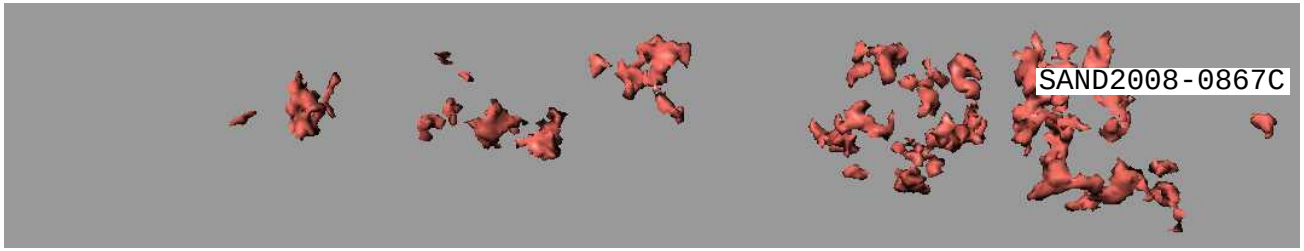


Direct and Computed Tomographic Spectral Imaging

Paul G. Kotula, Ilke Arslan (CA), Luke N. Brewer, and Joseph R. Michael
Sandia National Laboratories, Albuquerque, NM

Lucille Giannuzzi
FEI Company, 5350 NE Dawson Creek Drive, Hillsboro, OR

Felix de Haas
FEI Company, 5600 KA Eindhoven, The Netherlands

Thanks to FEI NA TEM Applications Team for assistance with the 3D model

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

National Nuclear Security Administration

Sandia National Laboratories

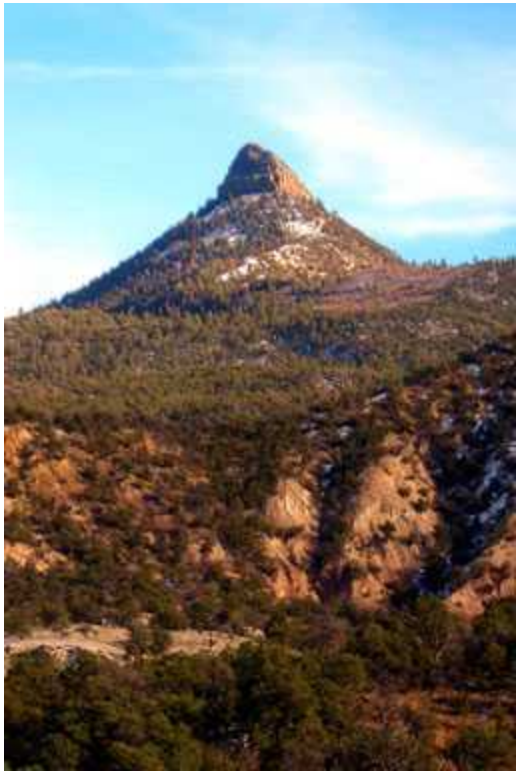
Why do we need 3D?
Because 2D may not tell the whole story!

Cerro Pedernal, Jemez Mountains, NM

Looks like a classic butte

"Pedernal, 1942" by Georgia O'Keefe

Georgia O'Keeffe Museum

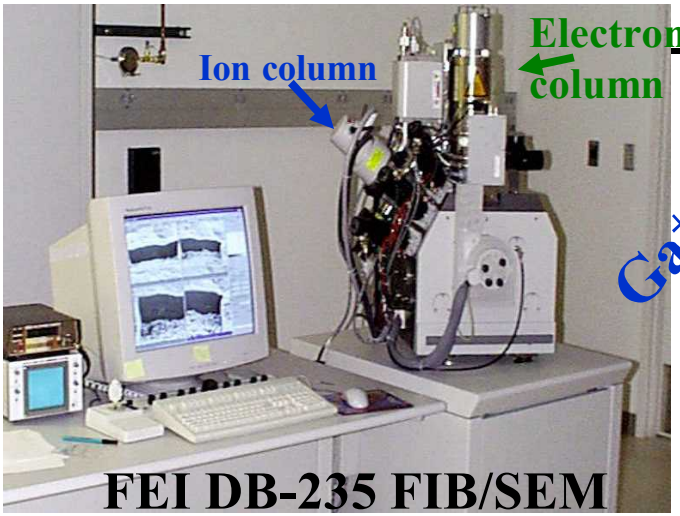


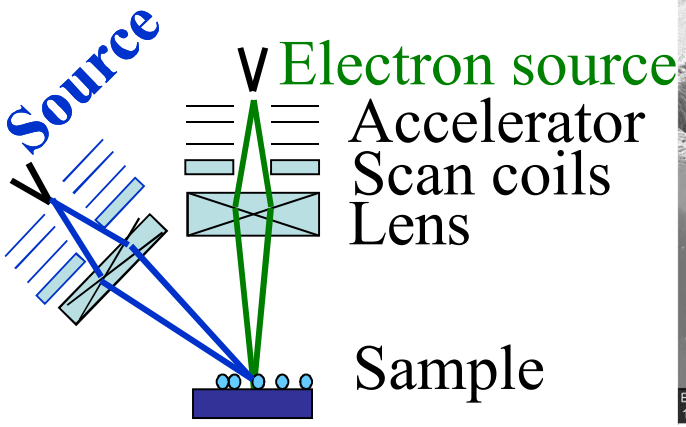
From another perspective we see it's really a narrow ridge

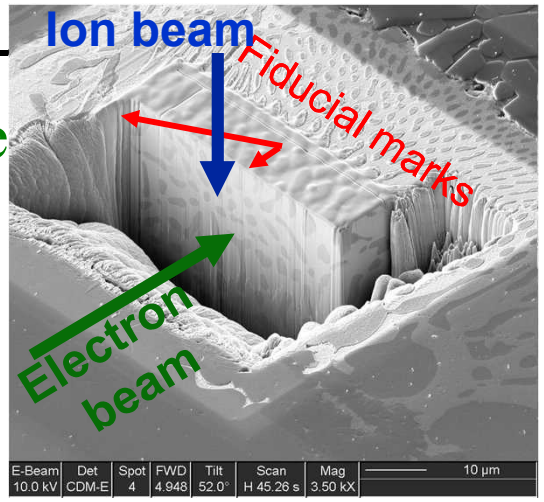
MICROSCOPY and MICROANALYSIS 2008
ALBUQUERQUE
NEW MEXICO
August 3 – 7, 2008

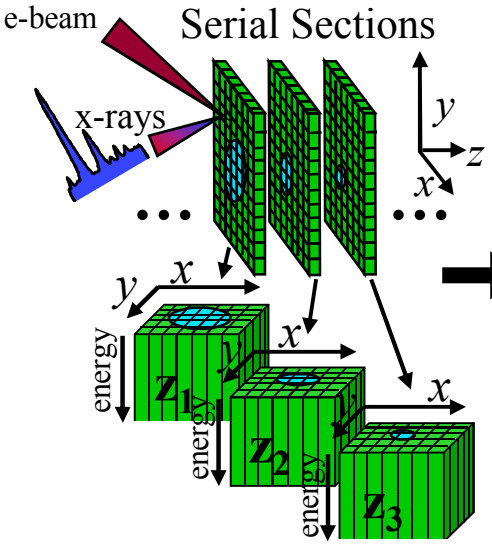
Sandia National Laboratories

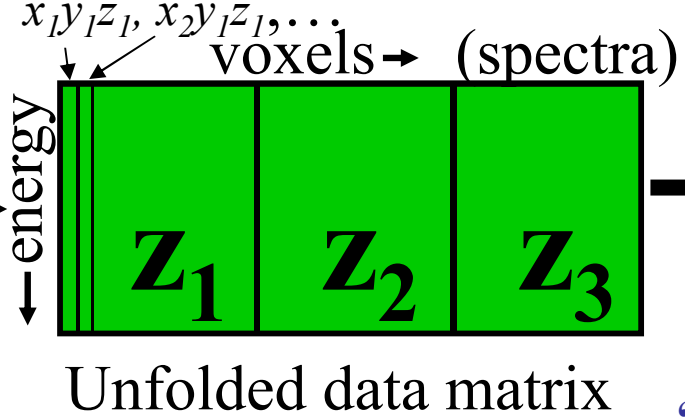
Tomographic Spectral Imaging and Multivariate Statistical Analysis



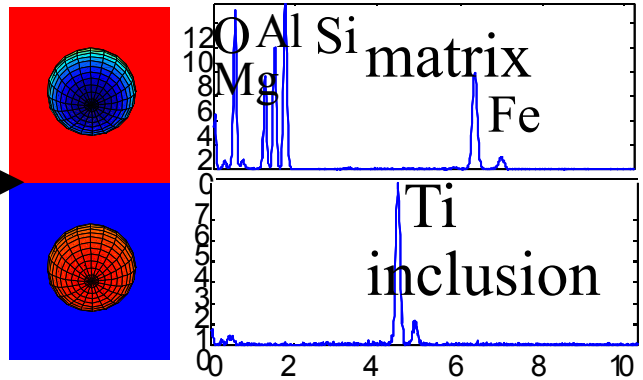








Advanced statistical analysis



'Component images and spectra'

