after inserting the specimen and local electrode
- Enhanced specimen yield (for some materials systems) due to the DUV laser
- Improved reconstruction (more uniform specimen shape) through photon absorption uniformity using DUV laser
- Improvement in detection sensitivity achieved through a lower mass spectral background





LEAP 6000 – DUV IMPROVES YIELD AND/OR THROUGHPUT

- Specimen yield through a silicon/ SiO_2 /silicon structure (yield test specimen) is improved with DUV
- The experiments to the right show:
  - If you have a specimen with low UV yield (20%), it improves to 90% with DUV (same laser energy)
  - If you initially have a specimen with good yield, you can increase throughput by 5X running faster with DUV

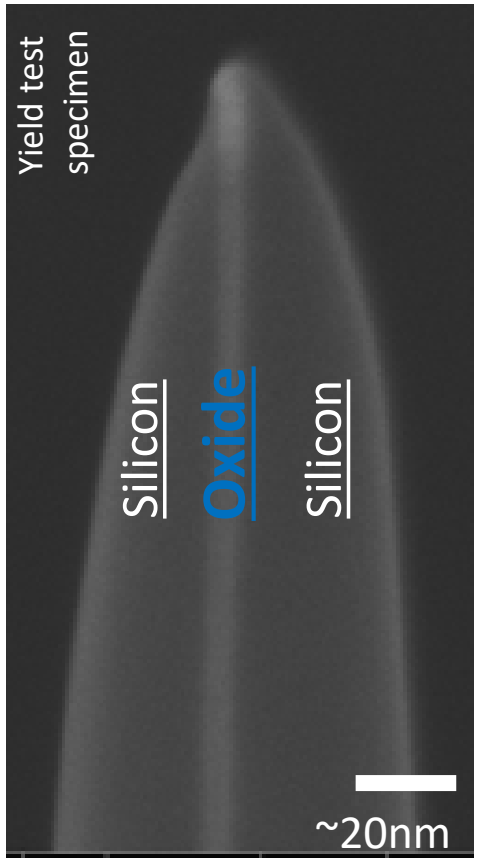


Prosa, T. J. et al. Microscopy and Microanalysis (2015).

LEAP 6000 – DUV IMPROVES RECONSTRUCTION

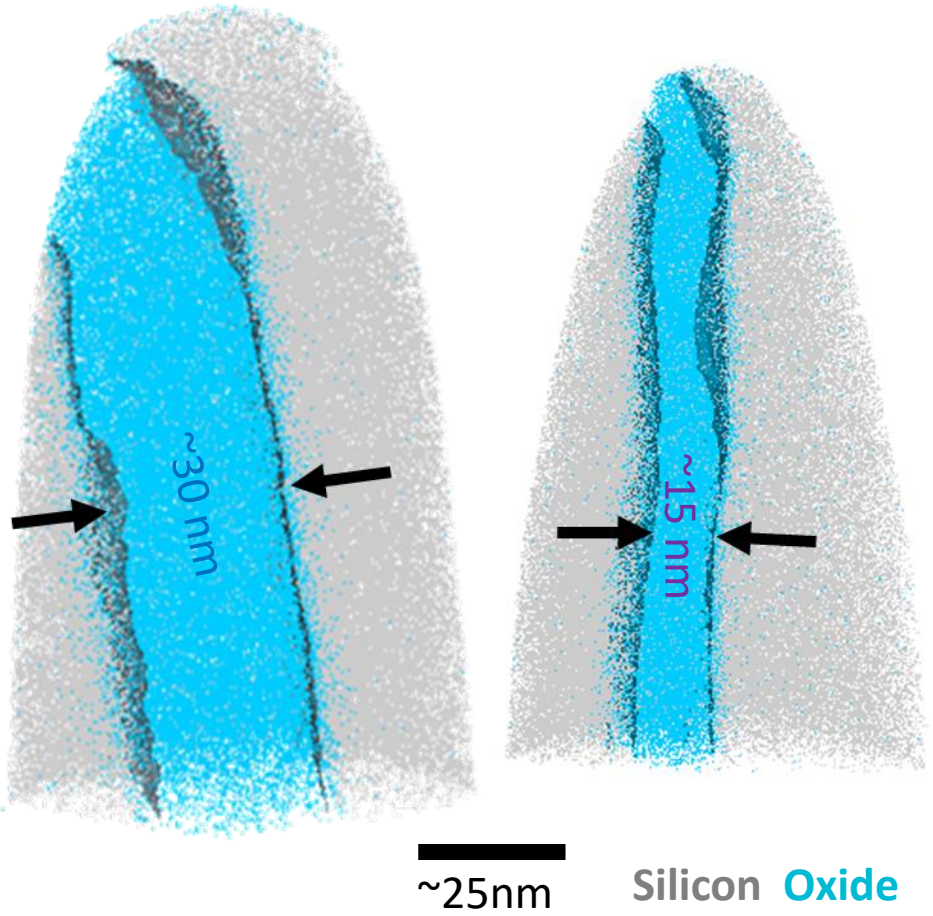
- Cross-section-oriented Si/ SiOx(12nm) / Si yield specimen
- A default reconstruction was done by using an average initial radius measurement observed from SEM images
- Oxide is a higher field material, resultant non hemispherical end form results in nonuniform magnification
- Measured ~30 nm for UV
- Measured ~15 nm for DUV
- <u>For this system:</u> DUV use results in more even evaporation endform and a more accurate reconstruction

