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Electron-Beam Microanalysis for Failure Analysis

Dr. Patrick Camus

Failure Analysis - Definition

- Investigation of a material to determine the root cause of unexpected performance.
- Investigation includes imaging and may include chemical analyses.
- Imaging analysis can include a range of magnifications and a number of incident radiations.
- Chemical analysis could be in-situ, specimen removal, or complete “destruction” of sample.

Failure Analysis Techniques

- Typical imaging techniques include:
  - Visual inspection
  - Optical microscopy
  - Ultrasonic
  - X-ray imaging
  - Electron Beam microscopy
- Chemical analyses include:
  - X-ray emission
  - Optical emission
  - Chemical dissolution
  - Plasma / optical

Electron-Beam Techniques

- Scanning electron microscopy (SEM) Imaging
  - Advantages are large depth of focus of image
  - Spatial control of electron beam
- Energy dispersive spectroscopy (EDS) X-ray elemental analysis
  - Be to U detection at reasonable spectral resolution
- Wavelength dispersive spectroscopy (WDS) X-ray elemental analysis
  - High spectral resolution but serial collection
- Electron back-scattered diffraction (EBSD) crystallography analysis
  - Orientation and phase analyses in SEM

Purpose of Webinar

- Application of modern electron-beam microanalysis techniques to investigate the microstructure and microchemistry anomalies that might have contributed to a failure in 2 metal samples.

Equipment

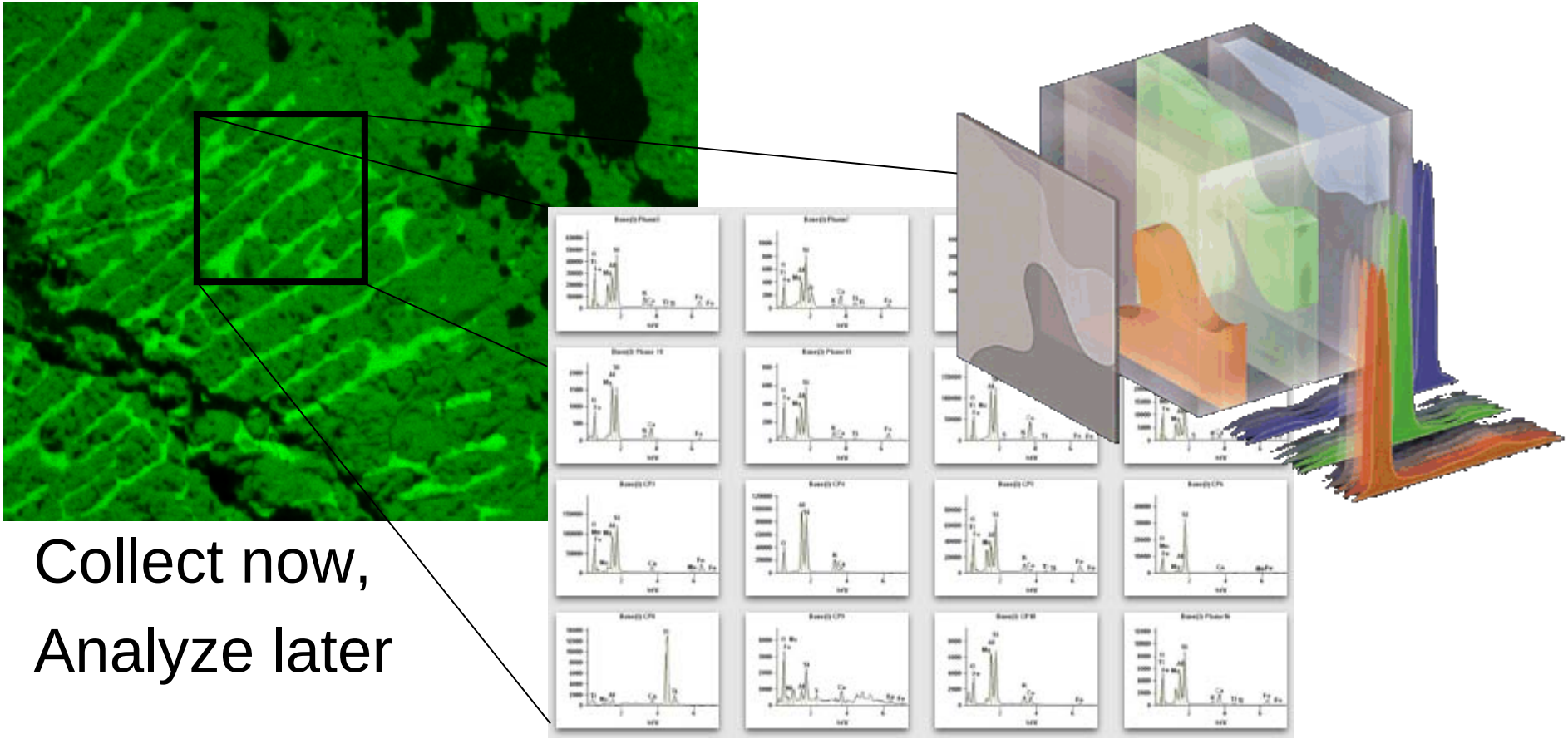
- Field-Emission (FE) SEM
- NORAN System 7 microanalysis system
- UltraDry EDS detectors
  - Dual 30 mm^2 active area SDD mounted 90 from each other (SW and SE on electron image)
- MagnaRay WDS spectrometer
- QuasOr EBSD detector

Analysis Methods

- EDS
  - Spectral Imaging
    - Elemental mapping
  - Phase analysis
- WDS
  - Spectral acquisition
  - Mapping acquisition
- EBSD
  - Crystal identification
  - Phase mapping

Spectral Imaging Definition

- Electron image with x-ray data cube
  - Full x-ray spectrum at each scan location (position in map)