Sandia National Laboratories

Direct Tomographic Spectral Imaging (TSI) via FIB Serial Sectioning

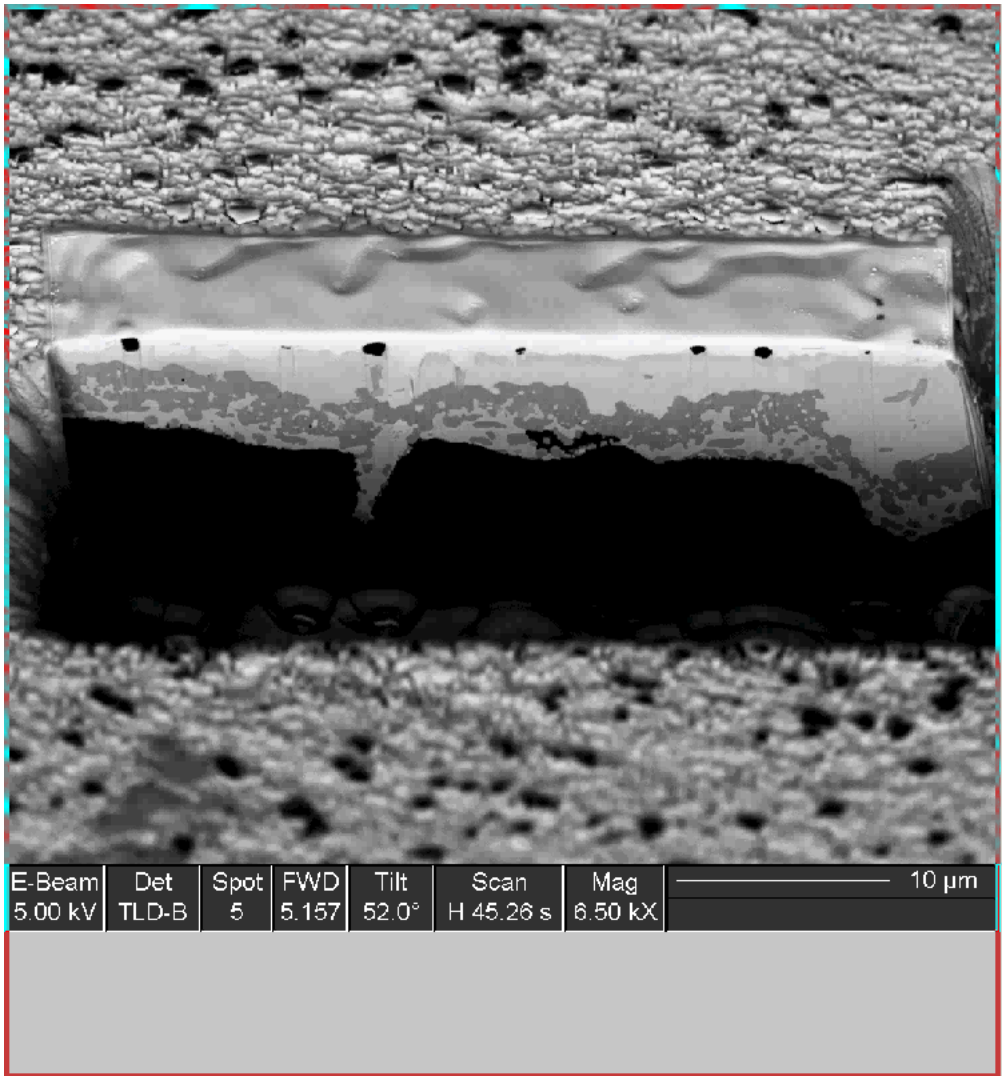
- 5kV spectral image acquired from volume elements
- Automated spectral image analysis algorithms are the same as we showed previously for 2D, 1D*
- Focused ion beam sectioning of a braze joint
  - 2D x-ray spectral image acquired from each of 10 slices
  - 0.5 μm/slice, 40 μm by 16 μm by 5 μm volume analyzed
  - Data set analyzed as a whole in 2.6 seconds to run on a Dual Xeon 3.8 GHz processor PC
  - Limited x-ray peak coverage at 5 kV--poor spectral resolution and significant peak overlap expected
  - 8 components found, 5 rendered here (leave out Ga re-sputter)
- Result is a comprehensive chemical analysis of a 3D volume with no preconceptions

Sandia National Laboratories

Analysis of Ag-2Zr/Alumina Braze

FIB/SEM/EDS

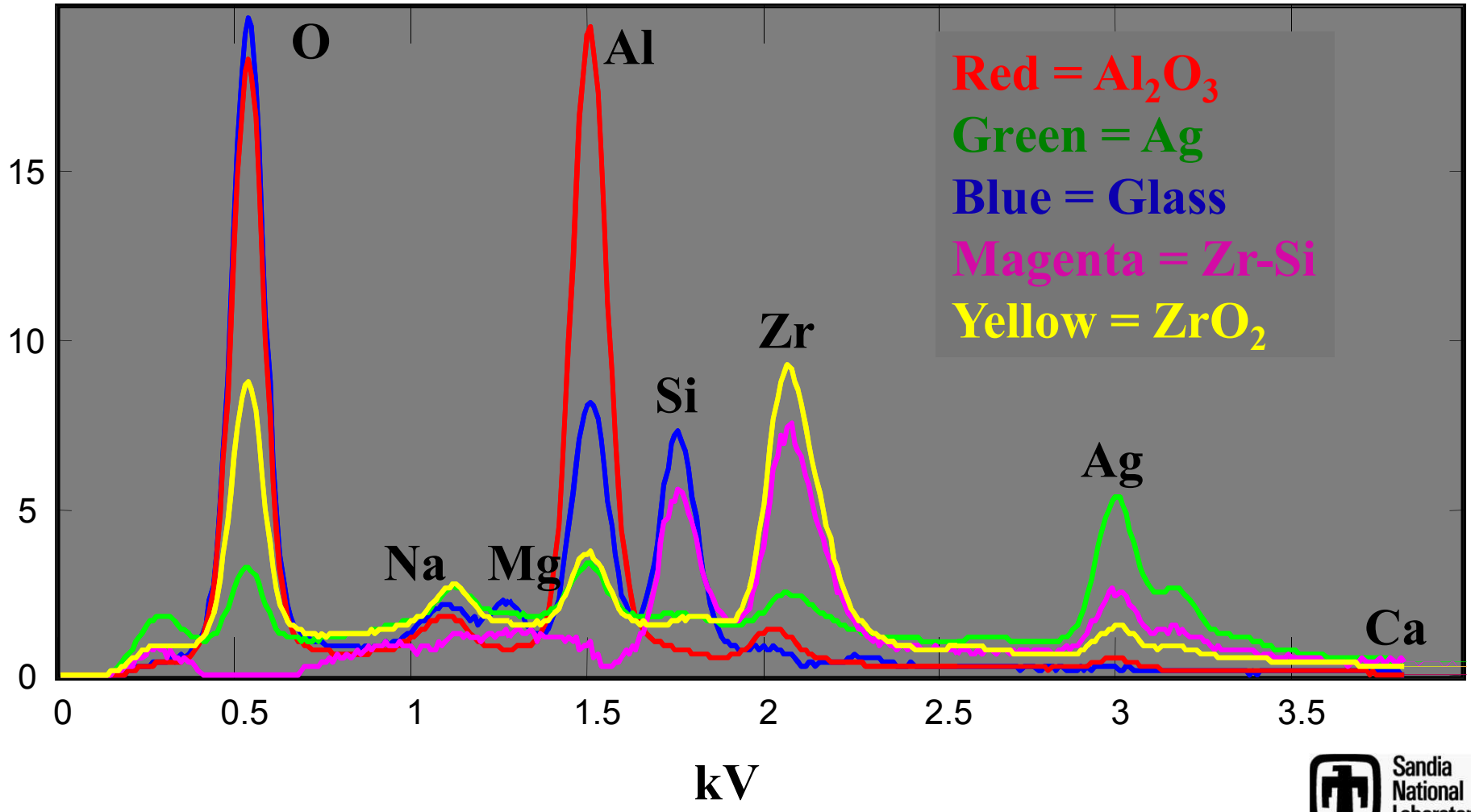
Movie of electron images



Sandia National Laboratories

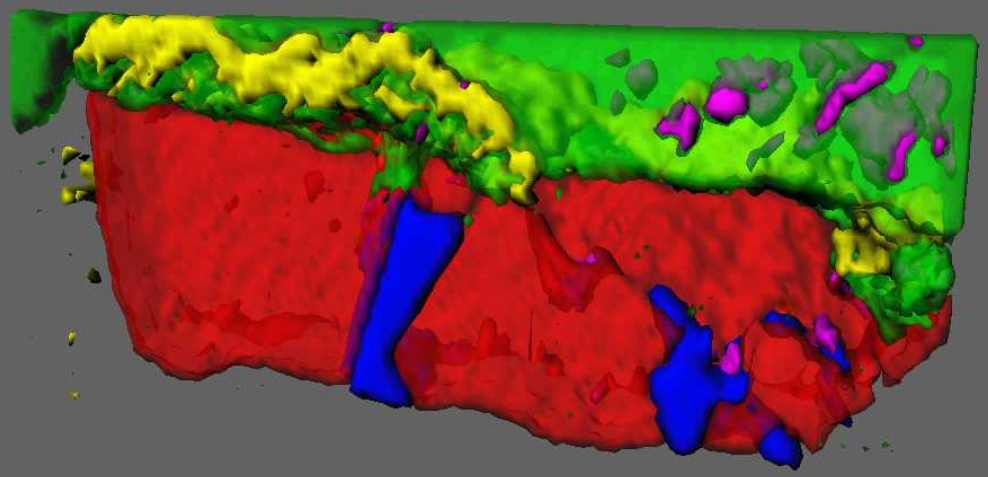
MSA-derived spectral components

Analysis of Ag-2Zr/Alumina Braze (5kV)