<u>SEM Image</u>
After FIB

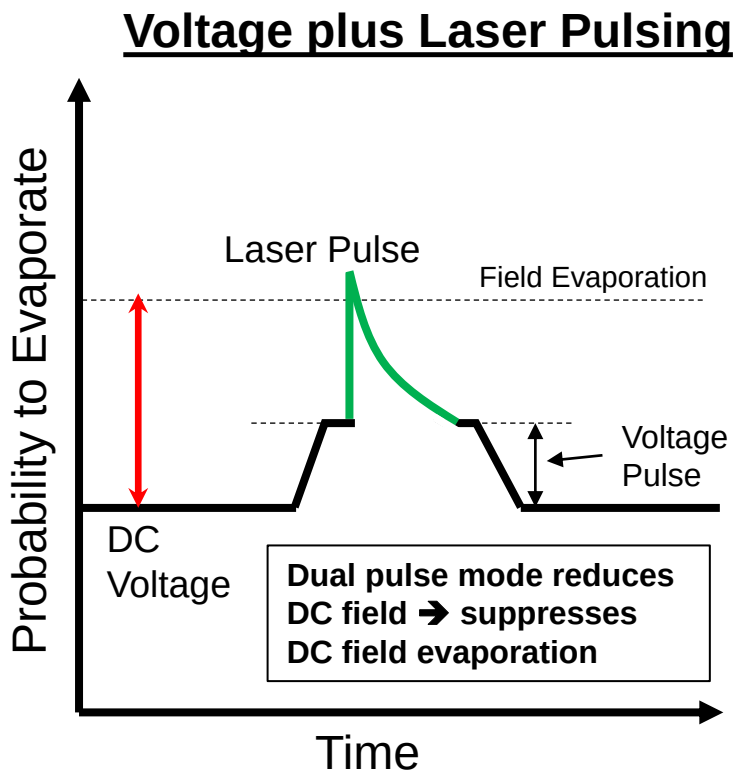


<u>5000 UV laser</u>
Default Recon

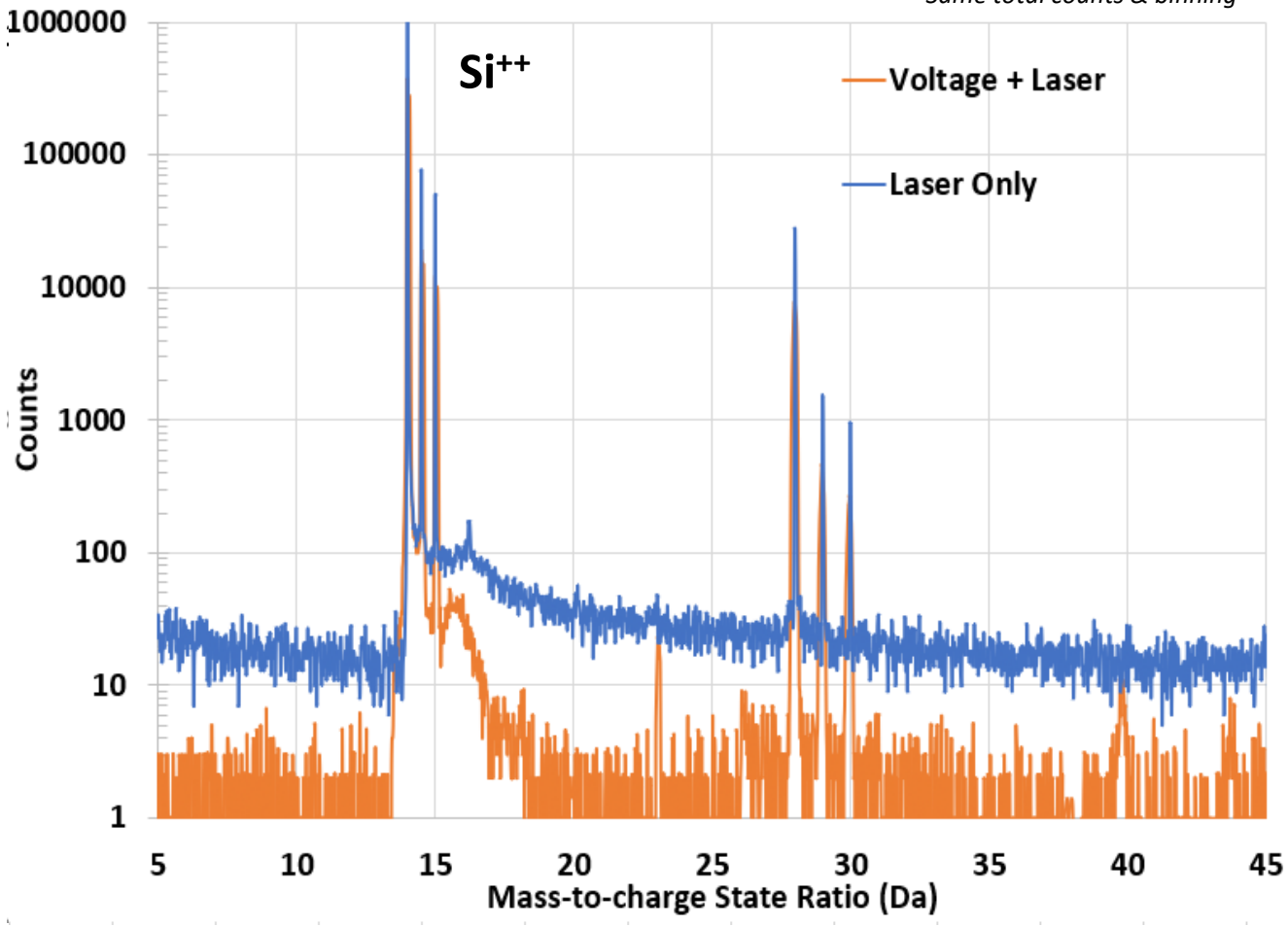
<u>6000 DUV laser</u>
Default Recon



LEAP 6000 MULTI-MODAL PULSING IMPROVES SIGNAL-TO-NOISE*



Same total counts & binning



- Feasibility of novel voltage plus laser pulsing system to improve SNR has been shown
- Preliminary results indicate method is effective for range of materials - benefit up to ~10x
- Supply chain / vendor issues impacting development

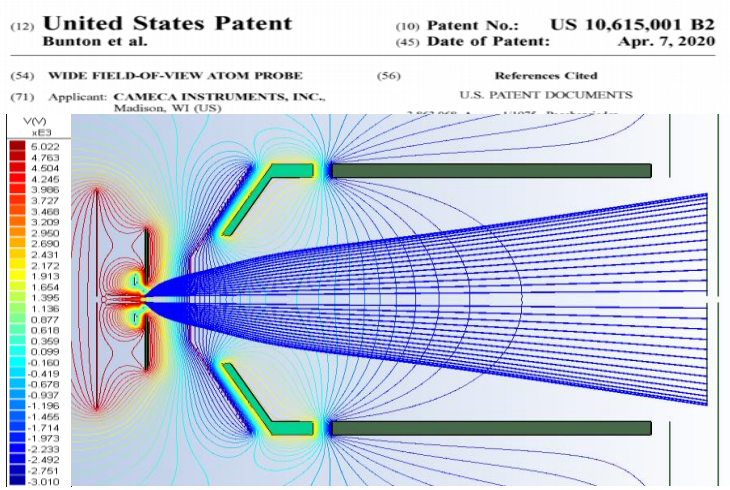
Based on T. F. Kelly, Micro. Microanal. (2011)
Also see D. J. Larson et al., Micro. Microanal. (2022)

* *Depends on materials and laser energy*

INTRODUCTION TO THE INVIZO 6000™

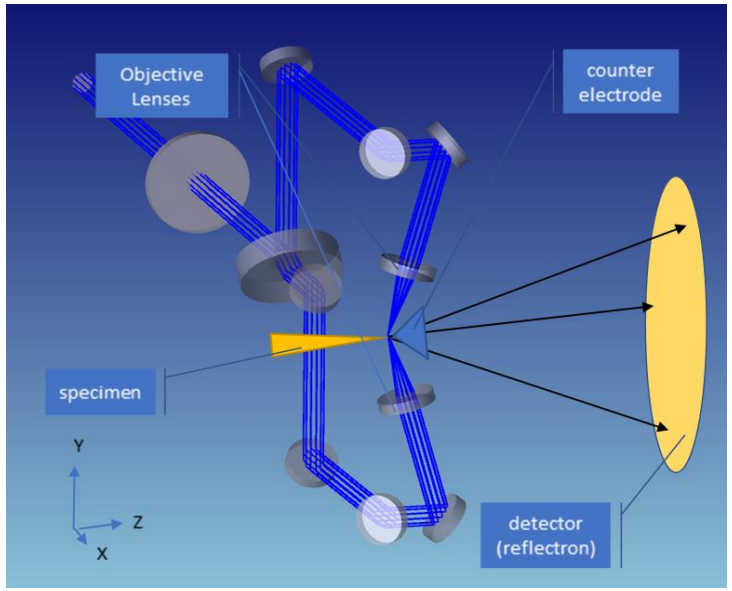
Goals of the Invizo design:

- Improve both specimen yield and data quality
- Provide substantially increased analysis volumes for increased understanding of materials and enhanced analytical capability
- Improve reconstruction



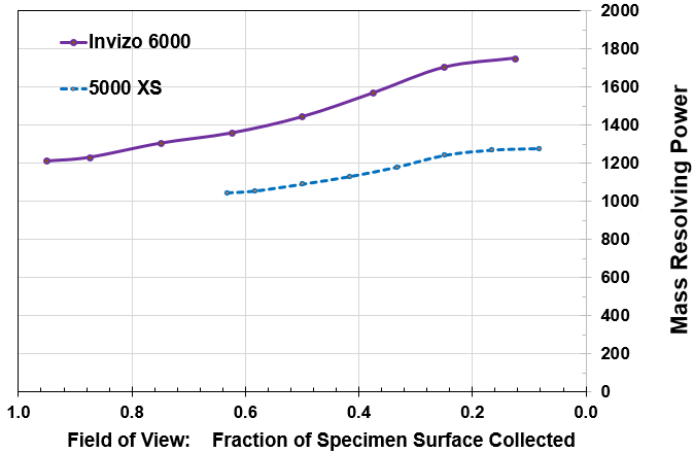
Invizo technology to achieve goals:

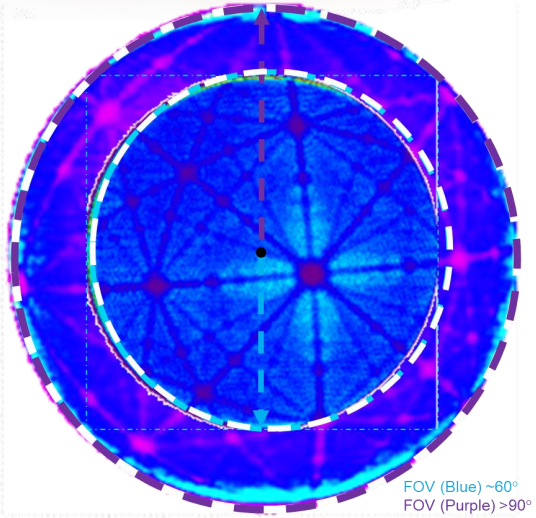
- Patented double Einzel lens design enables increased field of view AND enhanced mass resolving power to be achieved simultaneously
- Deep UV (257.5 nm) laser with ~1µm spot size
- Advanced beam delivery optics developed to enable symmetric specimen illumination with two beams



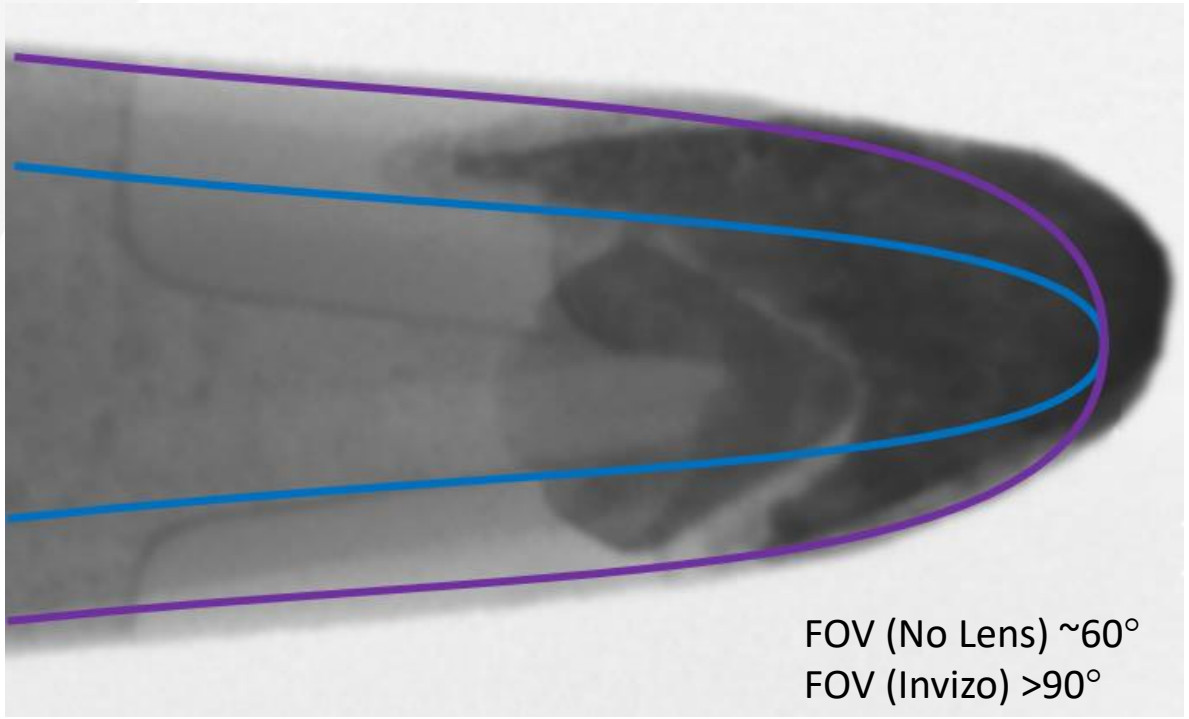
KEY BENEFITS OF THE INVIZO 6000

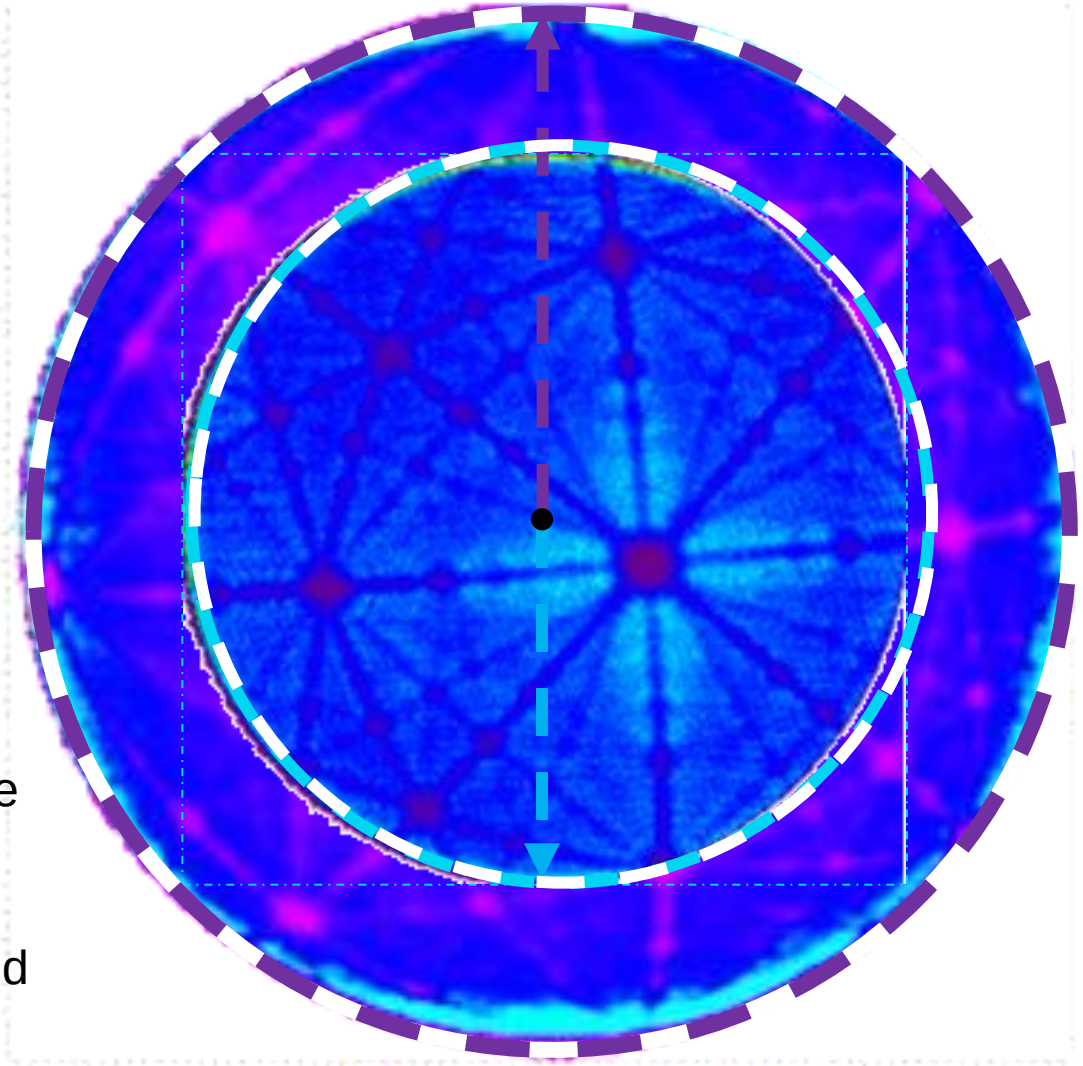
- Enhanced specimen yields (for some materials) and data quality due to DUV wavelength, geometry, and enhanced data acquisition controls
- The Invizo provides ultra wide field of view – in some cases it captures the entire internal volume depending on the specimen details
- Extends APT to measurement of longer length-scales - improved capability to analyze larger features of interest within the field of view