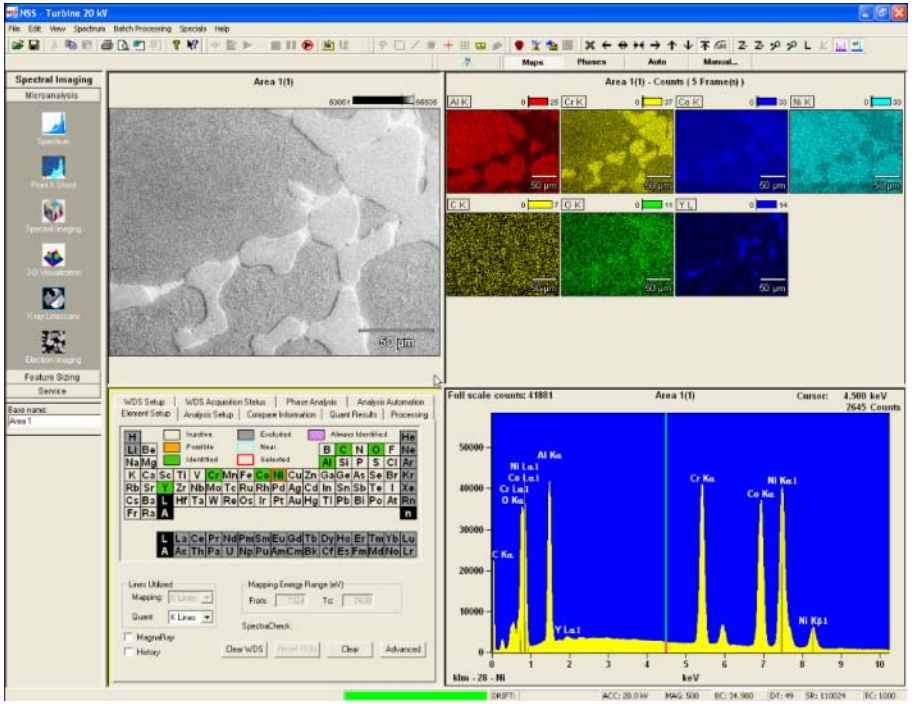


Collect now,

Analyze later

Spectral Imaging Acquisition

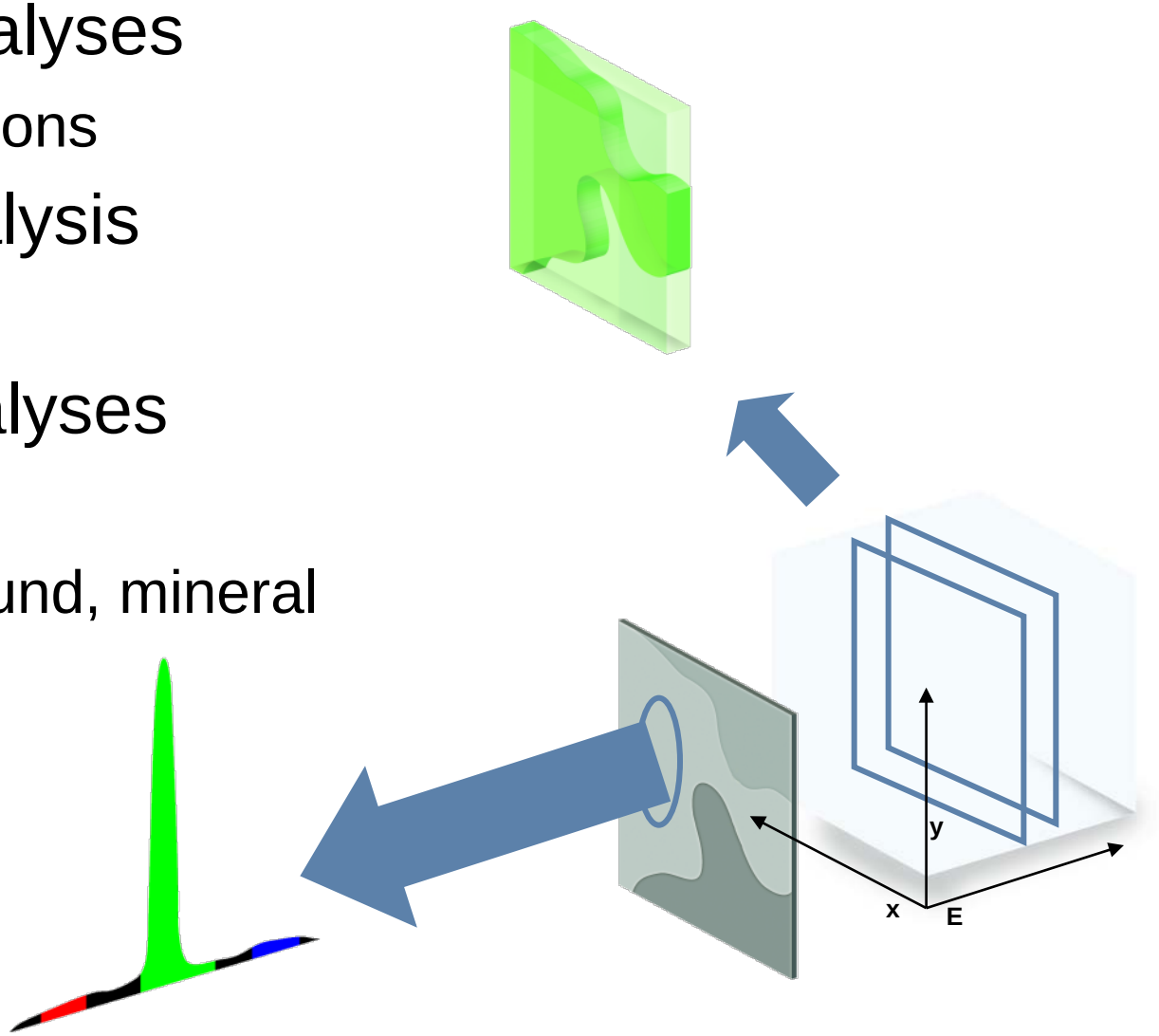
- Multi-frame collection
- Acquisition times are now measured in minutes, possibly seconds.



An analyst can cover more area in less time.

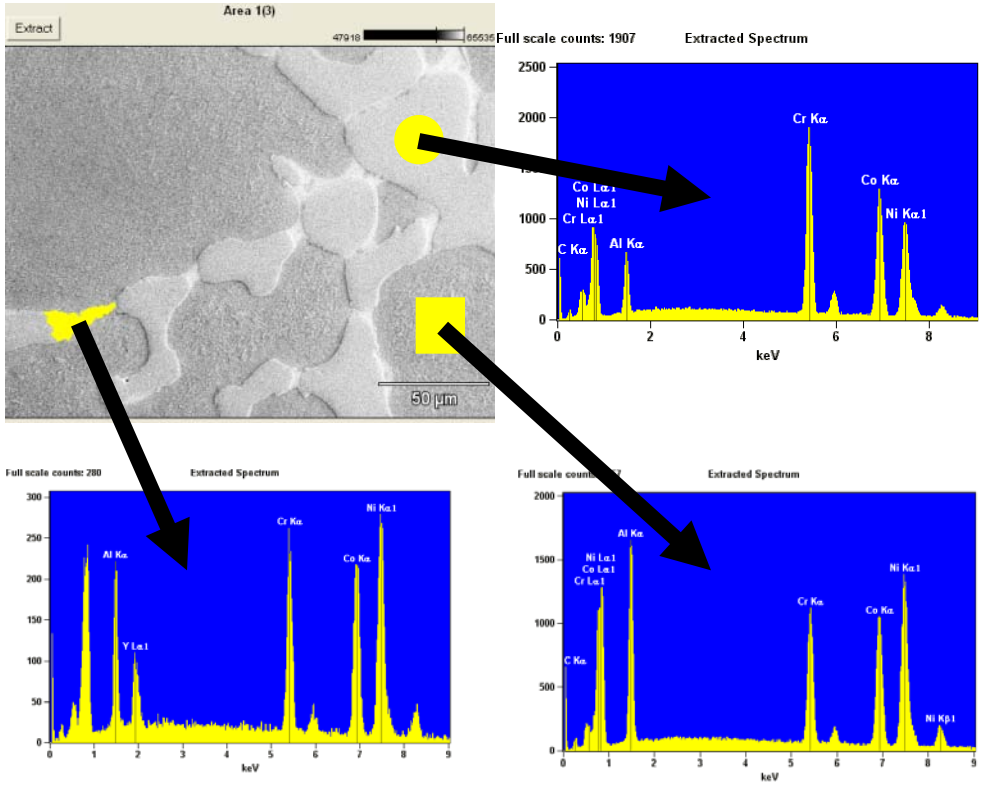
Methods of Spectral Imaging Analysis

- Image-plane analyses
  - Spectral extractions
- Energy-axis analysis
  - Elemental maps
- Whole-cube analyses
  - Phase analysis
    - Alloy, compound, mineral