Sandia National Laboratories

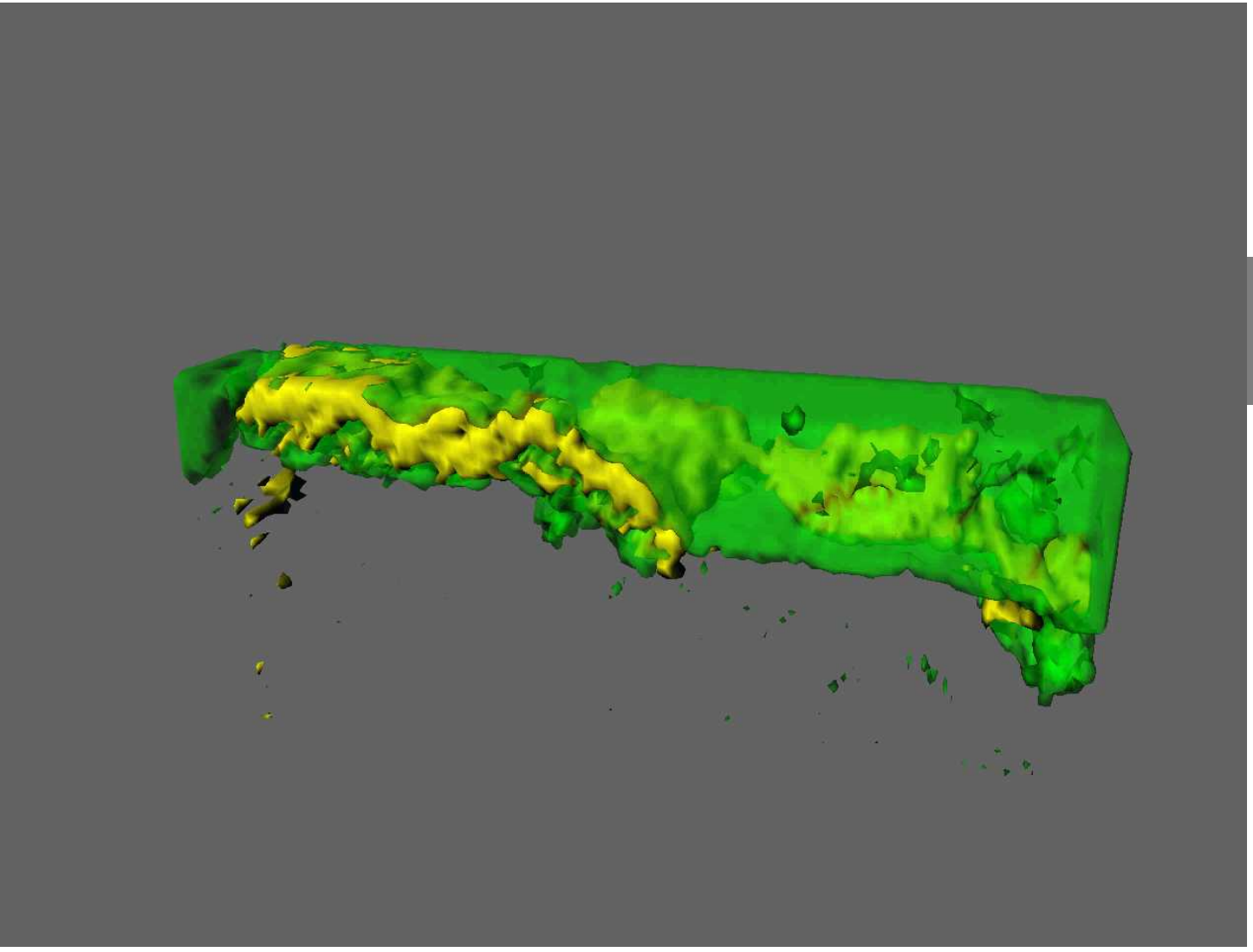
Analysis of Ag-2Zr/Alumina Braze: Everything



Red = Al_2O_3
Green = Ag
Blue = Glass
Magenta = Zr-Si
Yellow = ZrO_2

Sandia National Laboratories

Analysis of Ag-2Zr/Alumina braze: The braze constituents, Ag and Zirconia



Green = Ag
Yellow = ZrO_2

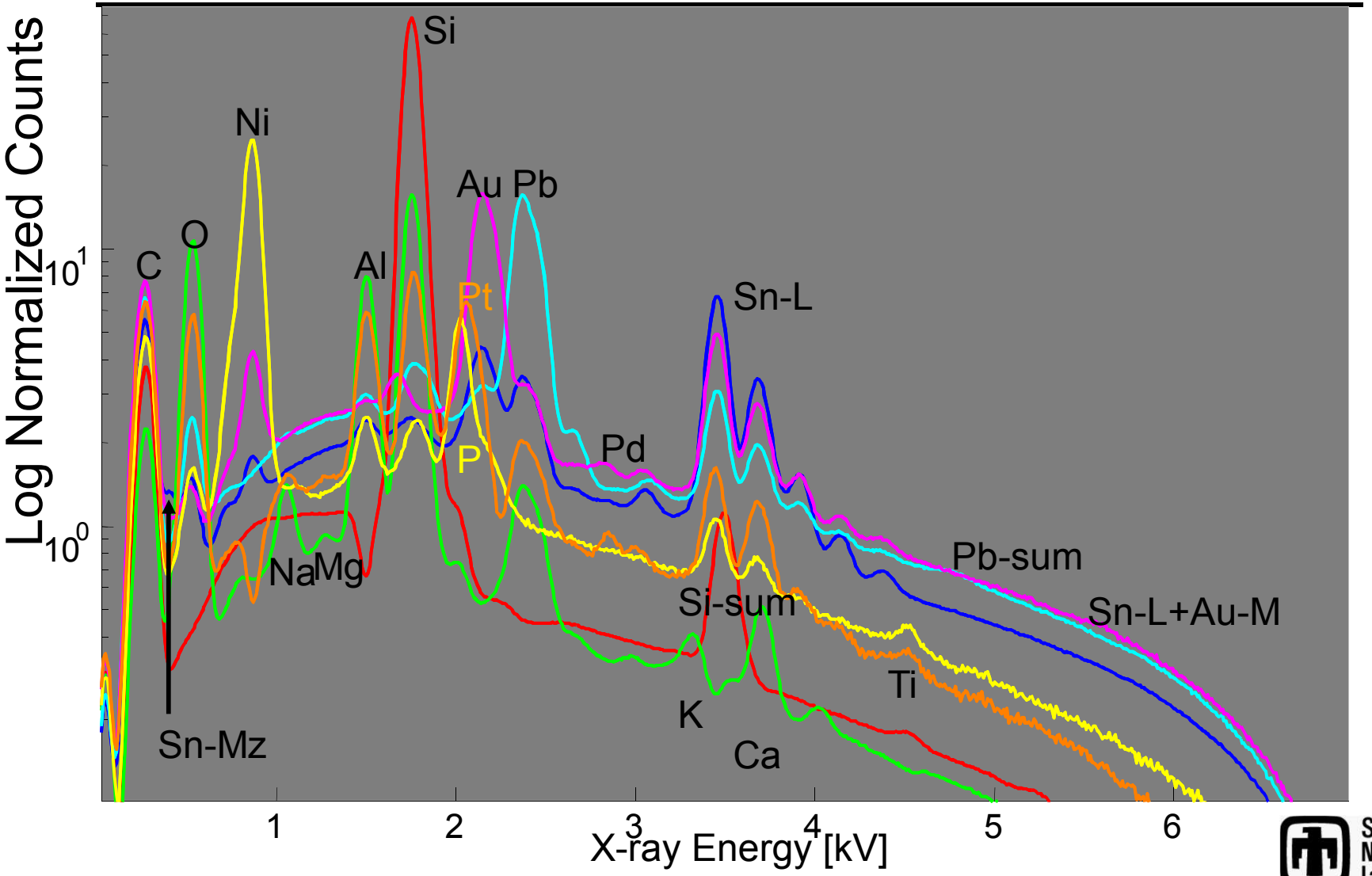
Zirconia in contact with only part of ceramic substrate