INVIZO DELIVERS MORE THAN DOUBLE THE DATA

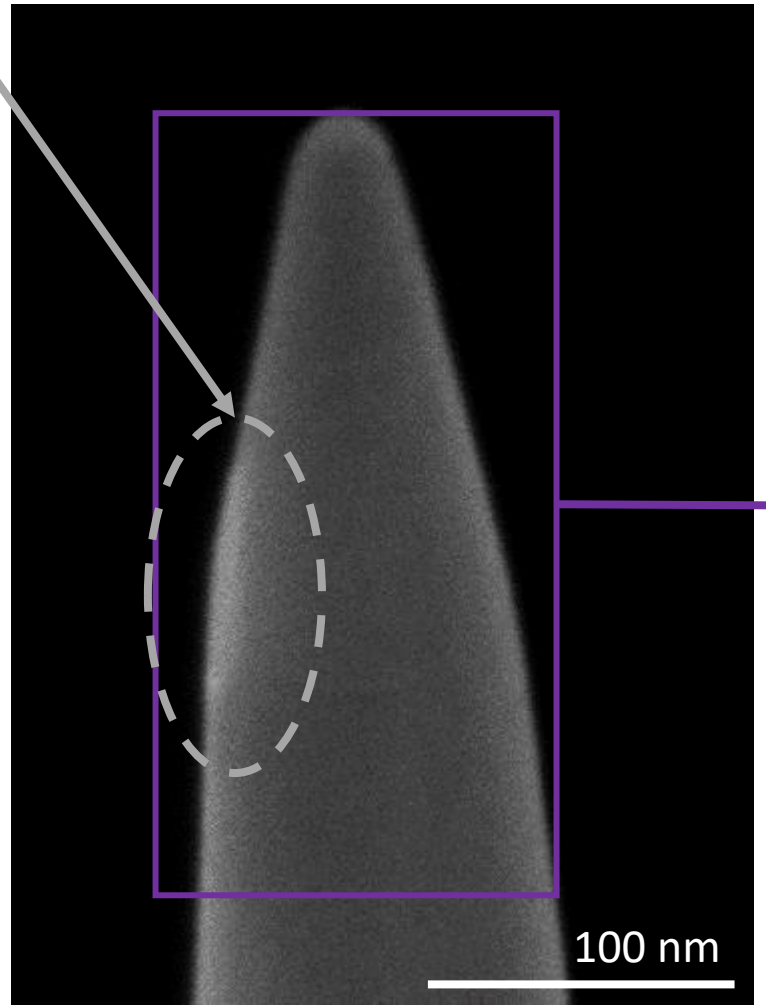


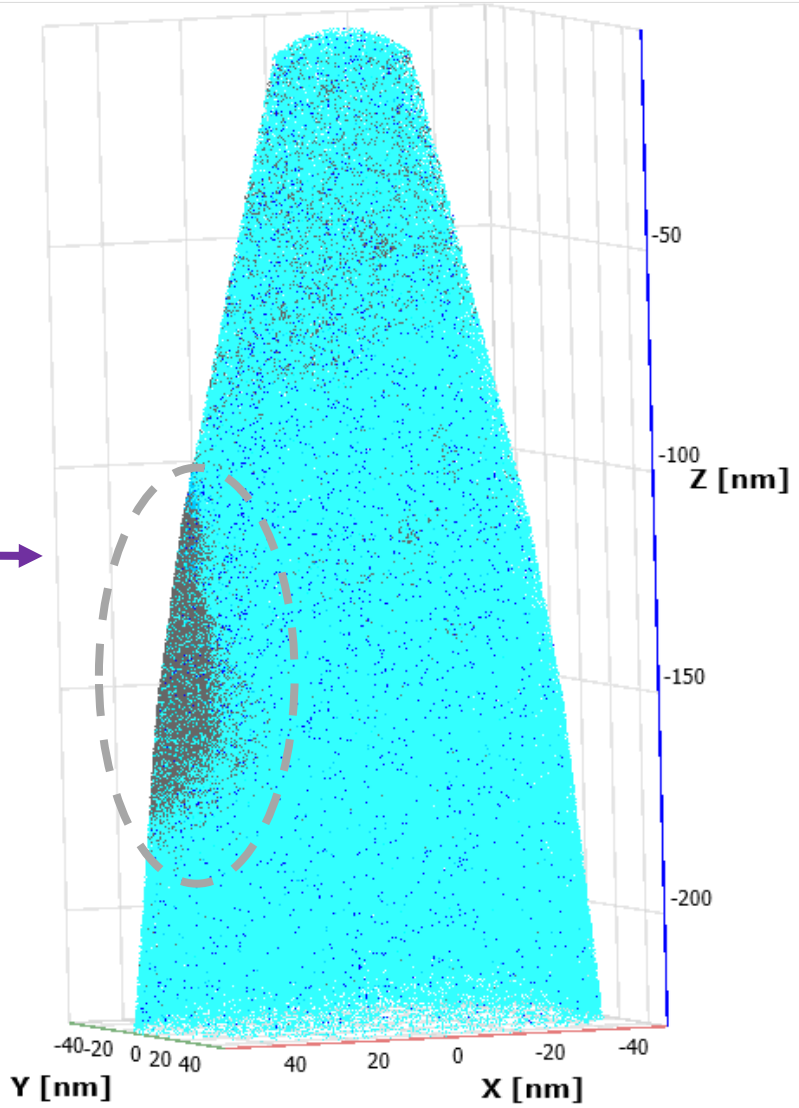


- Advanced lensing technology can provide more than twice the data from each specimen compared to the 5000 (specimen geometry dependent)
- Aluminum data obtained from the same specimen with and without the Invizo lensing systems active - Areal ratio measured at right
- R^2_{6000F} / $R^2_{No\ lens}$ = >2X ➔ **<u>Double the Data</u>**

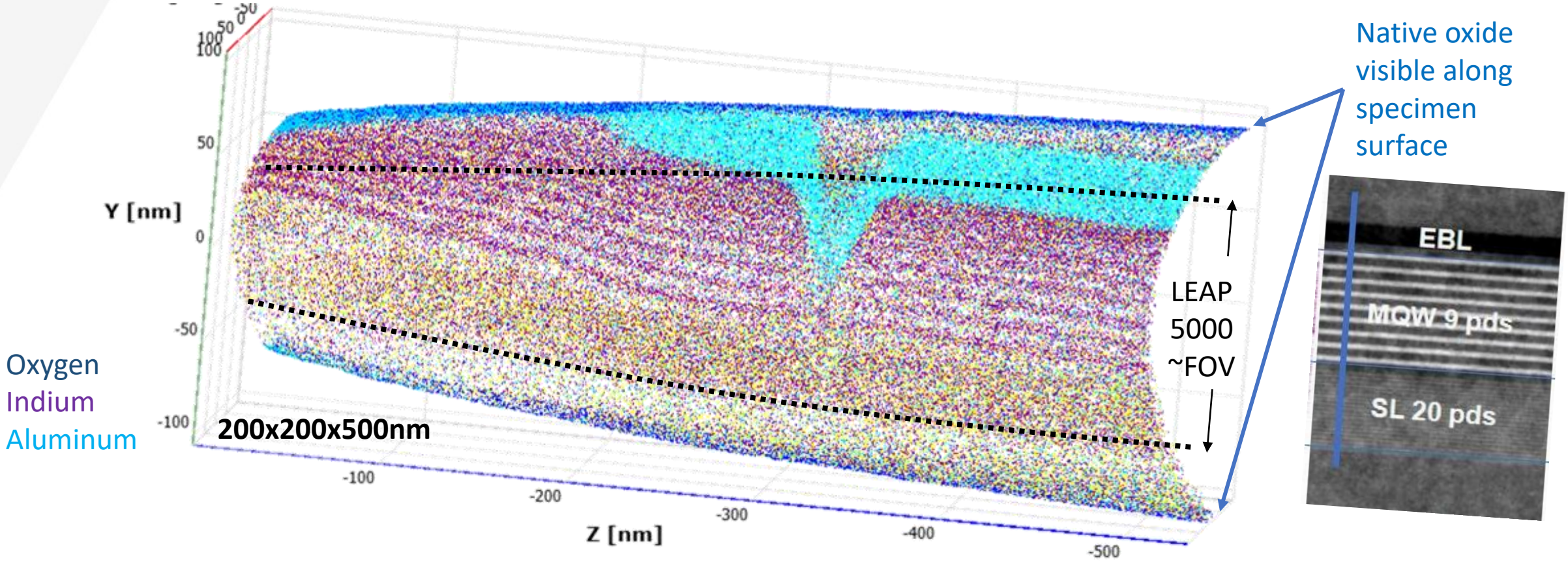
ULTRA WIDE FIELD OF VIEW

- The large precipitate observed in the SEM image at the surface of the specimen after specimen preparation
- It is visible in the atom probe data as well
- We are collecting very close to ALL of the data possible in this specimen