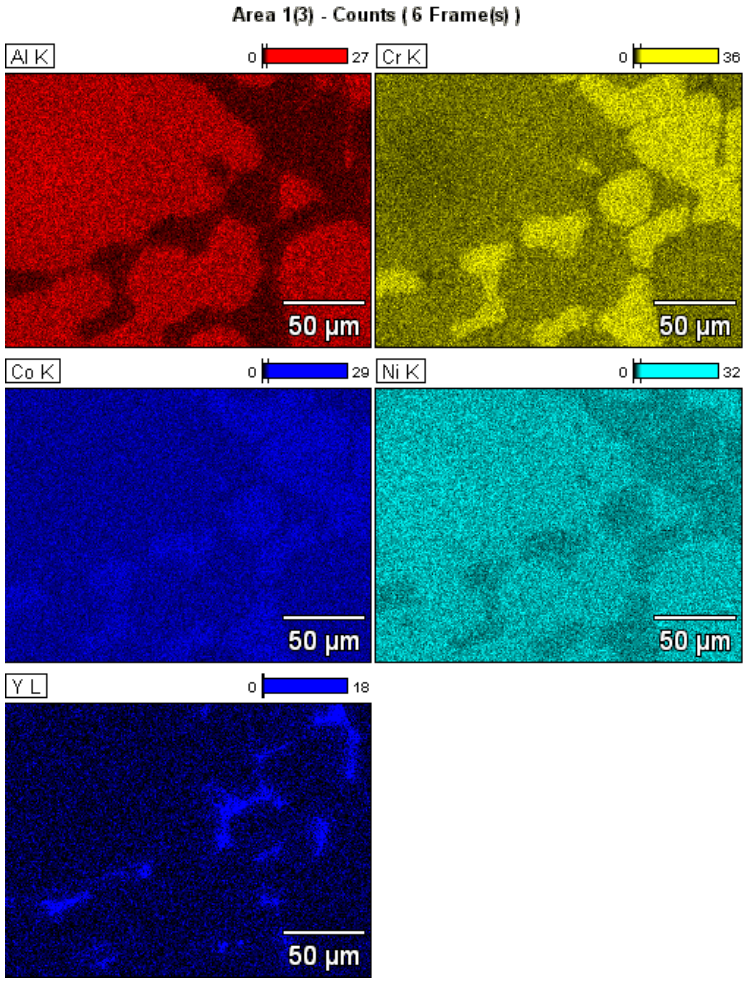


Traditional Spectral Imaging Analyses

- Image-plane analyses
  - Spectral extractions



- Energy-axis extractions
  - Elemental maps



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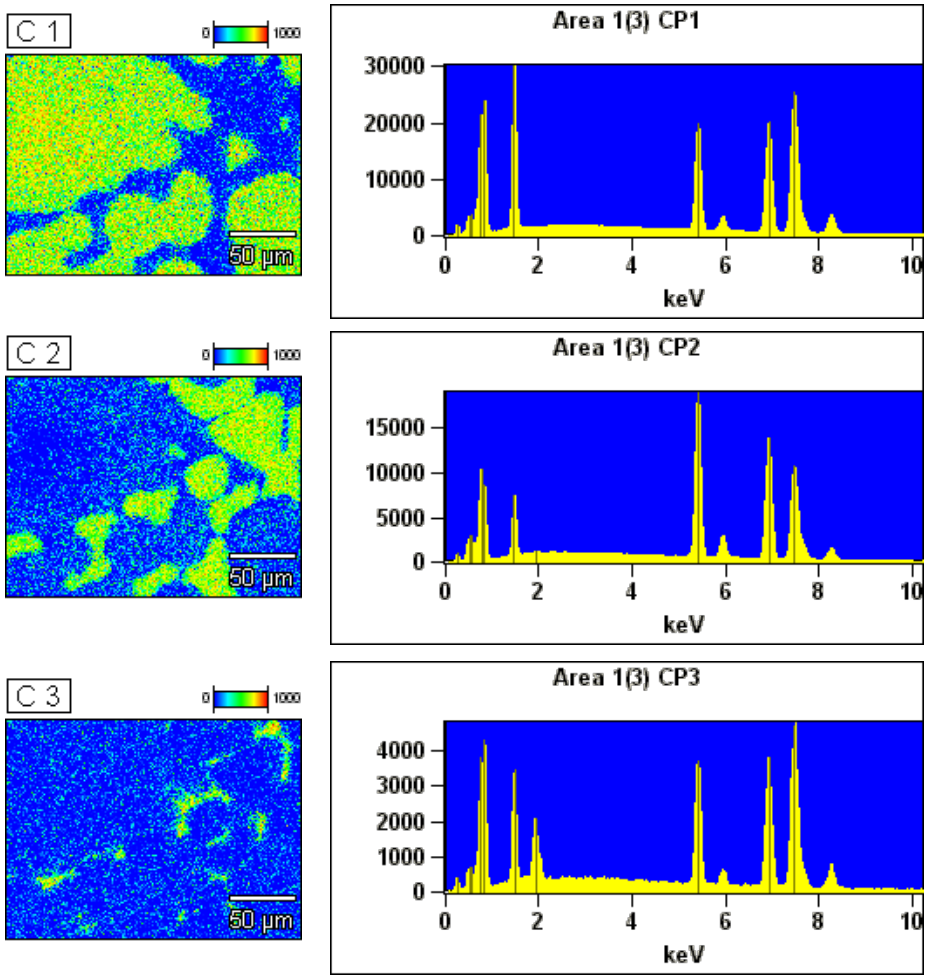
Automated Phase Analysis

- Multivariate Statistical Analysis (MSA) + Phase isolation + Phase labeling
  - No user-selection of elements and maps necessary
    - Whole spectra are analyzed.
  - Peak overlaps are typically not a limitation
  - Designed for low intensity (short acquisition time, 50-200 cpp) data sets
  - Low-pixel count phases have equal weighting.
  - Phase labeling compares phase spectra with user database of labeled spectra
  - Every analyst obtains the same answer
    - Decades of experience are not required to obtain the correct answer

The analyst has the highest confidence that every unique feature is found in the data set

COMPASS MSA Results

- Whole SI data-cube reduced to pairs of Map-Spectrum results
  - Each unique alloy, compound, or mineral spectrum is assigned to a map
- High confidence that every analyst gets the same answer, the right answer



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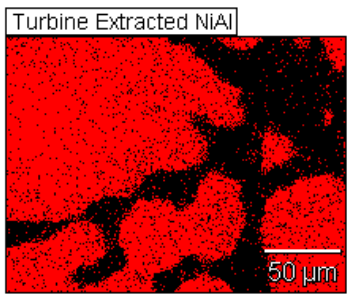
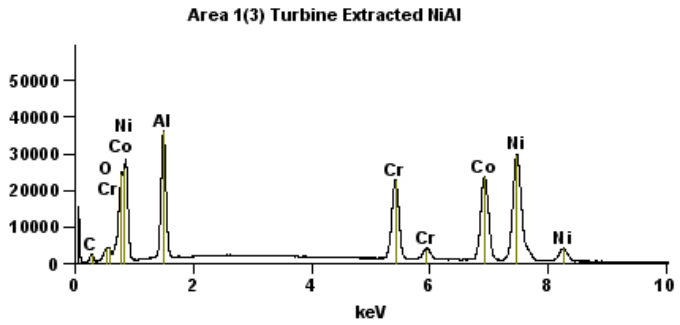
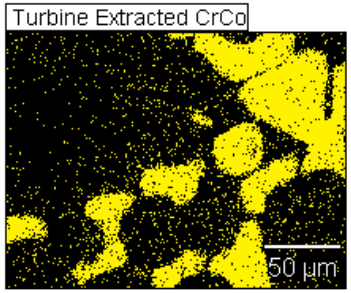
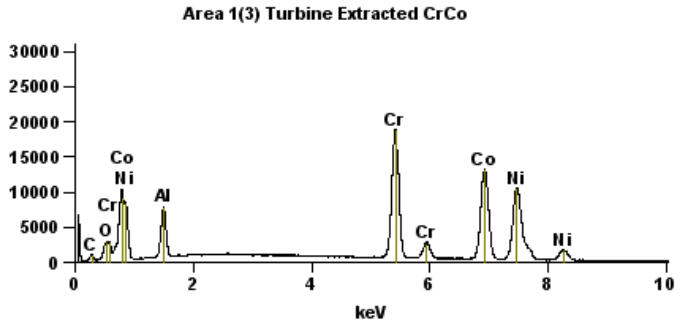
Phase Results