Sandia National Laboratories

Direct Tomographic Spectral Imaging (TSI) via Metallography

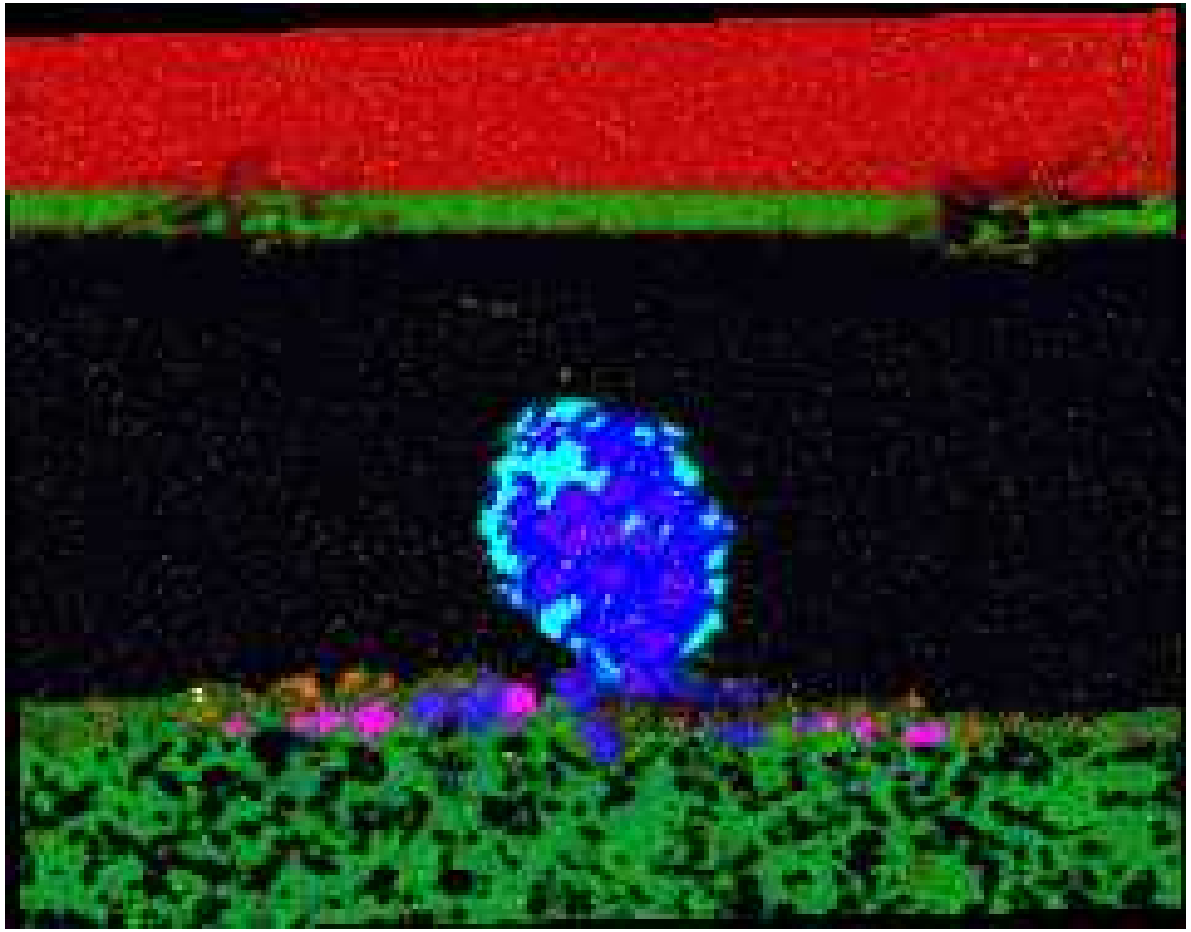
- 7kV spectral image acquired from volume elements
- Automated spectral image analysis algorithms are the same as we showed previously for 2D, 1D*
- Metallographic sectioning of two solder joints
  - 2D x-ray spectral image acquired from each of 64 slices
  - Data set analyzed as a whole (9Gbyte, 13 min analysis)
  - Limited x-ray peak coverage at 7kV-poorer resolution and significant peak overlap expected
- Result is a comprehensive chemical analysis of a 3D volume with no preconceptions

Spectral Shapes from 7kV TSI analysis



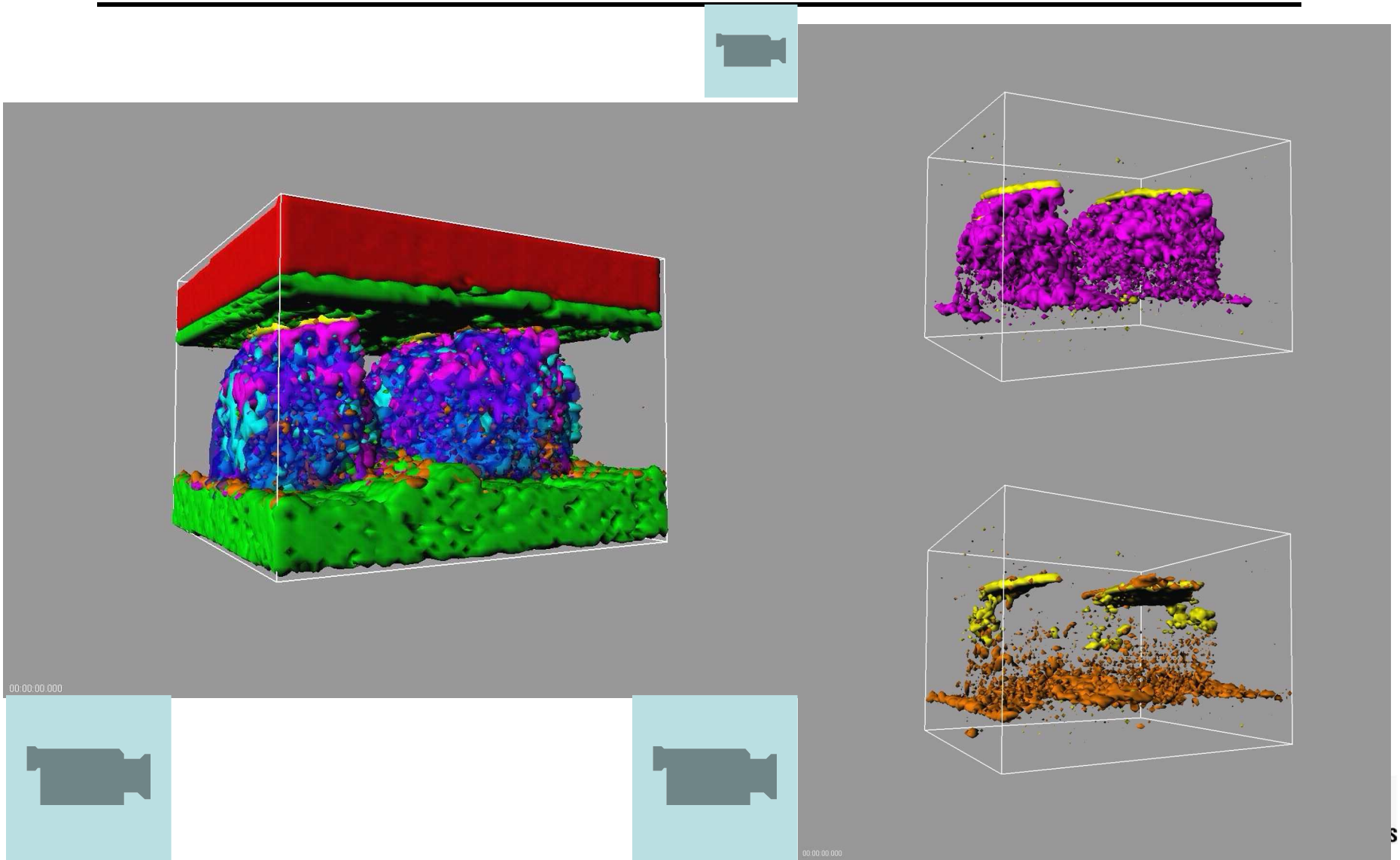
Sandia National Laboratories

Solder Joint Component Image Overlays



Red = Si
Green = Glass
Blue = Sn
Cyan = Pb
Magenta = Au-Sn
Yellow = Ni-P
Orange = Pt

3D Renderings of solder joints



Why Computed Tomographic Spectral Imaging?

- Spectroscopic computed tomography
  - Map known spectral features
  - Midgley, Weyland, Möbus
- Mapping, both x-ray and EFTEM is not comprehensive without a lot of work, also has potential for artifacts
- Complete spectrum from each pixel in 2D image for each projection
- All the chemical information from projections but comprehensive analysis of the multivariate data… (x,y,p,E) …is needed
- Reconstruction from unambiguous component images bearing correlations of various elements and at higher contrast than maps

Tomographic Spectral Imaging (TSI)

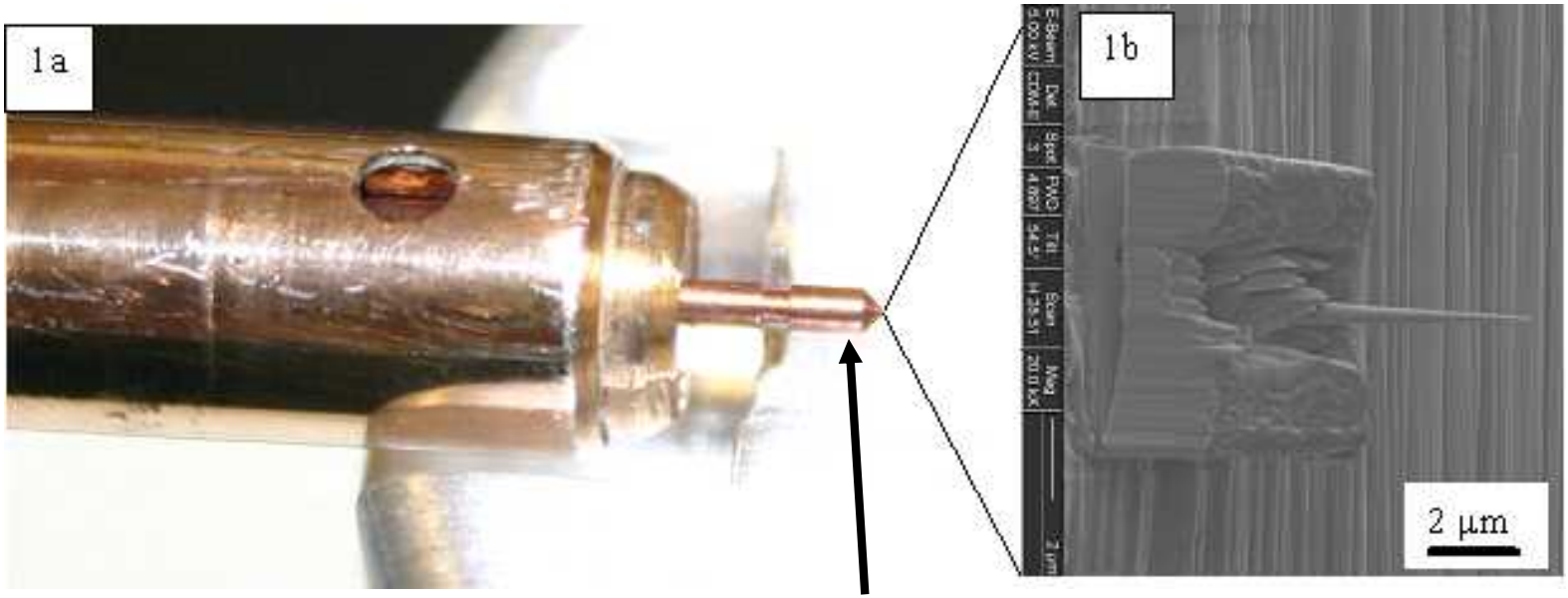
- **Acquisition parameters**
  - FIB-prepared needle of ODS (10-20nm alumina)-LIGA Ni, 100nm Ø at tip and 200nm Ø near base
  - Tecnai F30ST at 300kV, STEM mode with 3nm probe (FWTM), EDAX SUTW, TIA for acquisition
  - 2D x-ray spectral image acquired from each of 19 projections
    - 2000 nm by 400 nm area, 500 by 100 pixels (4nm/pixel)
    - 50msec dwell per pixel, about one hour per spectral image acquisition with drift correction
    - 19 hour total acquisition time (3 days in reality…manually acquired)
    - One million spectra
  - 10° intervals from -90° to +90°
  - No shadowing of EDS detector, specimen thickness is essentially constant in beam direction
  - $5x10^7 nm^3$ volume sampled