NEARLY FULL FIELD OF VIEW

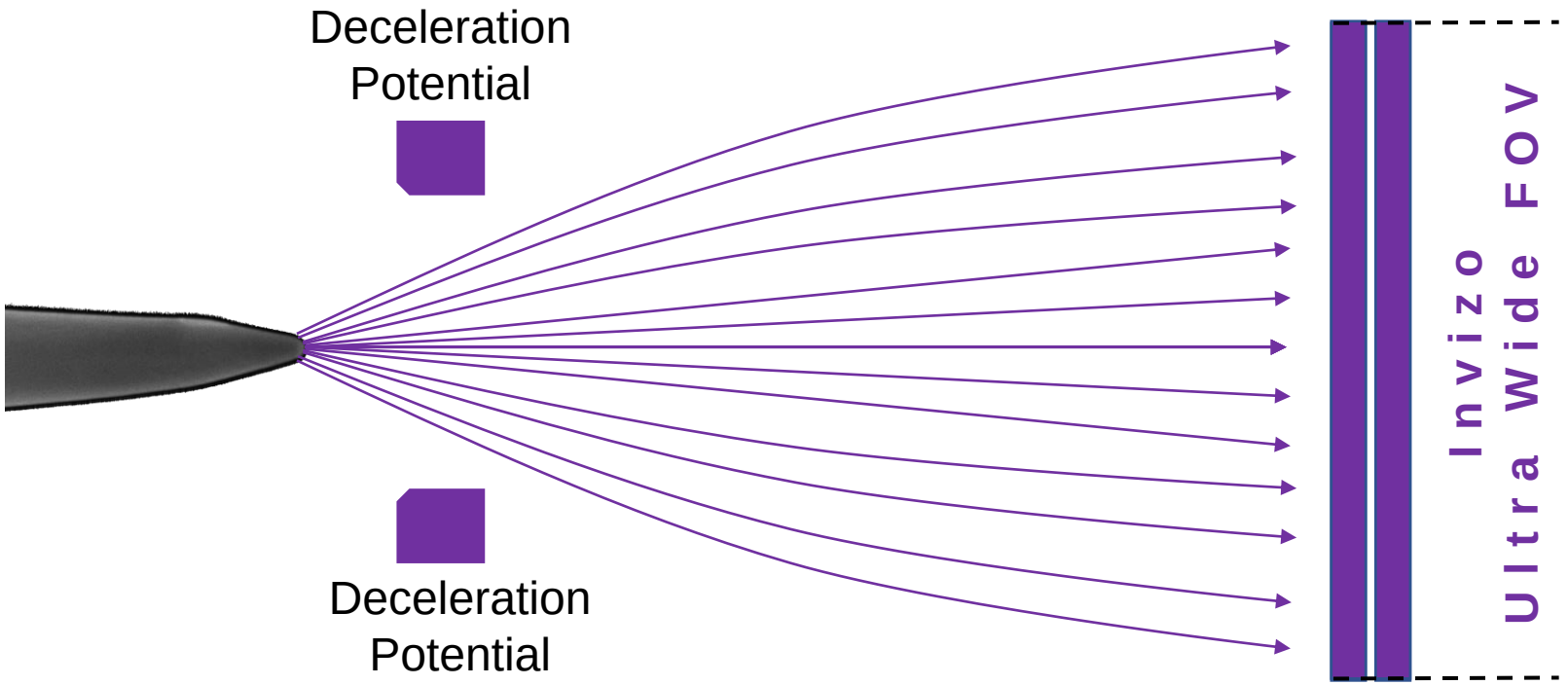


- The Invizo 6000 brings the capability to caption larger features in their entirety – the field of view in the above example is approaching 0.25µm at end of the dataset
- Large FOV means easier specimen preparation, which do not need to be as well centered with such a large data collection angle, the oxidized outer layer from specimen preparation is visible in this image

FLIGHT PATH DESIGN – ELECTROSTATIC COMPRESSION

- Novel electrostatic lensing design produces nearly full field of view XY data
- Mass resolving power maintained/improved due to:
  - Longer physical flight path ➔ longer flight times
  - Lensing design reduces time uncertainties (acceleration to higher total potential)

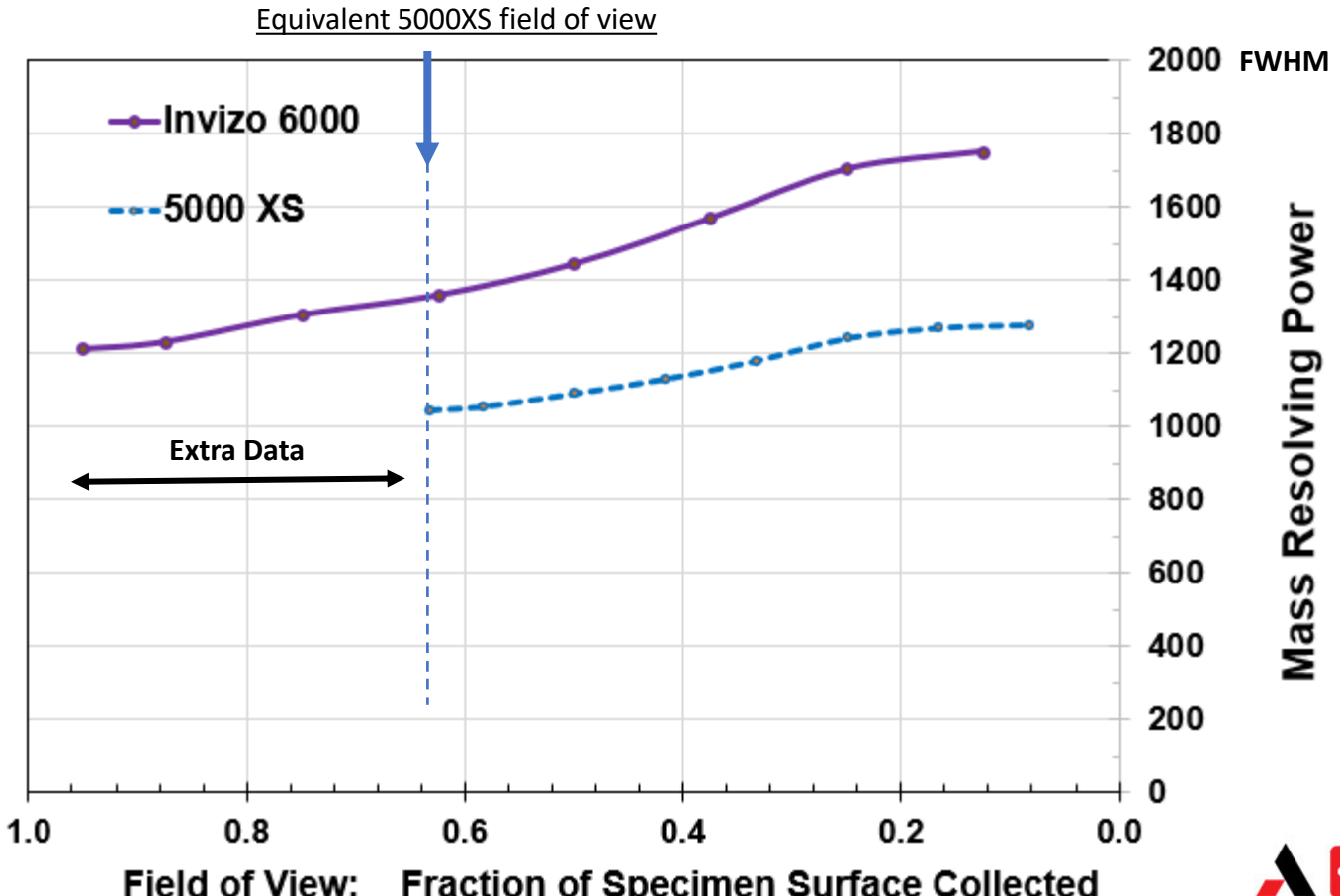
Patented Straight Path Design Increases Field of View