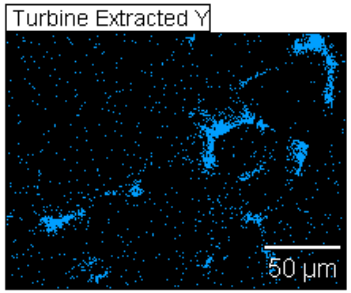
- Binary maps of alloy, compound, or mineral distributions from COMPASS
- Spectra extracted from SI data cube
- Phase labels defined by best match to database

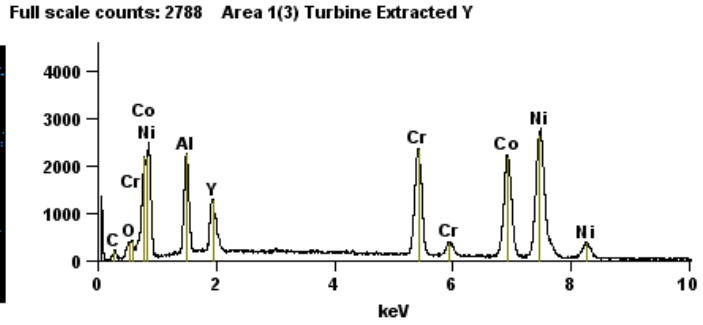












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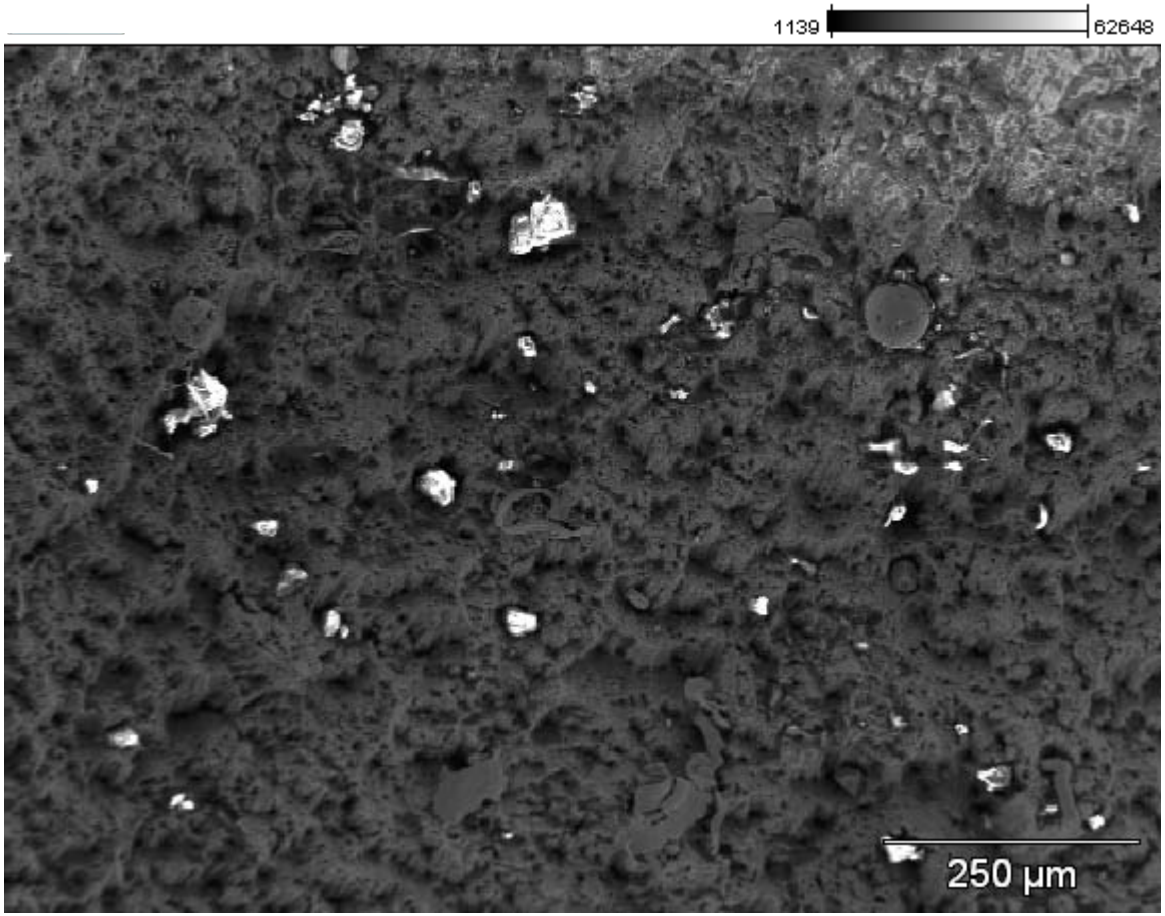
Microanalysis of a Failed Spring

Experimental Conditions

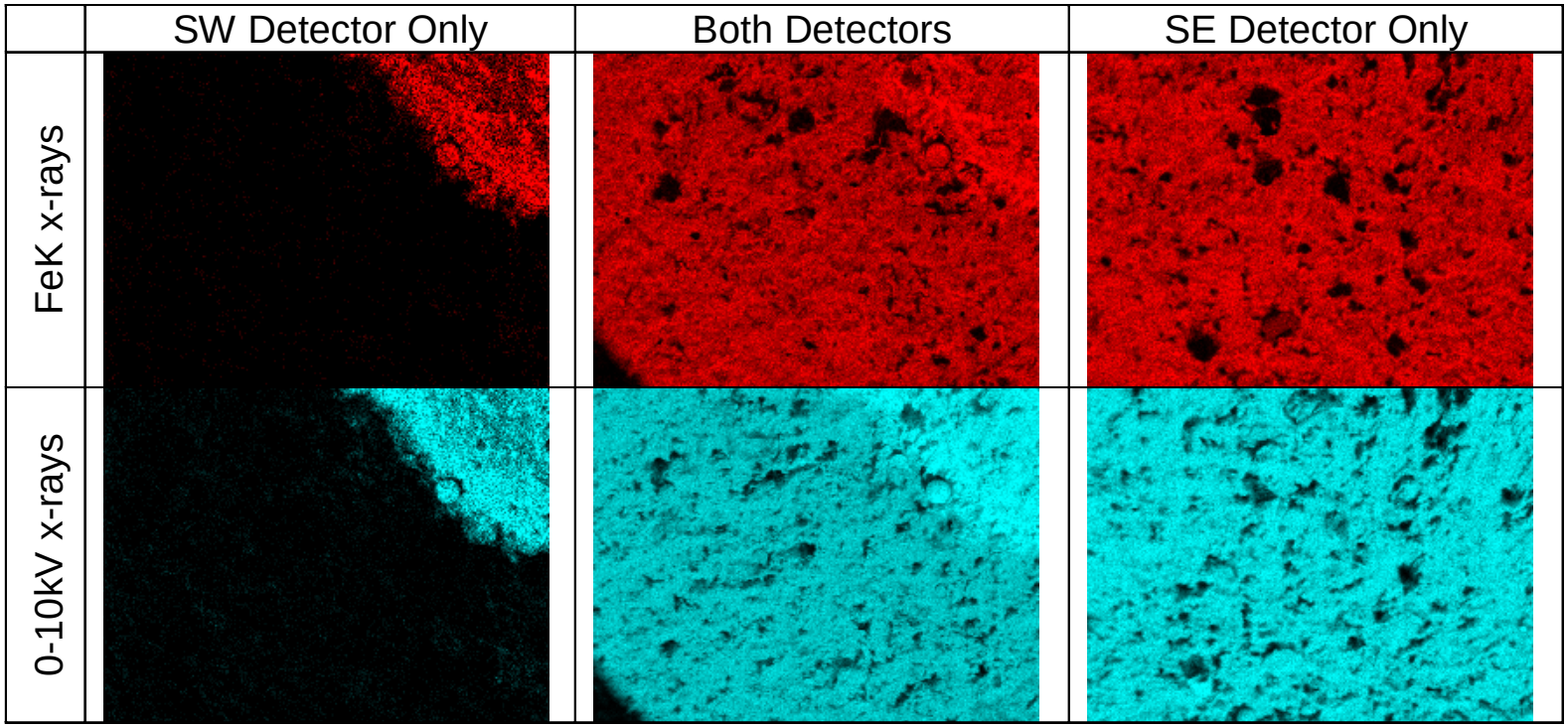
- 20 kV beam voltage
- Unmounted fractured sample
- 12 nA Beam current
- 45% electronics deadtime
- 76k cps Output count rate from dual EDS detectors

