Atom Probe Tomography using Extreme-Ultraviolet Light

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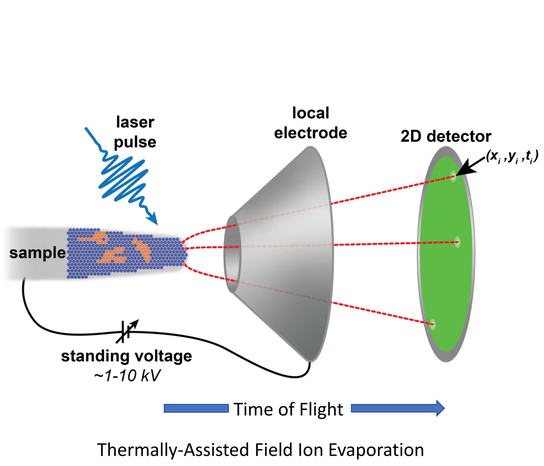
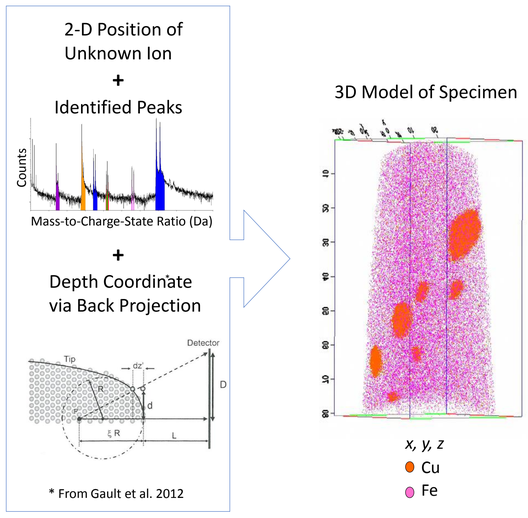
Atom Probe Tomography

Laser-Assisted Atom Probe Tomography (LAPT) is **time-of-flight mass spectrometry** coupled with **position-sensitive detection** on the scale of **individual atoms**



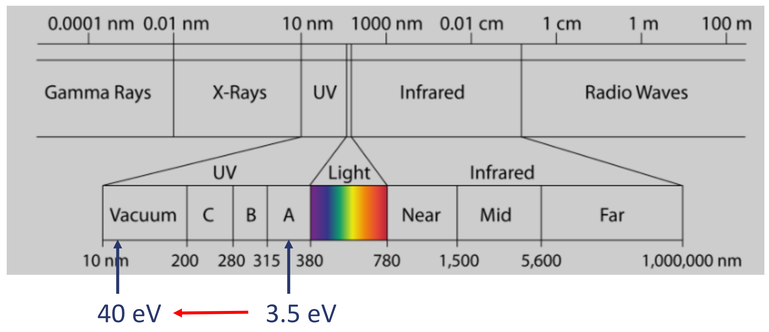
Thermally-Assisted Field Ion Evaporation



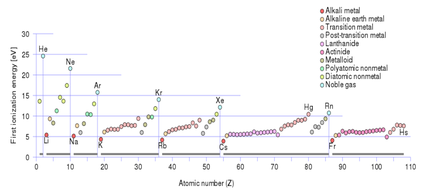
Strengths:
- Isotopic identification
- Compositional analysis
- sub-nm resolution
- 3D chemical mapping

Limitations:
- Ion emission persists after laser pulse → "thermal tails"
- Emission of molecular ions and neutral species
- Laser-pulse-energy-dependent composition bias

Extreme-Ultraviolet Light

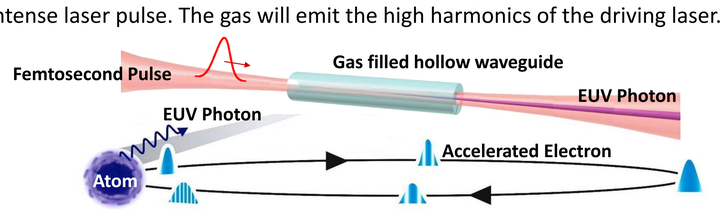


Above the **work function** and **ionization potential** of any material.



What is High-Harmonic Generation (HHG)?