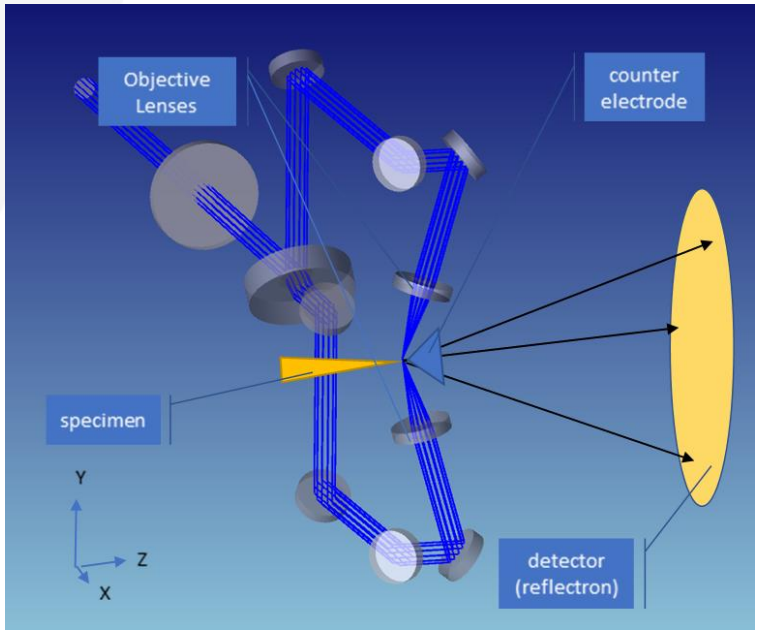
INVIZO 6000: IMPROVED MRP AND OVER A WIDER FOV

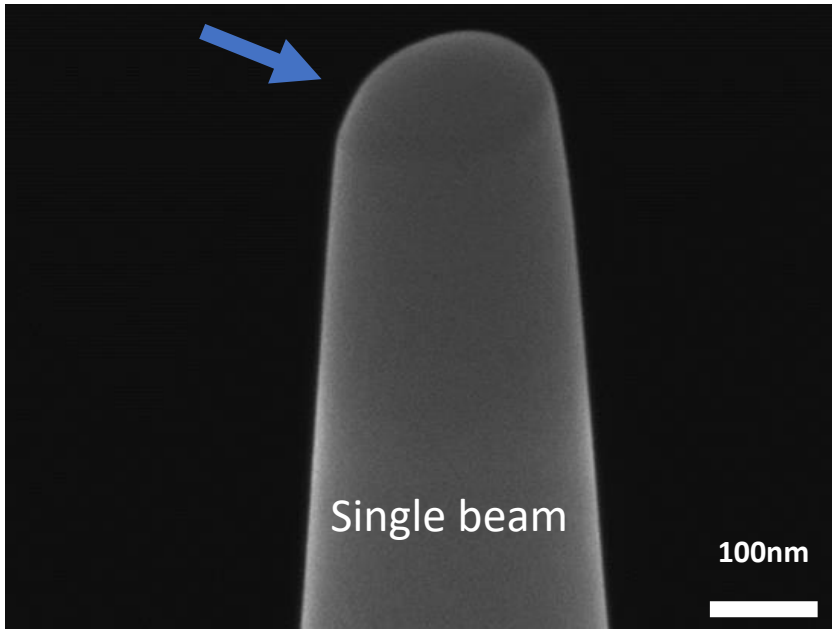
Mass Resolving Power(MRP)	LEAP 5000 XS (typical)	Invizo* (typical)
Half Max	1040	1230
Tenth Max	480	520
Hundredth Max	245	245

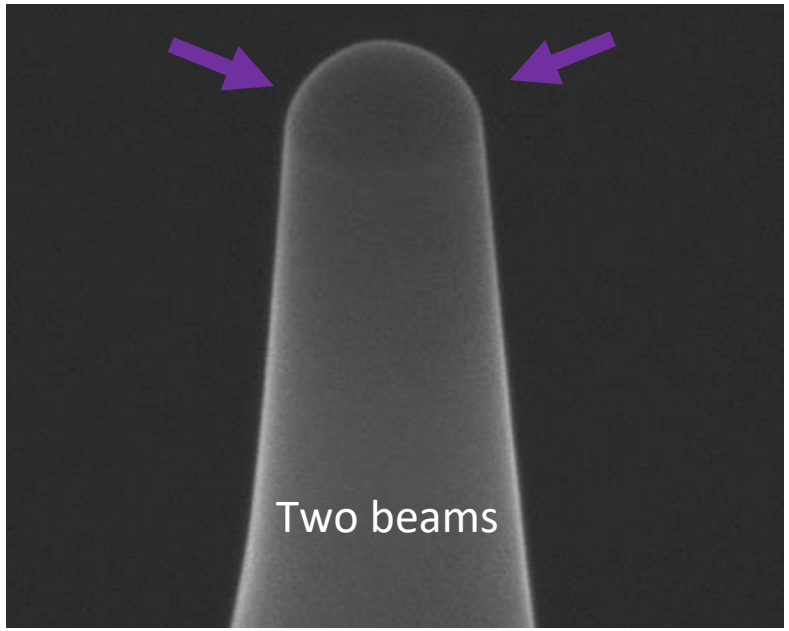
- Normally design tradeoffs involve the loss of performance in one metric to improve the other
- The innovative Invizo 6000 flight path provides better mass resolving power than the 5000 across the entire wide FOV
- The example Invizo data are from 30 laser pulsed aluminum and are in comparison to typical LEAP 5000XS runs