- Acquisition types, termination criteria and times
  - EDS Spectral
    - 10k VFS = ~ 1-3 seconds
  - WDS Spectral
    - 2 eV steps for 1 second
  - EDS Spectral Imaging
    - 100 x-ray counts per pixel => ~ 70-90 seconds acquisition
    - Phase analysis takes additional 1 minute

Electron Image

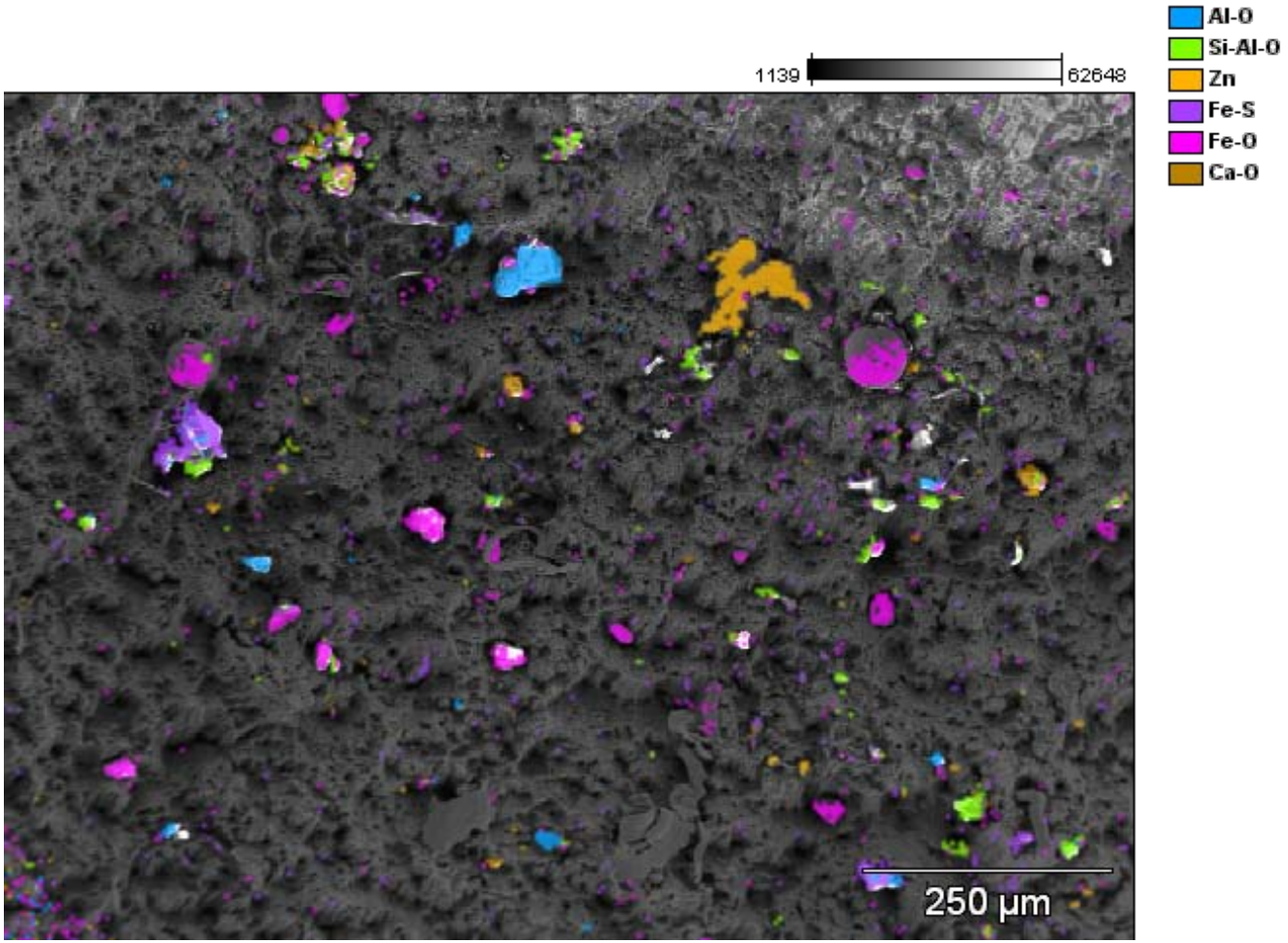


Effect of Surface Roughness on EDS Maps



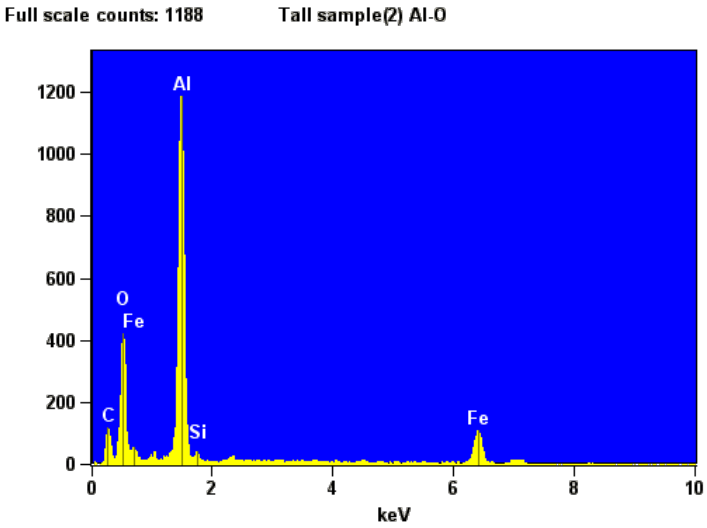
- Coarse fracture surface
  - Huge potential shadows in x-ray maps
  - Understanding Chamber+Detector geometry is critical
- Subtle surface roughness
  - Intensity variation or contrast is not composition effect
- Dual-EDS detectors reduce apparent surface effects