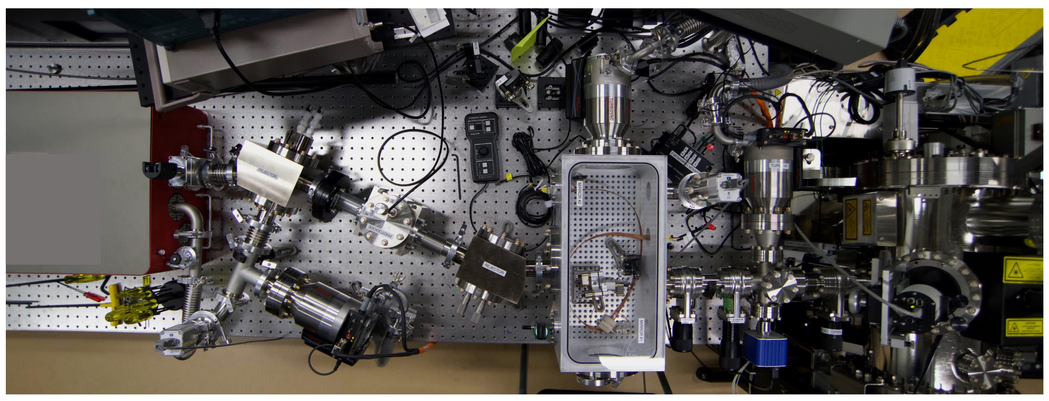
HHG is a nonlinear optical process where a gas target is illuminated by an intense laser pulse. The gas will emit the high harmonics of the driving laser.



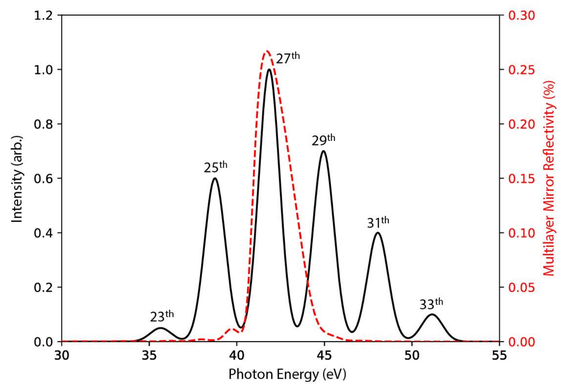
Gas	Photon energy (eV)	Wavelength (nm)
Kr, Xe	10 – 30	120 – 40
Ar	20 – 50	60 - 25
Ne	40 – 80	30 – 15
He	> 80	< 15

Absorption cross sections (material dependent) in the EUV range are generally several orders of magnitude larger than in the NUV range.

Extreme-Ultraviolet assisted Atom Probe Tomography at NIST Boulder



Commercial EUV source interfaced to a commercial atom probe. The HHG capillary is at ~ 40 Torr and the analysis chamber in the APT is at ~ 2×10^{-11} Torr. Vacuum beamline includes focusing and steering optics, filters, and diagnostics.



HHG spectrum (40-Torr Argon) and multilayer mirror (pair) reflectivity curve.

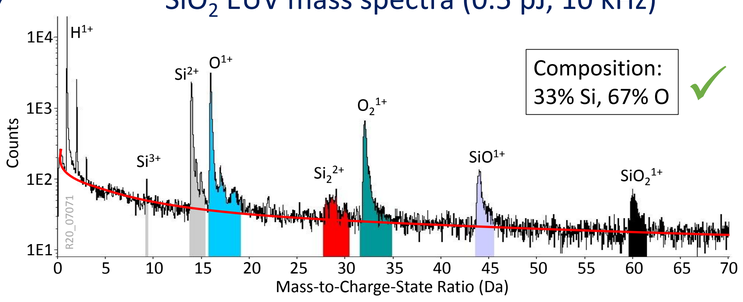
EUV vs conventional near-UV Atom Probe Tomography

	EUV	Near-UV	
Laser pulse width	10 fs	10 ps	
Focused spot size	50 µm	2 µm	
Photon energy	42 eV	3.5 eV	
Laser pulse repetition rate	10 kHz	250 kHz	500 kHz
Laser pulse energy	0.5 pJ	50 fJ	150 pJ
Photons/pulse	7.5×10^4	8.9×10^4	2.7×10^8
Fluence (J/cm^2) *per pulse*	5.1×10^{-8}	3.2×10^{-6}	9.5×10^{-3}

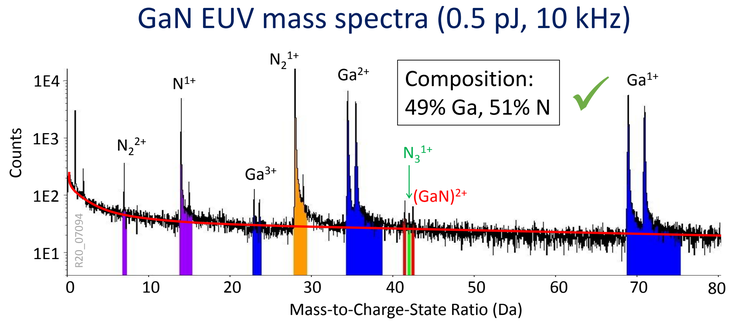
N. Sanford, *et al.* - US Patents: 9,899,197 (2018), 10,153,144 (2018); A.N. Chiaramonti, *et al.*, submitted