- **Multivariate Statistical Analysis**
  - Algorithms the same as used for 1D, 2D* and 3D data**
  - Data set analyzed as a whole in 1 minute on a Dual Itanium PC with 6GB RAM
  - 3 components found, 2 rendered here (left out caused by absorption of soft x rays)
  - Component images used for the reconstruction by FEI's Inspect 3D reconstruction software
  - Slices exported with Amira
  - Rendered with Imaris by Bitplane AG

- **Result is a comprehensive chemical analysis of a 3D volume with no preconceptions**

* P.G. Kotula, M.R. Keenan, and J.R. Michael. (2003) *Microsc. Microanal.* **9**, 1-17.
P.G. Kotula, M.R. Keenan, and J.R. Michael. (2006) *Microsc. Microanal.* **12, 36-48.

Tomography holder/FIB Preparation



Tip of the Fischione Model 2050 on-axis tomography holder

Atom-probe tip

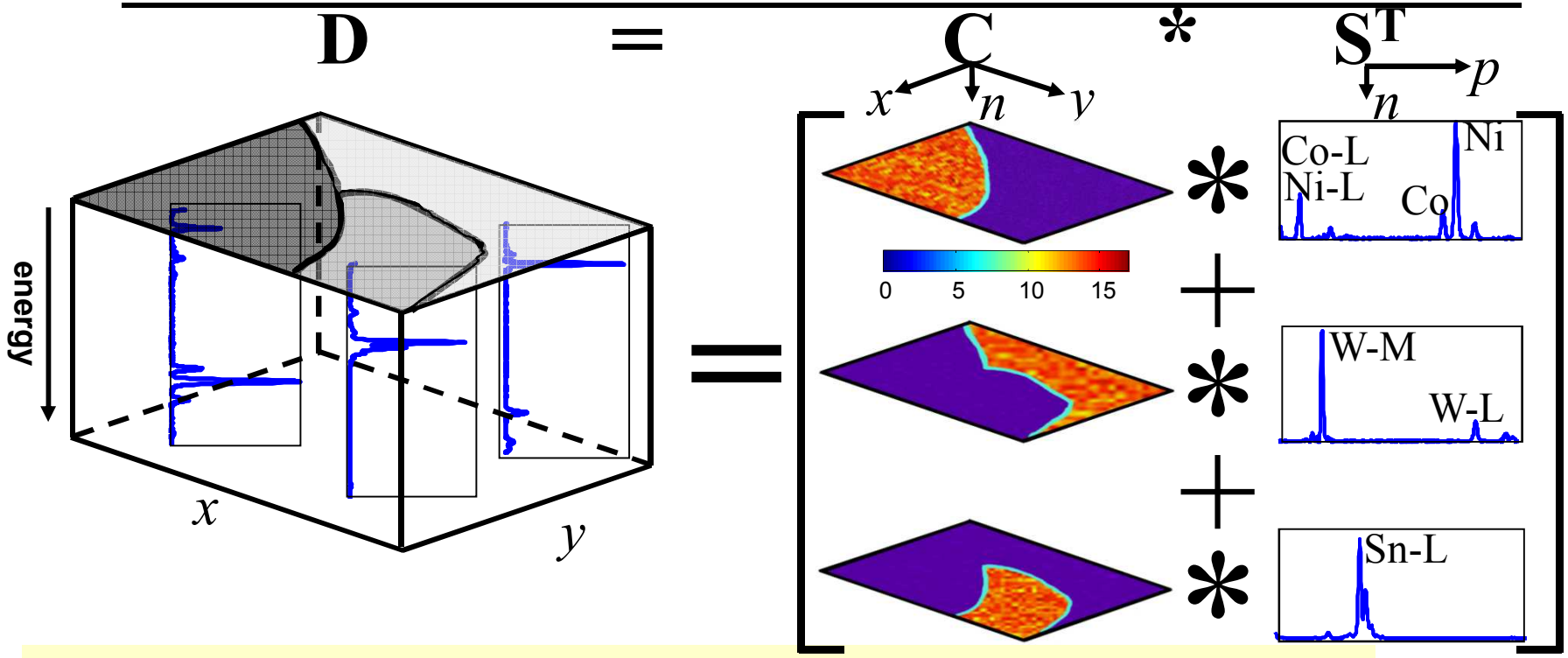
SEM image of the prepared needle

P.E. Fischione et al. (1989) *Colloque de Physique,* Colloque C8, Supplement au No. 11, Tome 50, November 1989, 36th International Field Emission Symposium, Oxford (Great Britain), Pages C8-555 to C8-560.

S.M. Schwartz and L.A. Giannuzzi, (2004) *Microsc. Microanal.* 10 (Suppl 2) 142-143.

Sandia National Laboratories

Multivariate Analysis:
All Methods Assume a Linear Additive Model

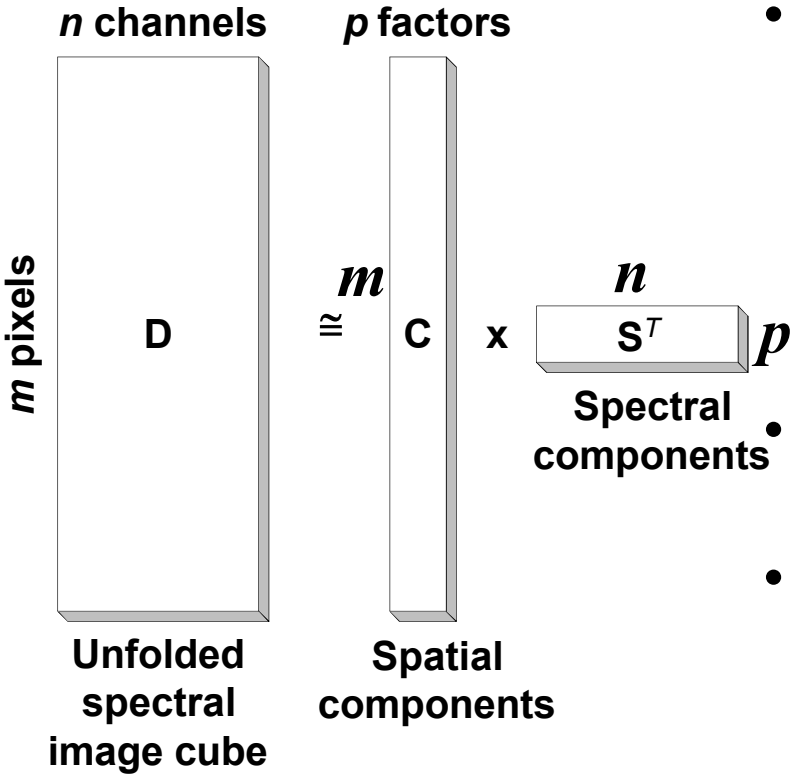


Multivariate Processing:

- **Scale data for Poisson counting statistics**
- **Determine the number of components to keep**
- **Factor the data matrix (D) into C and S**
- **Inverse scale the components**

Sandia National Laboratories

We have several options in our multivariate "Toolbox"