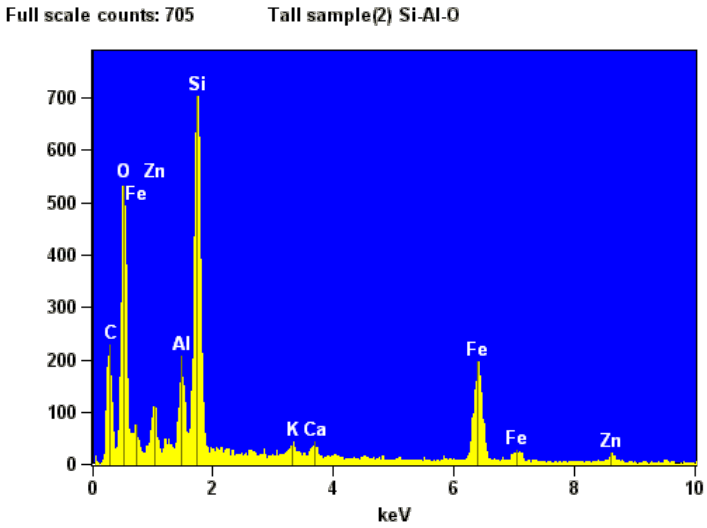
Spectral Imaging Phase Maps

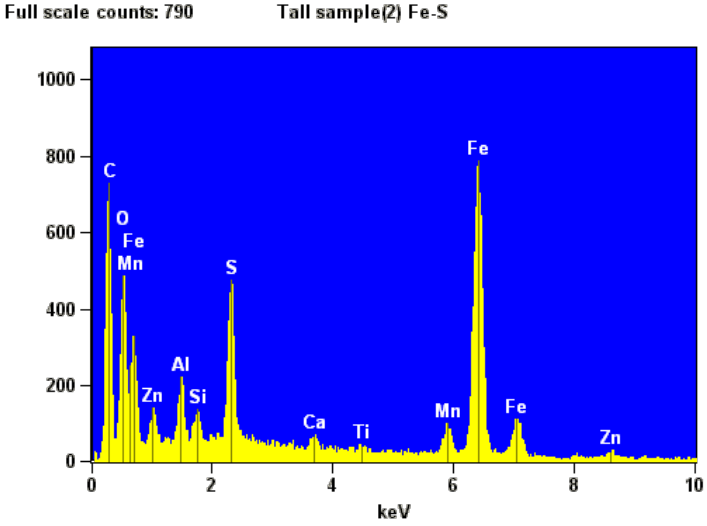


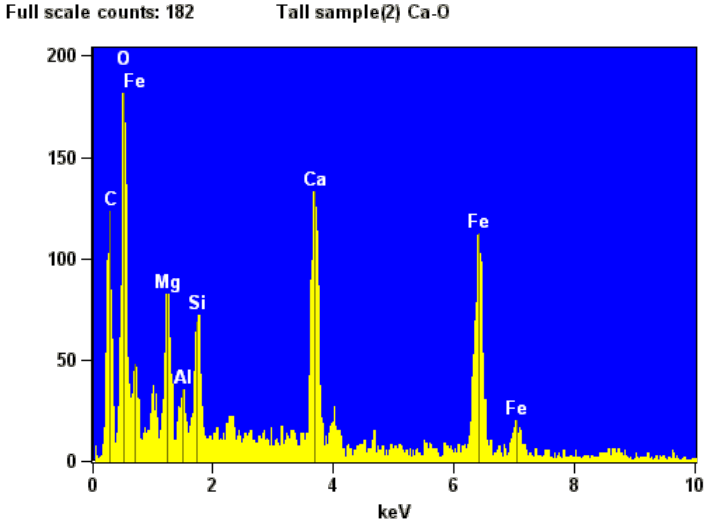
- Many different type of particles in many locations.
- Mapping is very efficient in finding all of the unique particles.

Phase Spectra









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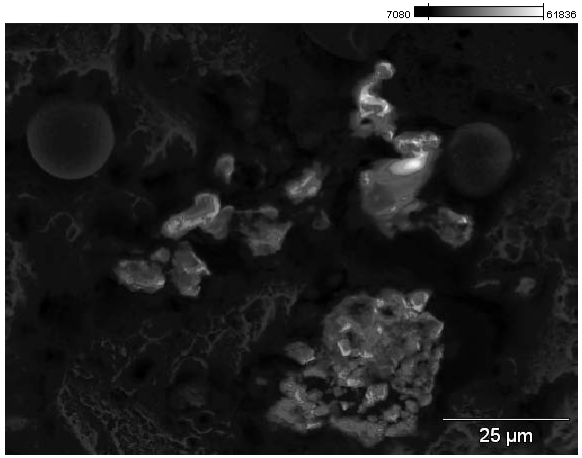
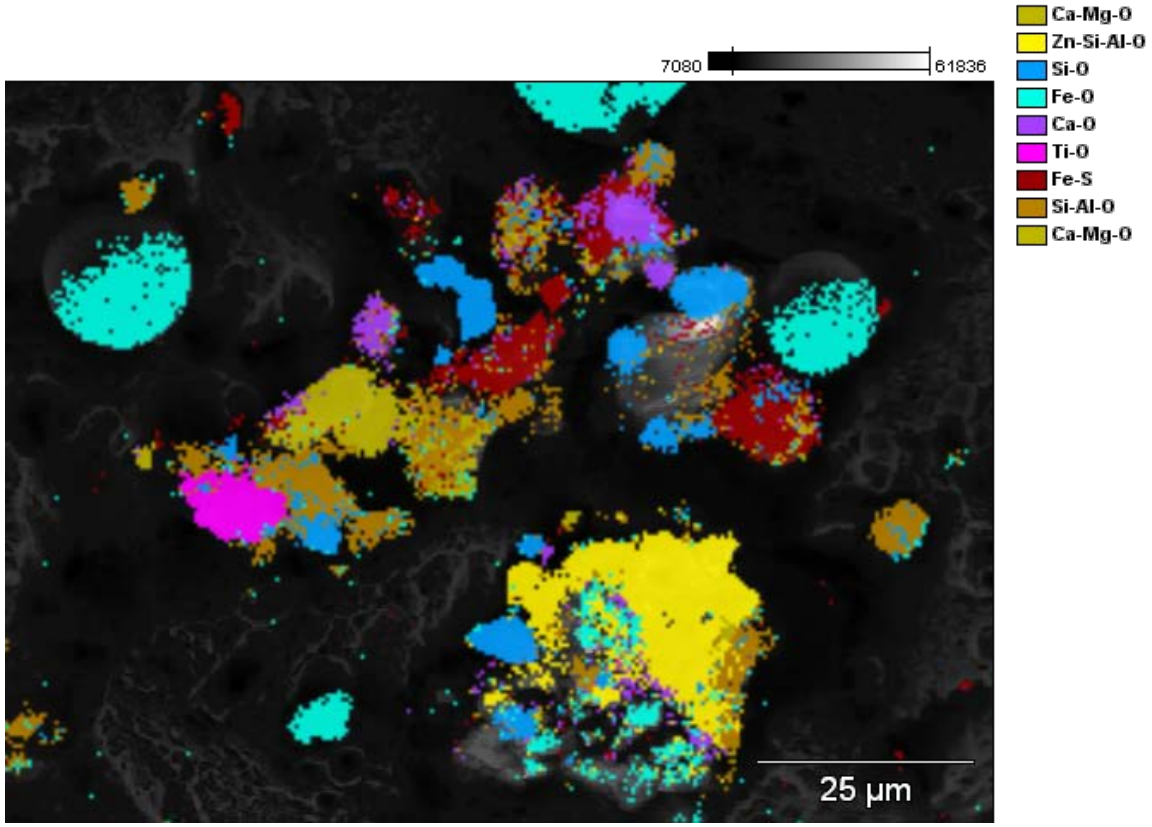
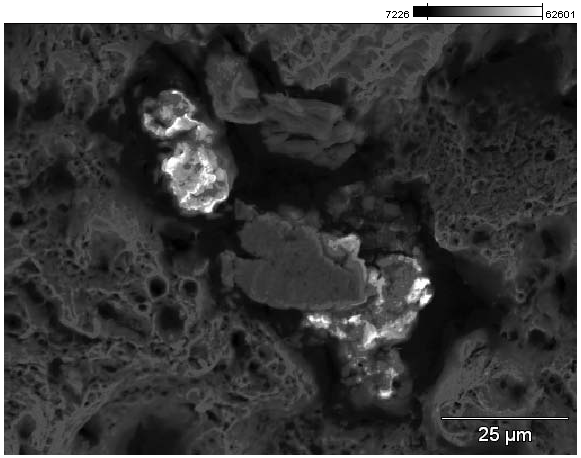
Multiple Particles 1

