

Nanometer-Scale Trace Analysis

Wallis F. Calaway, Igor V. Veryovkin, Jerry F. Moore, Michael R. Savina and Michael J. Pellin

Motivation/Major Accomplishments

- Trade off between sample size and achievable detection limit
 - For sputtering or laser desorption
 - Quantified by useful yield (atoms detected/atoms consumed)
- Laser post-ionization secondary neutral mass spectrometry (LPI-SNMS)
 - Useful yields significantly higher than competing techniques (e.g., SIMS)
 - Well suited for trace analysis of small samples.
- New reflectron time-of-flight (TOF) mass spectrometer
 - Especially designed to optimize useful yield
 - One of three instruments completed
 - Extraction from a large volume ($4 \times 4 \times 3 \text{ mm}^3$)
 - Transmitted >98% efficient.
 - Large ionization volume contains >40% of desorbed species
 - Calculated useful yield >30%
- Permits nanometer-scale analysis at trace levels
 - For detection limited is controlled by sample size
 - For rare or precious sample where sample consumption is important

Small Sample Analysis

Monolayer	Nanometer-Scale	Bulk
100 nm square	50 nm cube	$(300 \mu\text{m})^2 \times 0.1 \mu\text{m}$
1.5×10^5 atoms	6×10^6 atoms	4×10^{14} atoms
1 atom = 7 ppm	1 atom = 160 ppb	400 atoms = 1 ppt

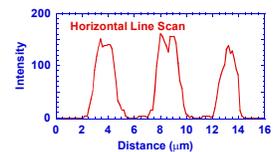
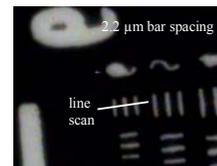
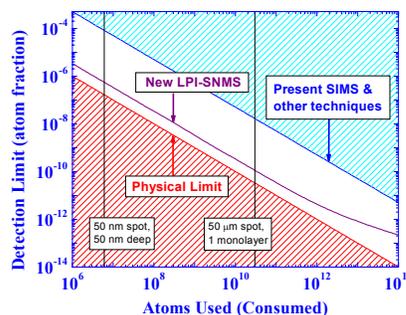
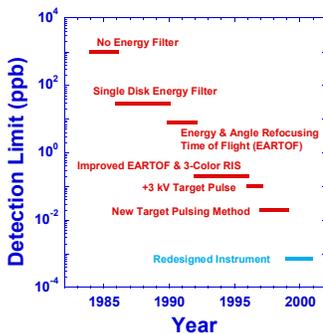
- All three examples demonstrate the same problem:
- Finite sample size. So few atoms, every atom counts.
 - Characterization must be accomplished before consuming the total number of atoms available.

Sub- μm Analyses Accomplished by Ion Sputtering or Laser Desorption

The new instrument incorporates

- a liquid metal ion source with 50 nm spot size for nanometer-scale analysis
- an in situ, high numeric aperture microscope
 - for imaging of sample surfaces
 - for laser desorption of samples for analysis.

Ultra-Trace Analysis and Detection Limit

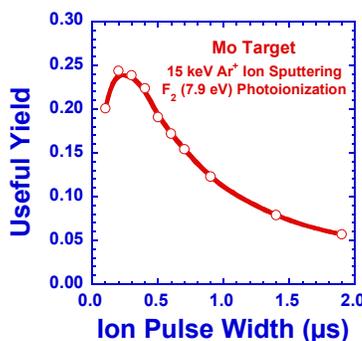


Microscope image

Measured line scan shows 0.5 μm resolution.

Measured Useful Yield = 24%

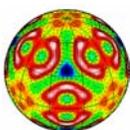
- Useful yield measured as function of primary ion pulse width.
- 1 in 4 atoms detected.
- Result X100 better than best SIMS
- Ion optics simulations predict 1 in 3 ions detected.
- Instrument improvements possible to reach calculated useful yield.



Impact and Future Directions

- Analysis of small samples at trace levels never before achievable (ppt)
- Most sensitive in the world for surface analysis
- Many new analytical applications now possible
- Future applications
 - Characterization of nanometer-scale surface features with monolayer depth resolution
 - Quantitative analysis of extremely rare isotopes
 - Detections of solar wind elements from the collectors of NASA's Genesis Discovery Mission (available in 2005)
 - Compositional analysis of 100 nm interstellar dust particles from NASA's Stardust Mission (available in 2006)

A New Time-of-Flight Instrument for Quantitative Surface Analysis, I. V. Veryovkin, W. F. Calaway, J. F. Moore, M. J. Pellin, and D. S. Burnett, NIM B (submitted, July 2003).



BES - DOE

This work was supported by the U. S. Department of Energy, Basic Energy Sciences, under contract W-31-109-ENG-38 and by NASA under Work Orders W-19,895 and W-10,091

MSD - ANL