SYMMETRIC HEATING – IMPROVED RECONSTRUCTION







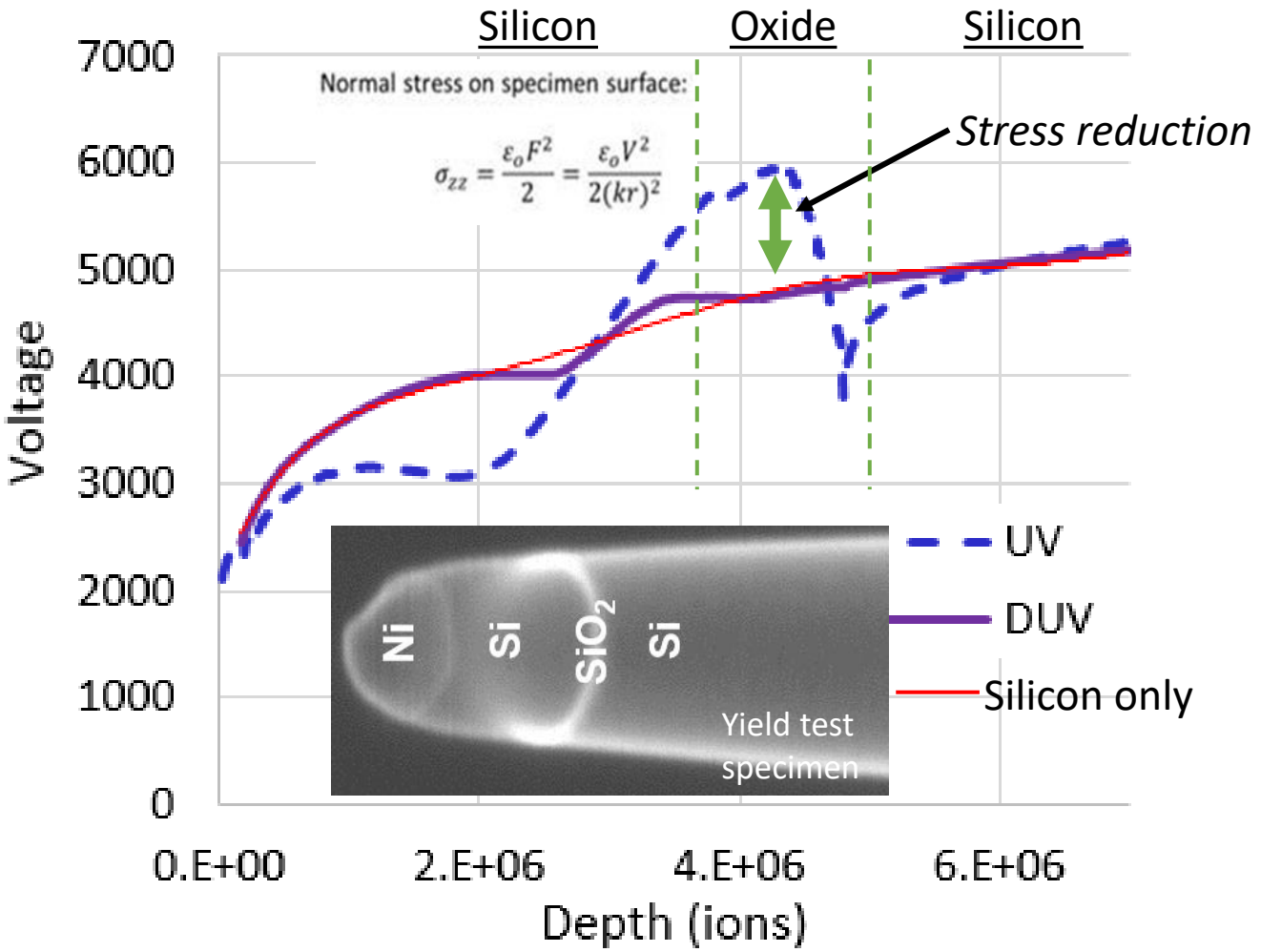
- Comparison of a representative specimen shape (silicon) from one-beam and two-beam laser conditions shown above right
- This feature provides improved reconstruction due to a specimen shape closer to hemispherical and more uniform data quality (MRP, CSR, etc.)

D. J. Larson et al., Micro. Microanal. 27(S1) (2021) 2464.

INVIZO 6000 DUV YIELD IMPROVEMENT VS UV - STRESS REDUCTION

- More uniform heat deposition from DUV results in reduced evaporation field (stress) differences producing more uniform specimen shapes (specimen dependent)
- The very wide field of view of Invizo
  - Enables improved evaporation control at lower detection rates (improved yield)
  - Earlier detection of changing phases / regions approaching the end of the specimen (enabling automation controls to adjust run conditions appropriately)

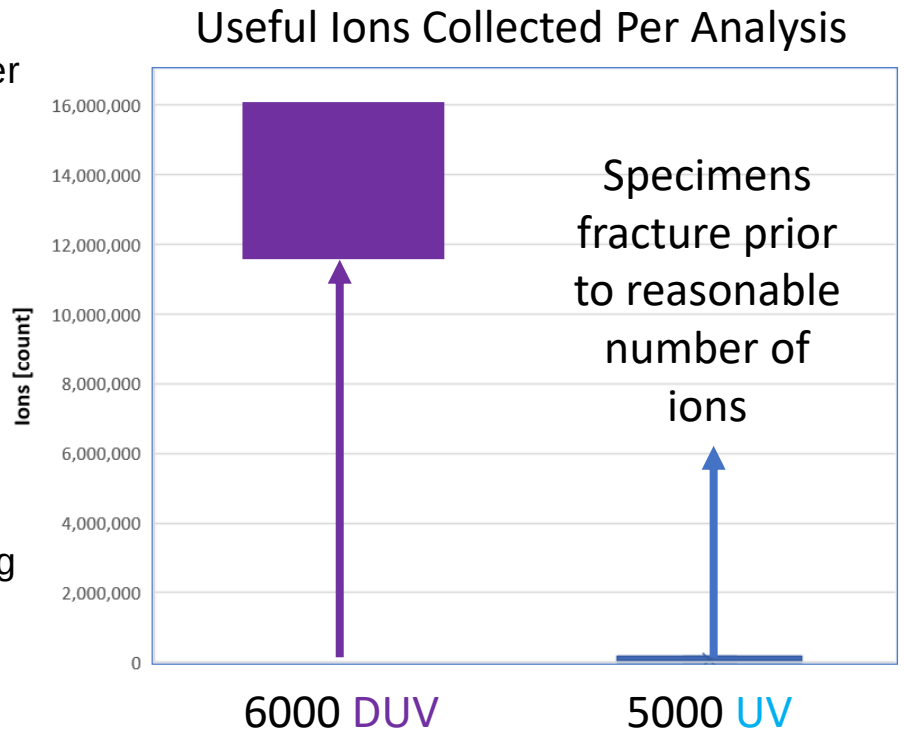
Operating Voltage Comparison*

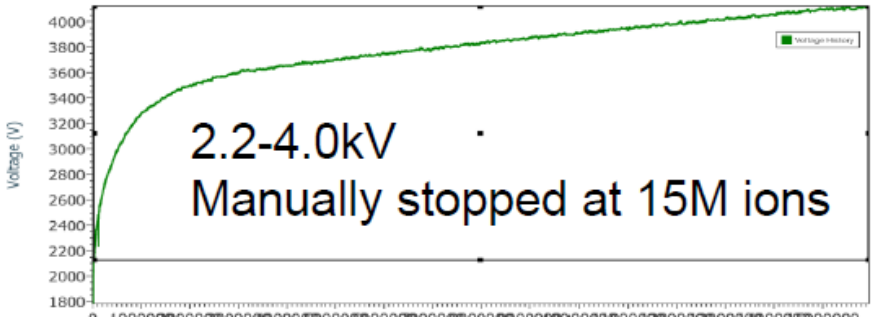


* *T. J. Prosa et al., Micro. Microanal. 25 (2019)*
T. J. Prosa et al., Micro. Microanal. 27(S1) (2021)

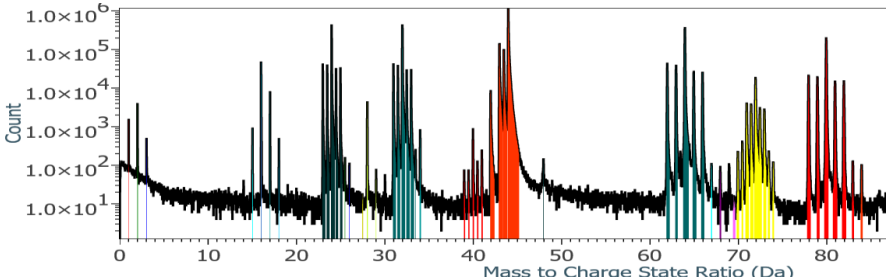
PEROVSKITES – IMPROVED SUCCESS RATES WITH DUV

- The deep ultraviolet (DUV) of LEAP 6000 enables higher yield and higher data quality of perovskites, some of which were very difficult to analyze with UV
- Excellent results with low background, high MRP, and high spatial resolution (lattice spacing resolution)
- Smooth evaporation and outstanding yield compared to the data acquired in LEAP 5000