EUV-APT Results

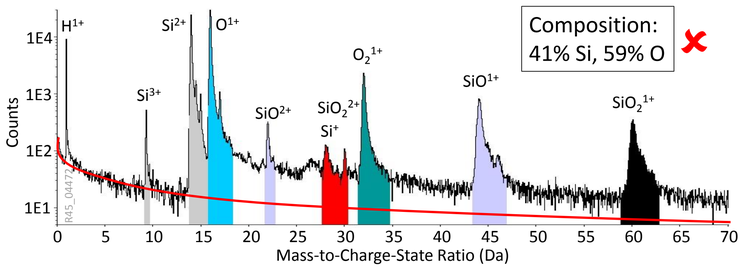
SiO_2 EUV mass spectra (0.5 pJ, 10 kHz)



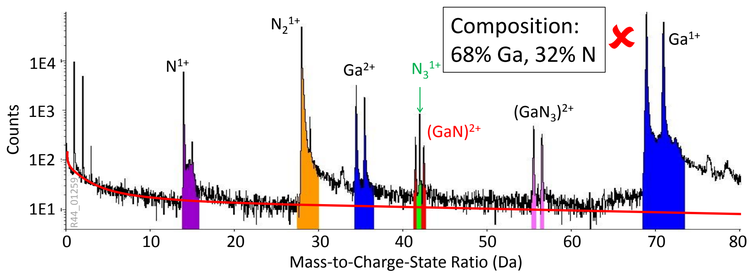
GaN EUV mass spectra (0.5 pJ, 10 kHz)



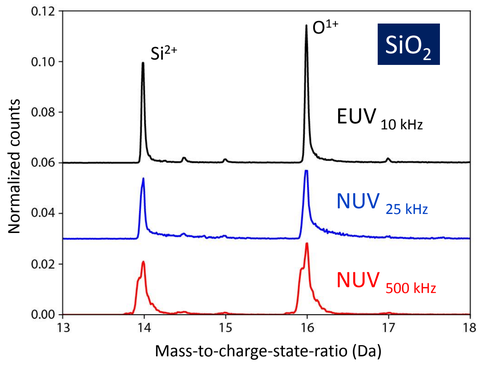
SiO_2 NUV mass spectra (150 pJ, 500 kHz)



GaN NUV mass spectra (50 fJ, 250 kHz)



Decreased peak widths & tails

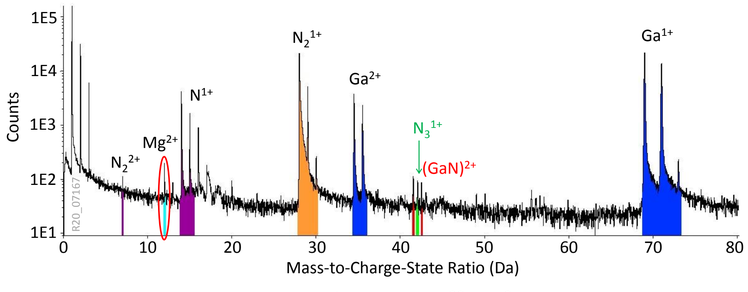


FWHM (Si^{2+}) ~ 40 mDa
FWHM (O^{1+}) ~ 40 mDa

FWHM (Si^{2+}) ~ 60 mDa
FWHM (O^{1+}) ~ 70 mDa

FWHM (Si^{2+}) ~ 110 mDa
FWHM (O^{1+}) ~ 130 mDa

EUV-APT of Mg:GaN test structure



- Measured Mg concentration of ~ 1.03×10^{20} cm^{-3}
- Concentration values consistent with SIMS and NUV-APT measurements

Outlook

- Improvements in vacuum beamline to reduce EUV losses and optimize focusing conditions resulting in higher photon flux at the sample position.
- Acquisition of new laser system with higher repetition rate.
- Incorporation of scanning electron microscopy capabilities into the UHV chamber for in situ specimen shape imaging.
- Perform EUV-APT experiments on material systems with poor near-UV absorption or that are difficult to run in conventional APT.
- Exploration of different EUV energies in the regime of 10 – 25 eV to better understand the capabilities of EUV light.
- Study the possibility of using the EUV light for imaging the specimen through ptychography techniques.

Summary

- Constructed the first reported EUV atom probe tomograph by combining a commercial pulsed EUV source with a commercial local electrode atom probe.
- Demonstrated field ion evaporation in SiO_2, GaN, and Mg:GaN.
- EUV-APT spectra shows a reduced formation of molecular ions, decreased peak widths, and reduced "thermal tails".
- Bulk stoichiometry has been recovered, within uncertainty, in all samples.

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