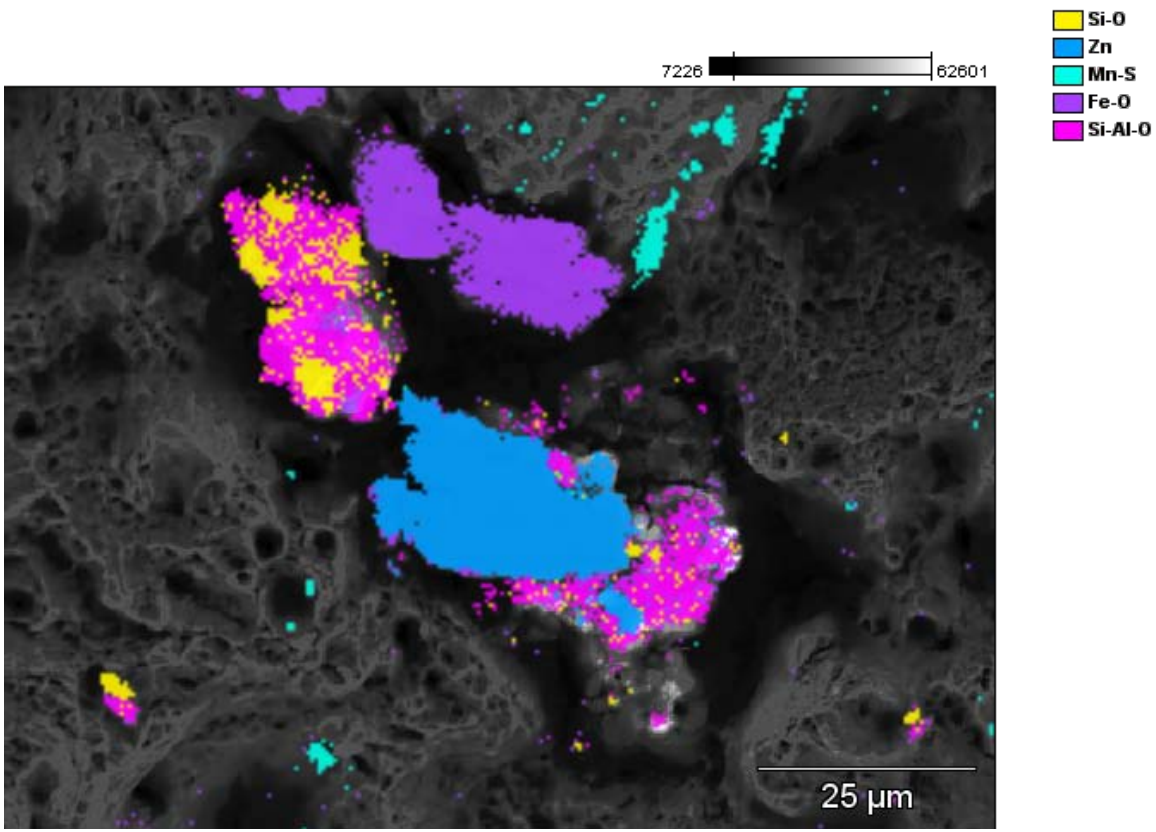




- Phase analysis finds all compounds.
- Point analyses may miss some compounds.

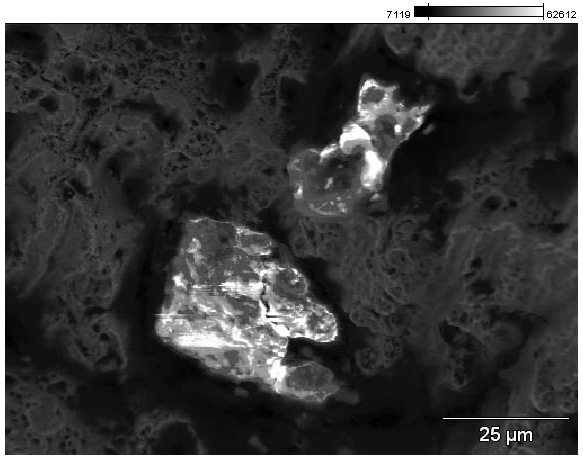
Multiple Particles 2

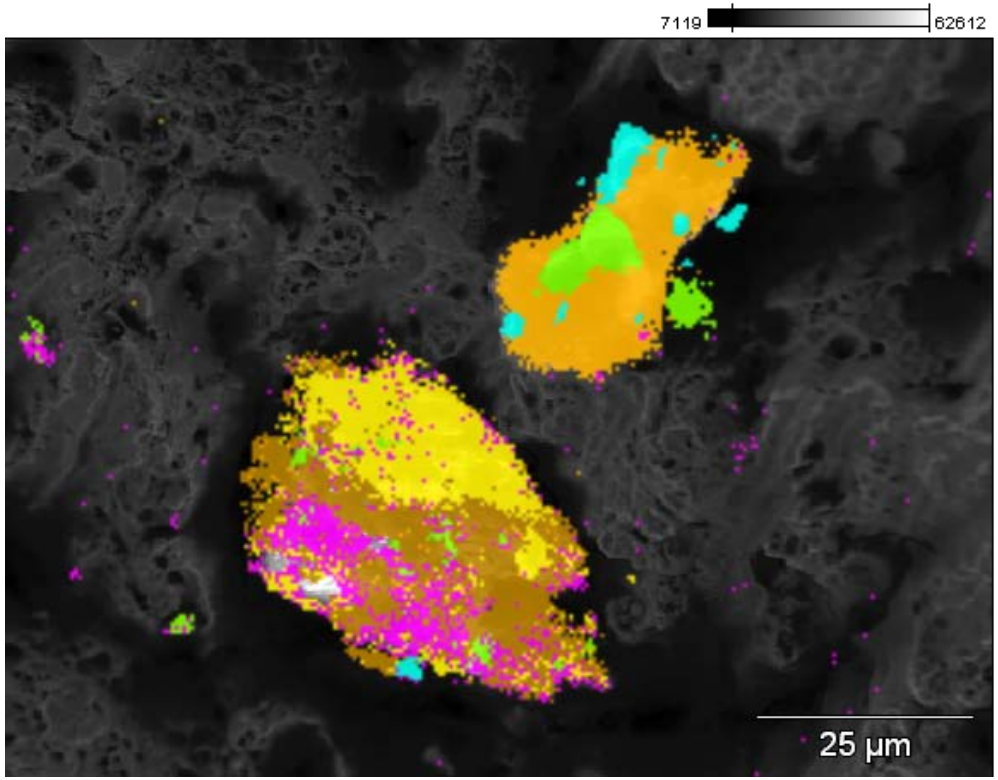




- Phase analysis finds all compounds.
- Point analyses may miss some compounds.