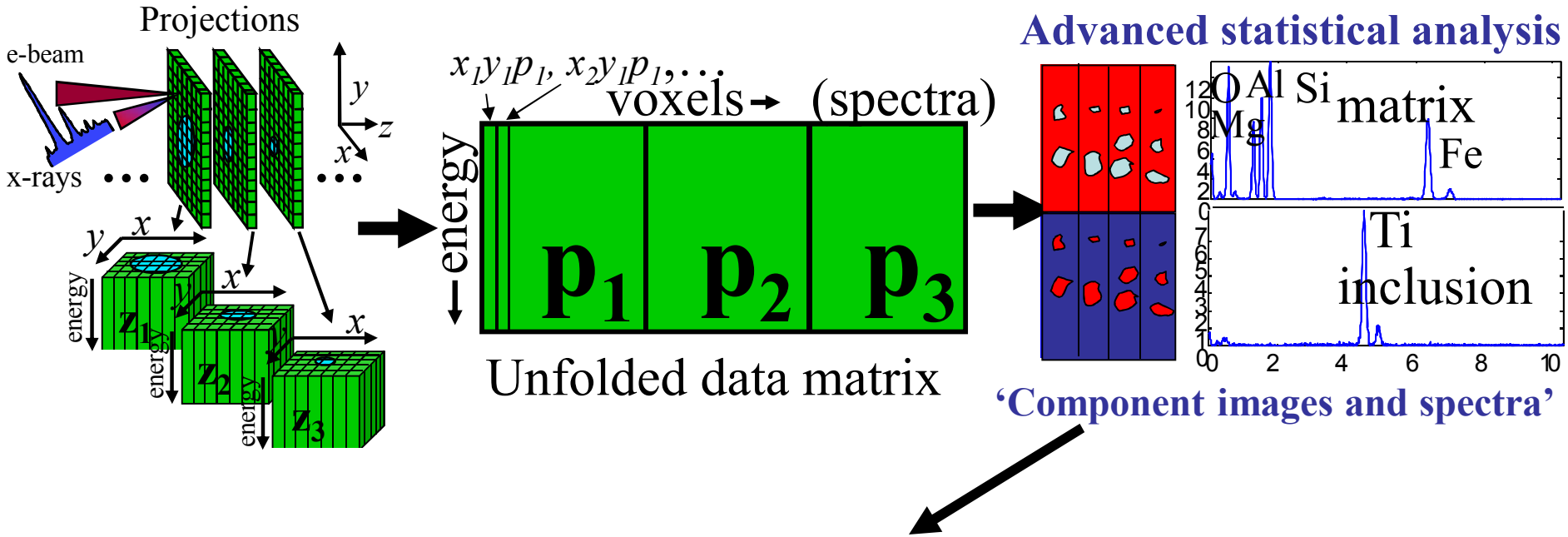


- Principal Component Analysis (PCA)
  - Factors are orthogonal
  - Factors serially maximize variance
  - Provides best LS fit to data
  - Non-physical constraints:
  - Factors are abstract
- PCA + factor rotation (Varimax)
  - Rotate factors to "simple structure"
- MCR-ALS
  - A refinement of Rotated PCA
  - Non-negativity of C and/or S
  - Equality, closure and others
  - Constraints may not be effective

Analysis goal: Obtain an easily interpretable representation of the data

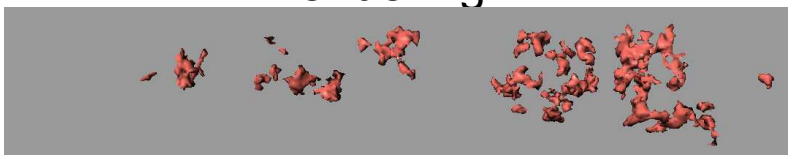
Sandia National Laboratories

Tomographic Spectral Imaging and Multivariate Statistical Analysis



Reconstruction of component images into 3D model (Inspect 3D)

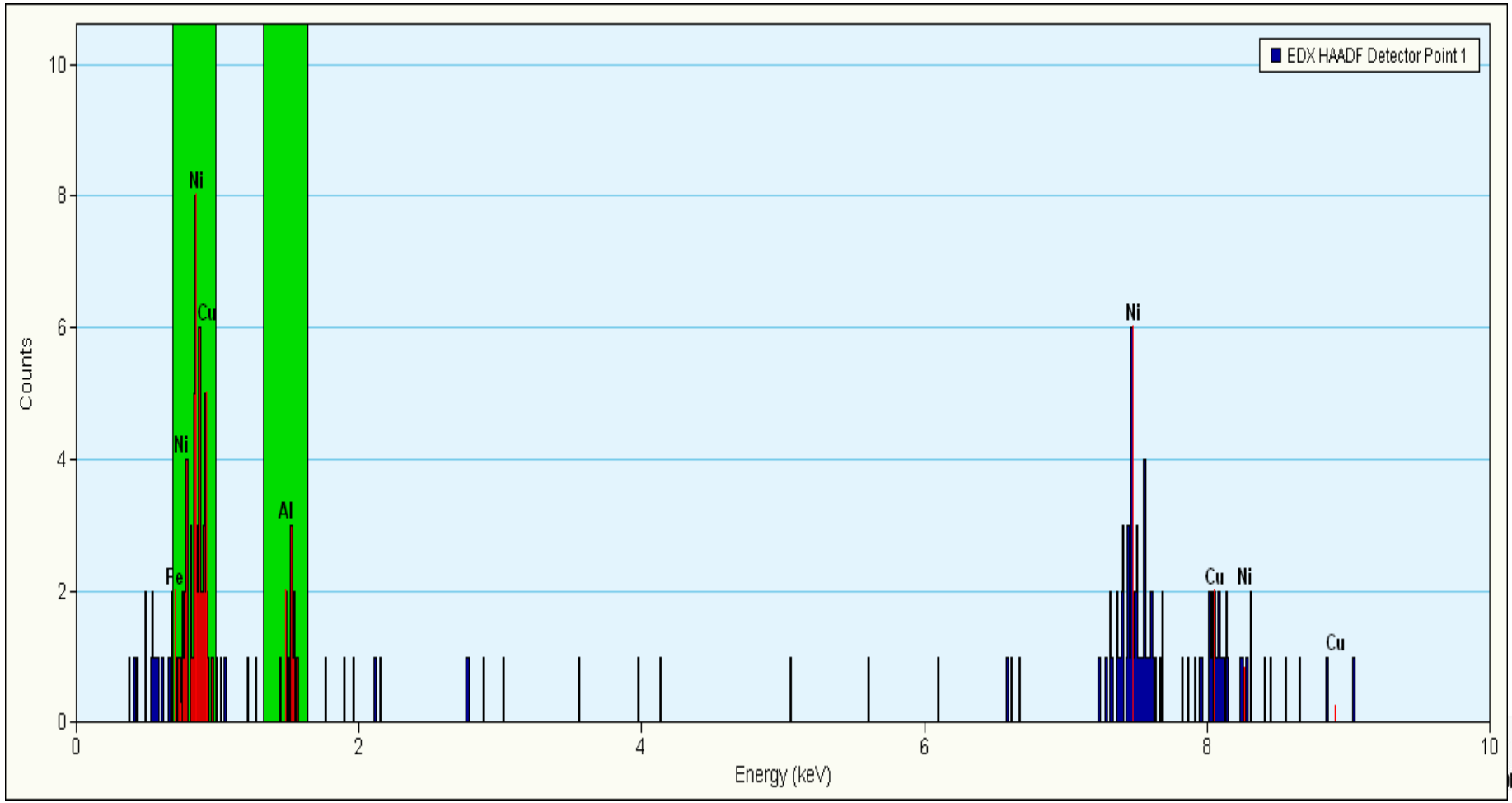
Rendering



Sandia National Laboratories

Raw spectra have low signal

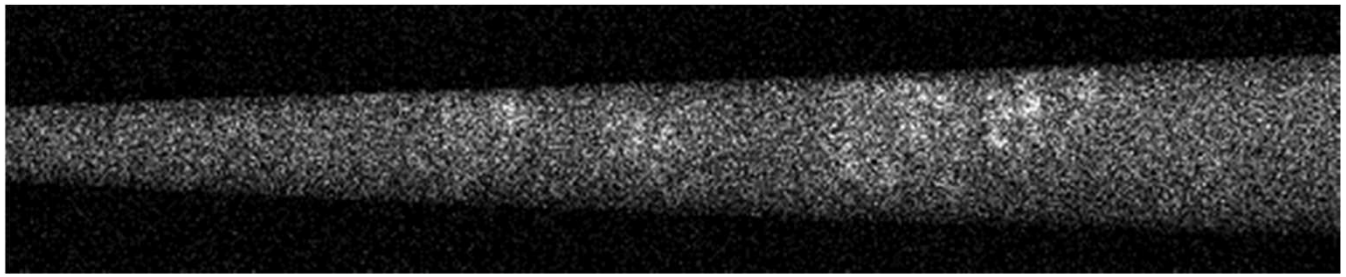
Spectrum with beam on an alumina particle is mixed due to projection