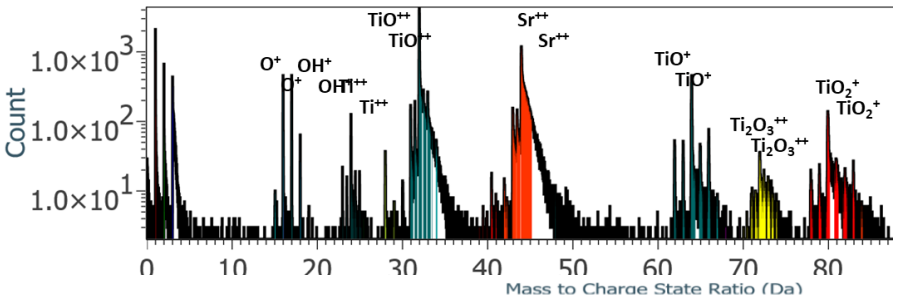




DUV MRP 1030 FWHM



UV – MRP 525 FWHM



Name	Specimen	Good Hits	Laser Power (pJ)	Temperature (K)
R6002_241362	Perovskite_ORNL:M-11	15,909,754	5	25
R6002_241361	Perovskite_ORNL:M-10	11,758,545	20	50
R5100_241382	Perovskite_ORNL:M-16	63,959	300	50
R5100_241381	Perovskite_ORNL:M-10	184,085	150	50
R5100_241380	Perovskite_ORNL:M-10	10,847	80	50
R5100_241378	Perovskite_ORNL:M-15	64,569	50	50

With J. Poplawski (ORNL)

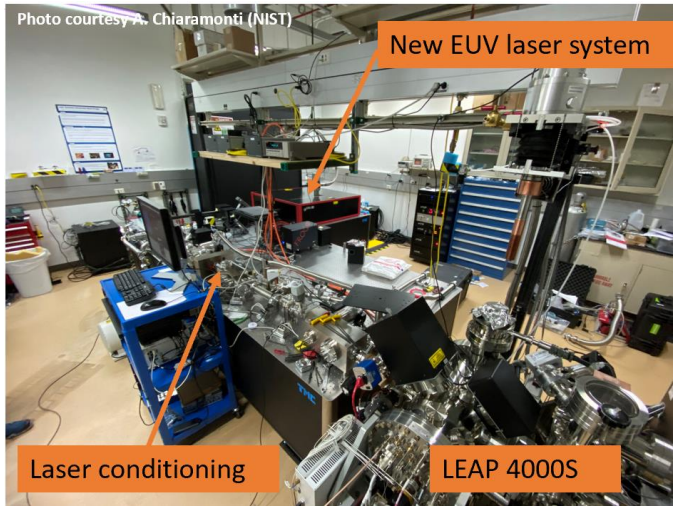
SUMMARY: THE CAMECA 6000 SERIES PRODUCT LINE

LEAP 6000 XR Feature	Result	Benefit
Deep UV laser wavelength	More uniform heat deposition and reduced evaporation field (stress) differences producing more uniform specimen shapes	• Improved yield • More accurate reconstruction
Hybrid V+L pulsing operation	Improved signal-to-noise in mass spectrum	• Higher sensitivity • Easier peak identification
LEAP Automation	Enhanced ease of use and throughput	• Off-hours operation • Faster return on investment • Reduced training requirements

Invizo 6000 Feature	Result	Benefit
Deep UV laser wavelength	More uniform heat deposition and reduced evaporation field (stress) differences producing more uniform specimen shapes	• Improved yield • More accurate reconstruction
Dual beam illumination	More uniform heat diffusion resulting in more uniform field evaporation	• More accurate reconstruction • Improved MRP uniformity
Ultra-wide field of view with long flight path	Capture a greater fraction of the specimen with high mass resolving power	• Simplified specimen preparation with higher change to caption region of interest • Better concentration statistics and larger feature capability • Improved yield

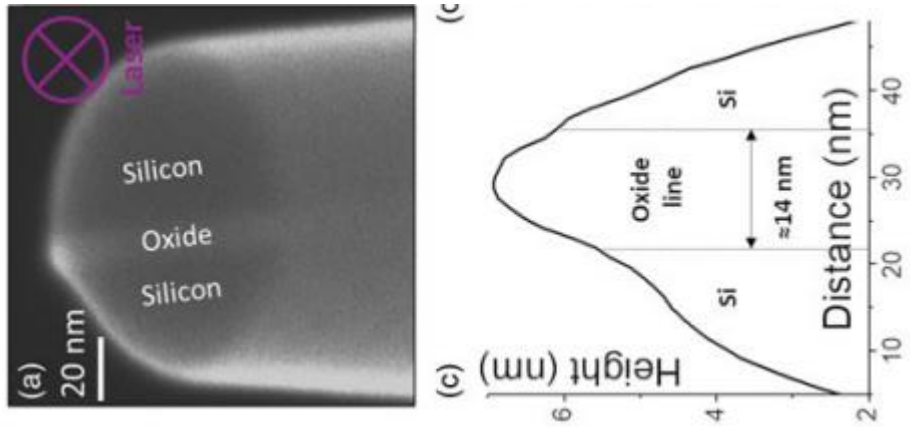
CAMECA CURRENT EXTERNAL RESEARCH COLLABORATIONS

Extreme UV Laser Atom Probe



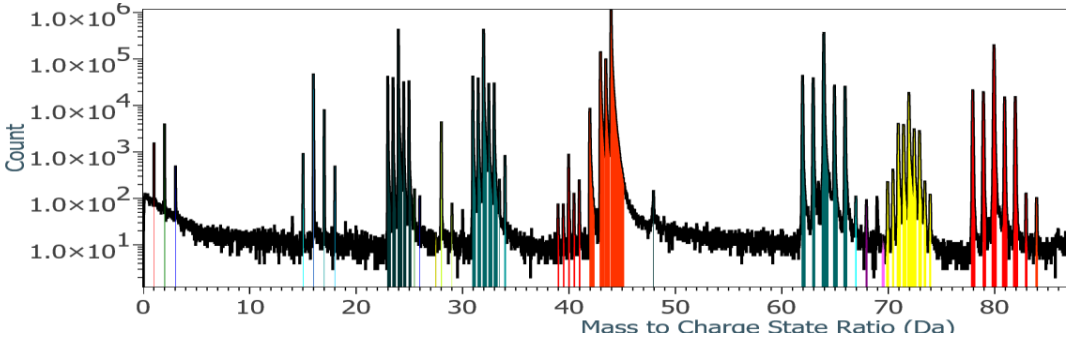
A. Chiaramonti, L. Miaja-Avila et al. (NIST)

APT+AFM



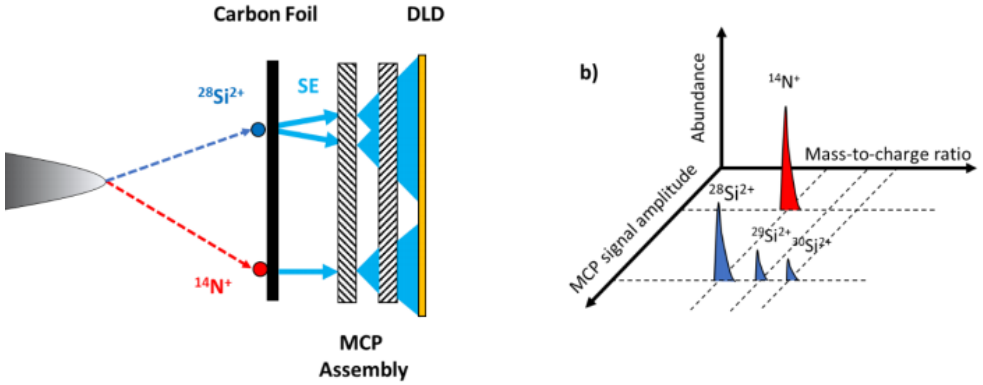
P. van der Heide, Claudia Fleischmann et al. (IMEC)

Improved Yield - Perovskites



J. Poplawski (ORNL)

Detector Development



F. Vurpillot et al. (Normandie Université)

THANK YOU!

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