Raw maps have low signal

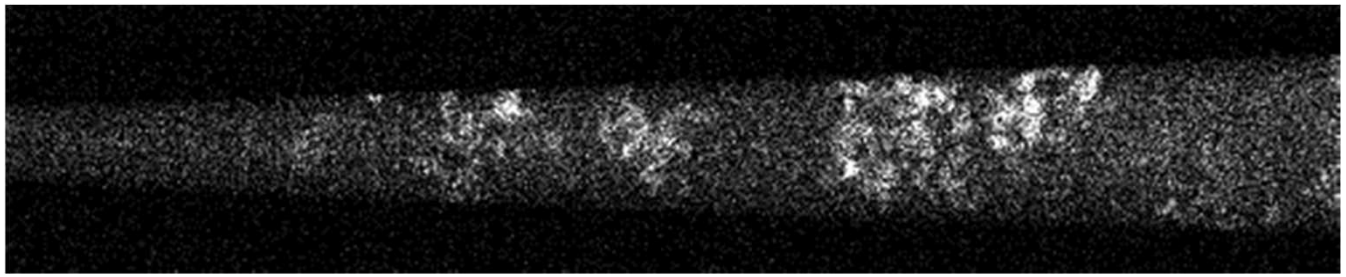
Potential also for artifacts

O-K



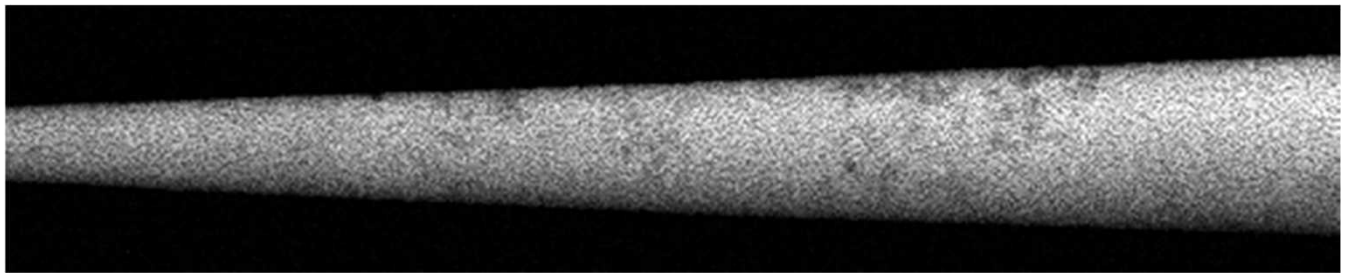
0-13 counts

Al-K



0-11 counts

Ni-L



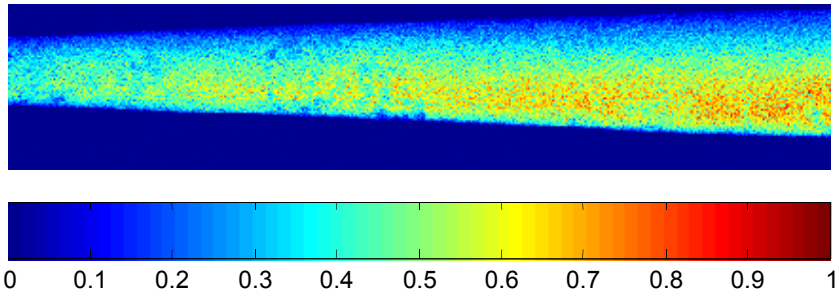
0-86 counts

Sandia National Laboratories

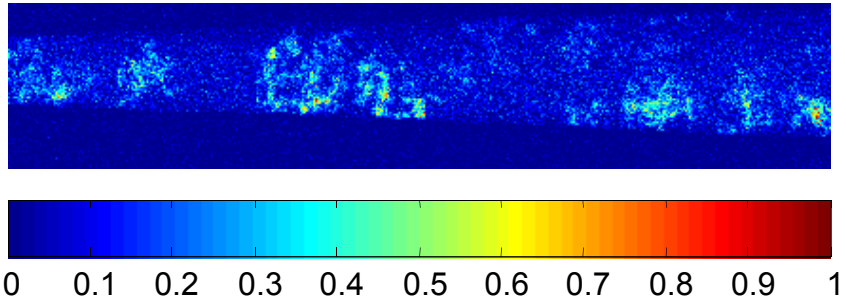
MAS results from one slice

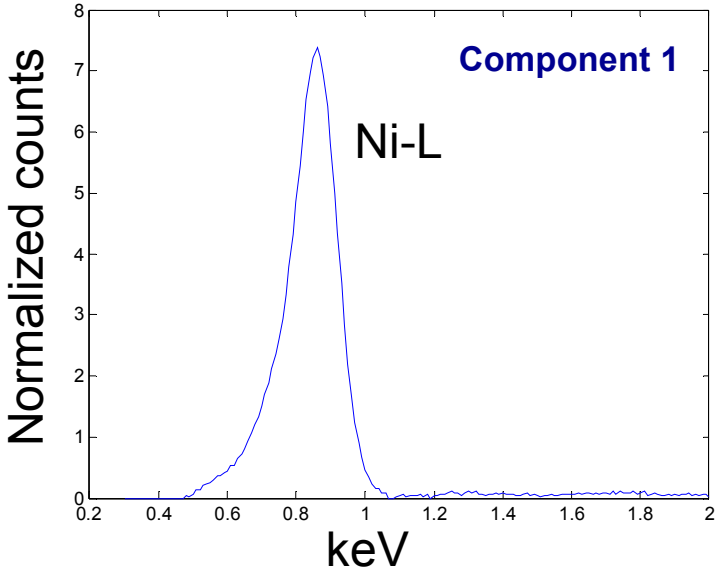
Over 25 counts full-scale, all chemically relevant…more than twice those in the map

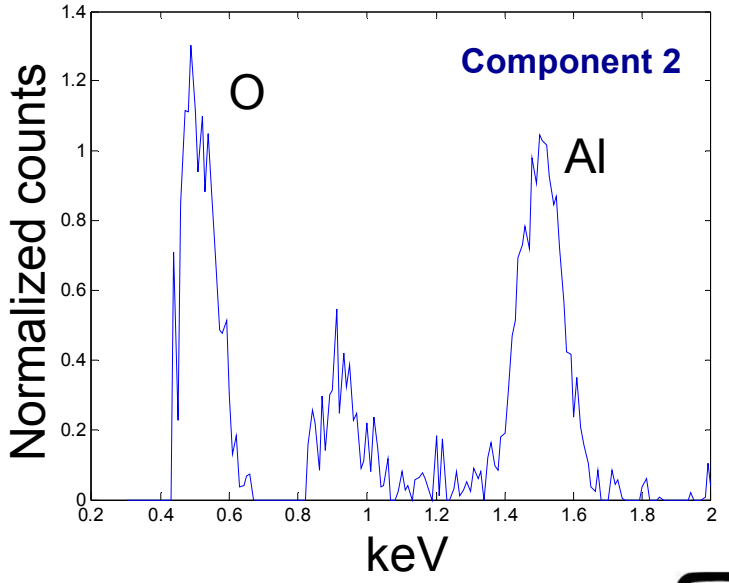
Ni component image



Alumina component image







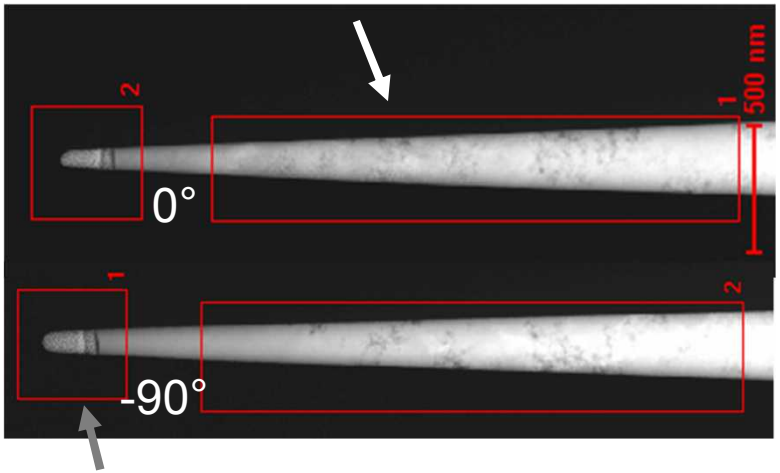
MSA has successfully deconvolved these overlapped signals on a sub-pixel basis

Sandia National Laboratories

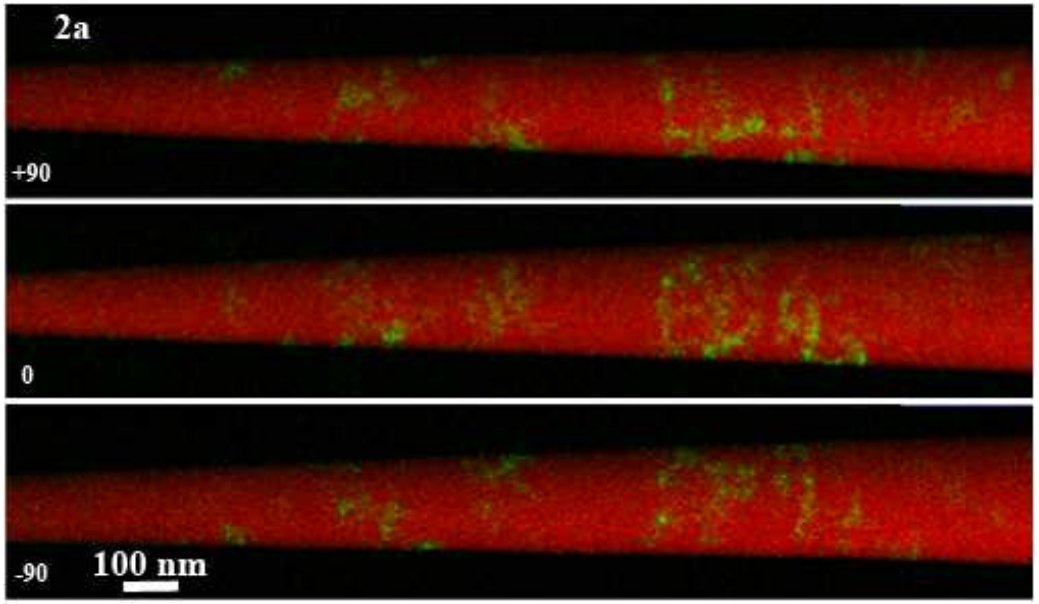
MSA of the entire projection series

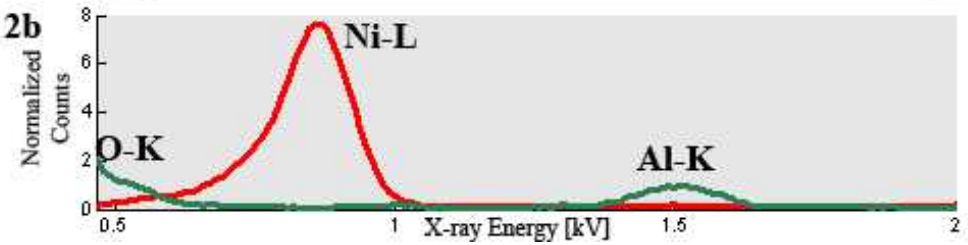
Color overlays of component images

Region of spectral images 2000nm x 400nm



Drift-correction region

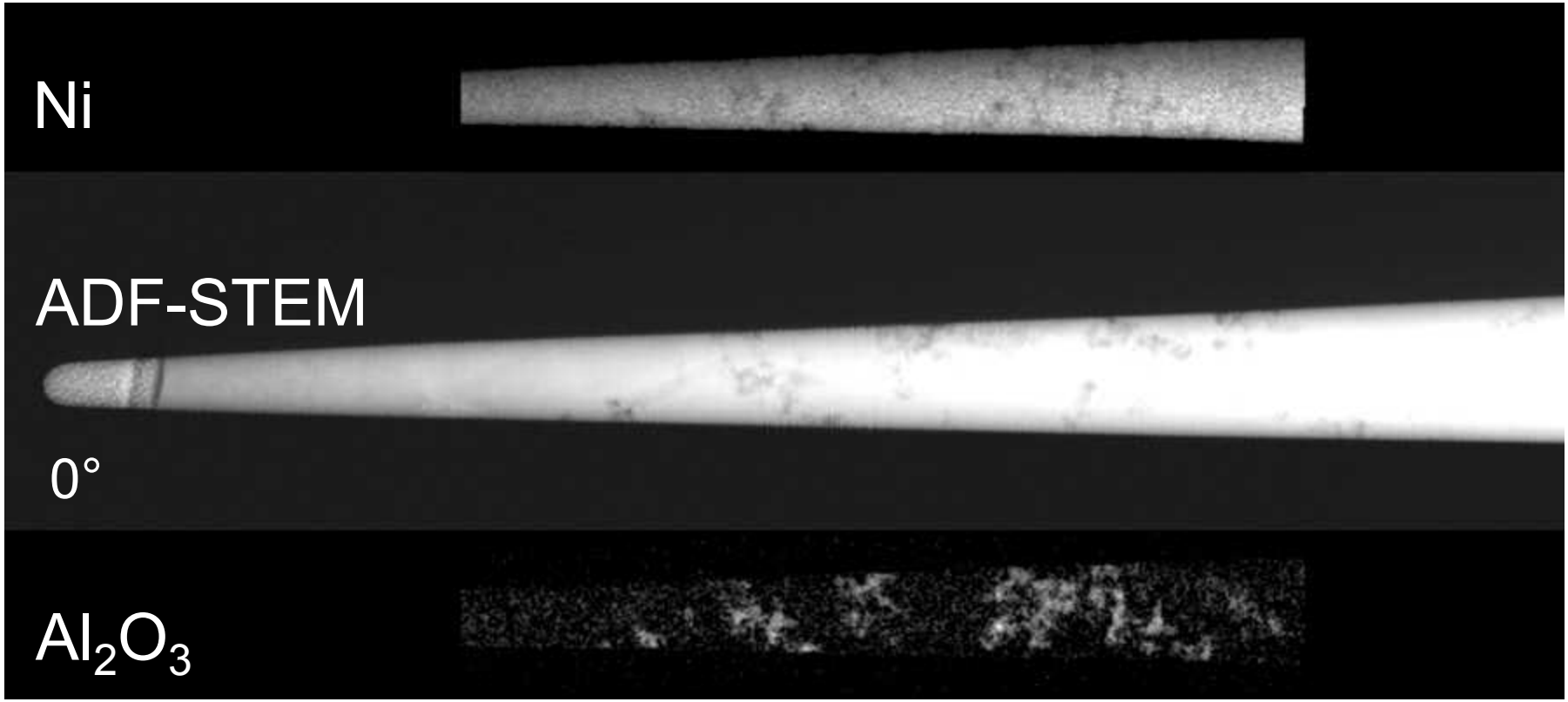


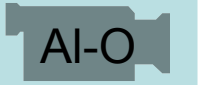


Sandia National Laboratories

90° to -90° in 10° increments

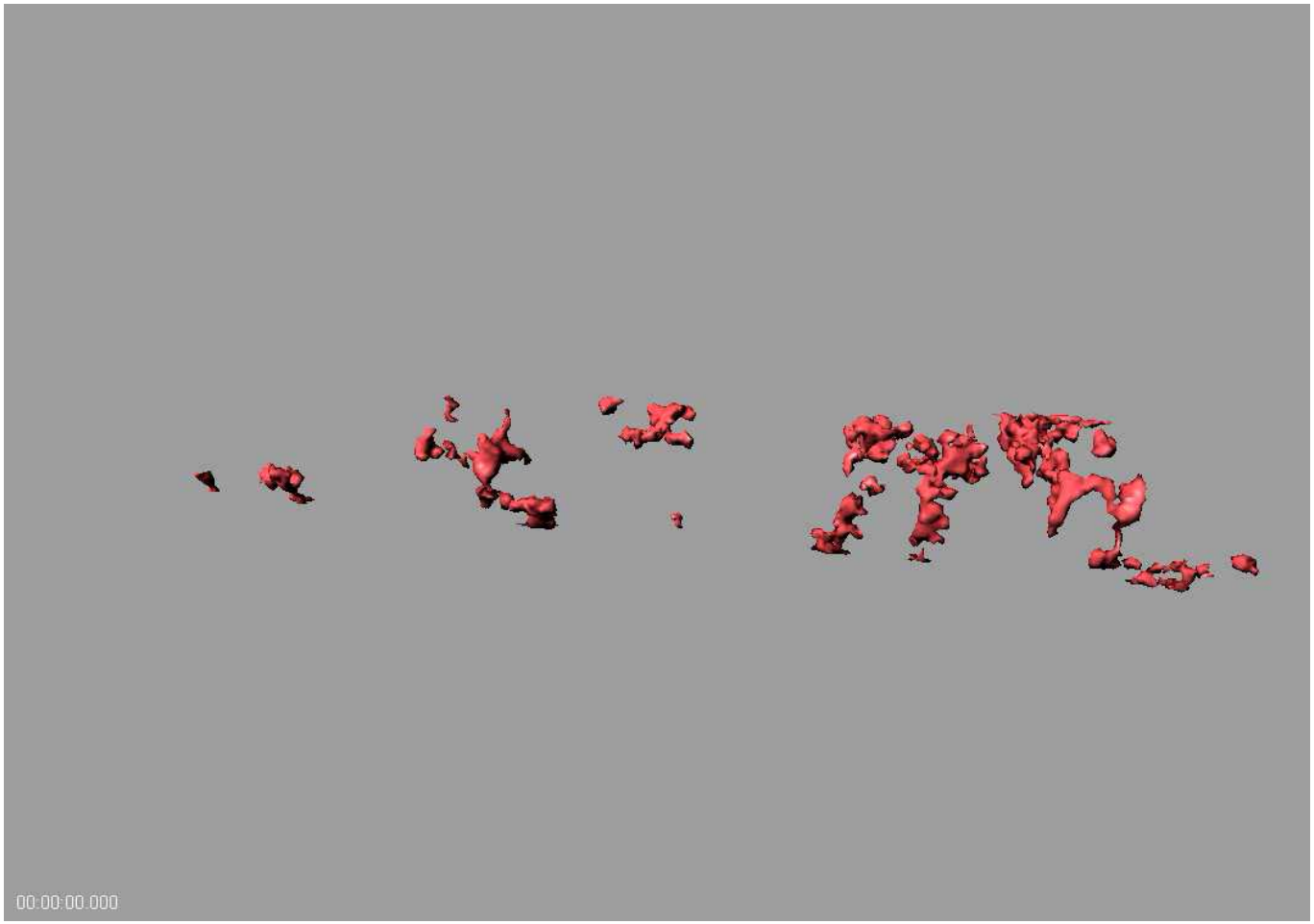
MSA results by projection (not reconstructed)



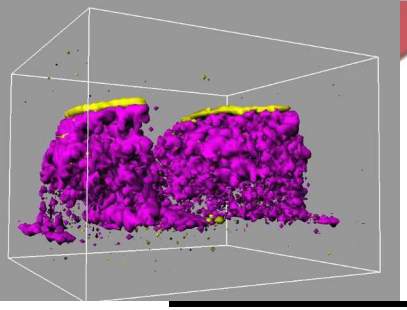


Sandia National Laboratories

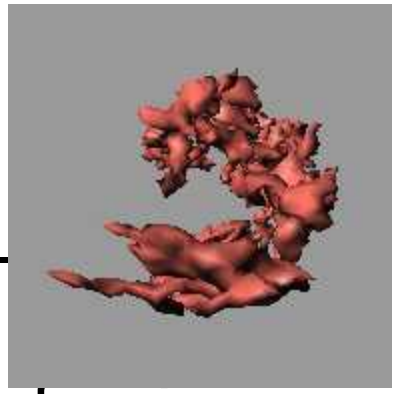
Reconstructed isosurface of the alumina particles



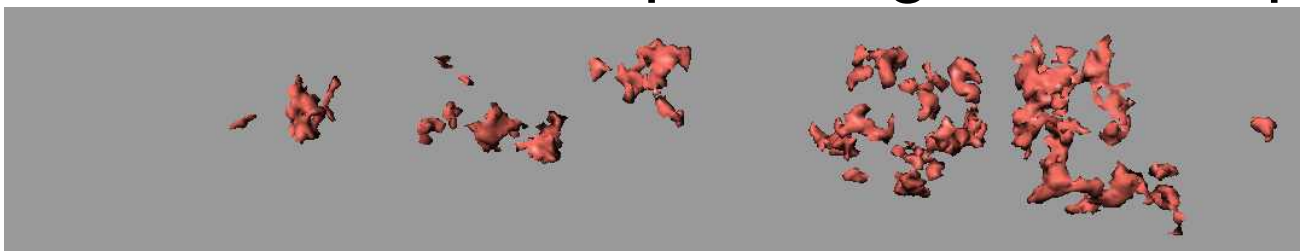
Sandia National Laboratories



Conclusions



- Unambiguous chemical contrast
- MSA takes advantage of large amounts of noisy data…statistically aggregating signal
- Noisier data would work just as well
- Sub-pixel spectral deconvolution
- Better reconstructions than possible with maps…map intensity includes Bremsstrahlung from other elements overlapping in projection
- MSA can deconvolve pathological overlaps



Sandia National Laboratories