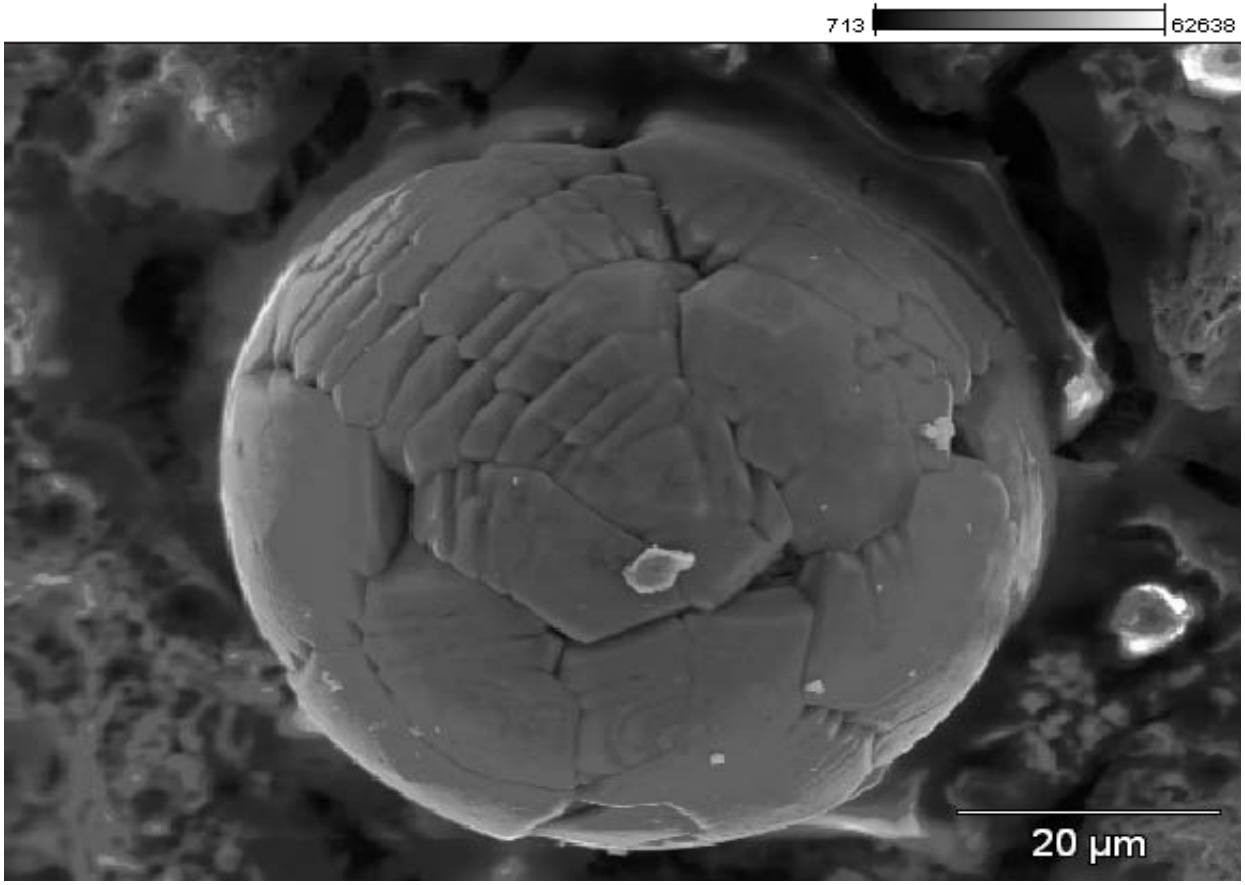
Multiple Particles 3



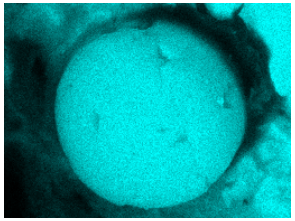


- Phase analysis finds all compounds.
- Point analyses may miss some compounds.

Fe-O Particle Analysis

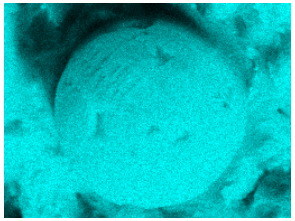


SW All x-rays

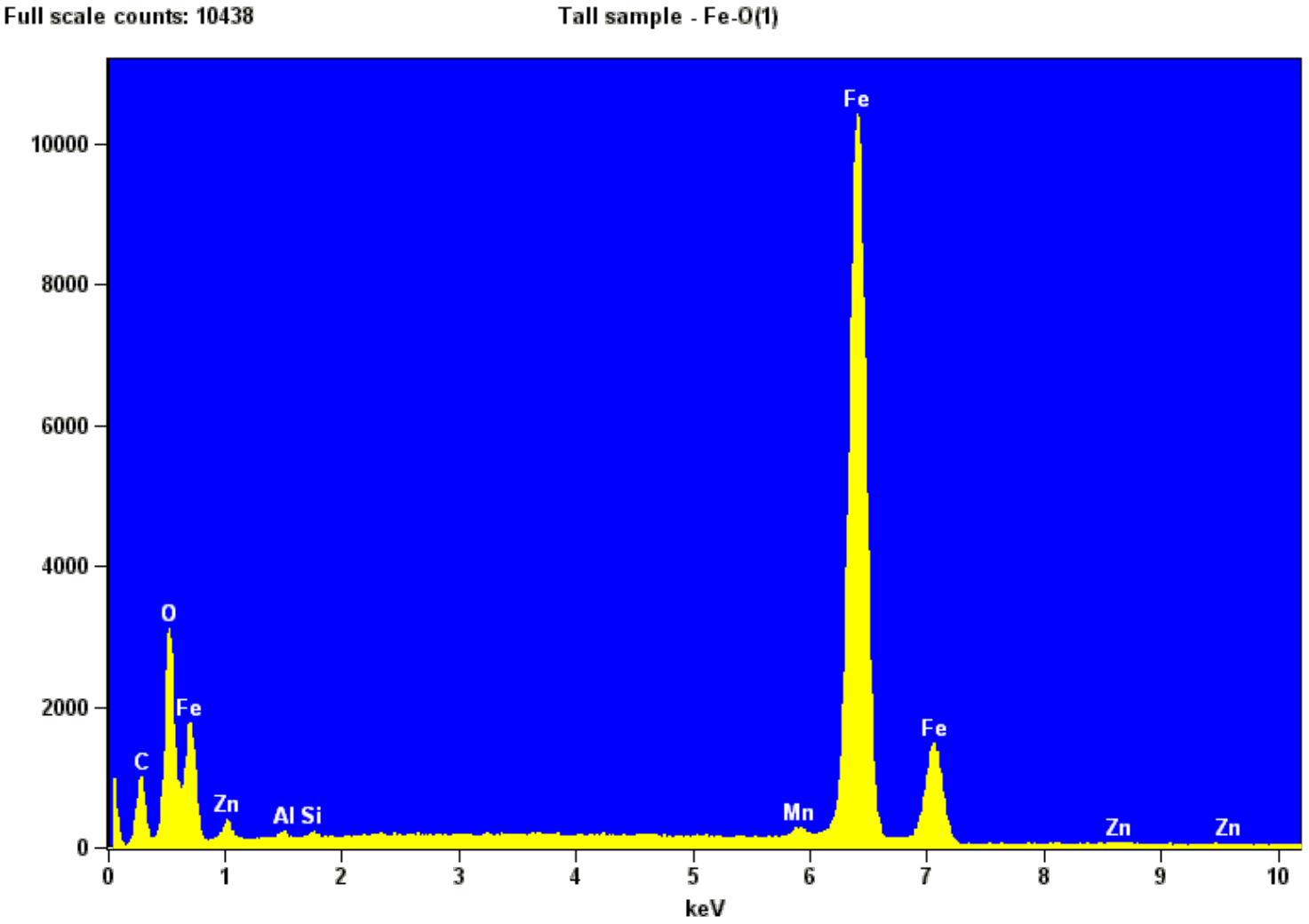


- Surface topology will cause difficulties with shadowing.

SE All x-rays



Fe-O Particle – EDS



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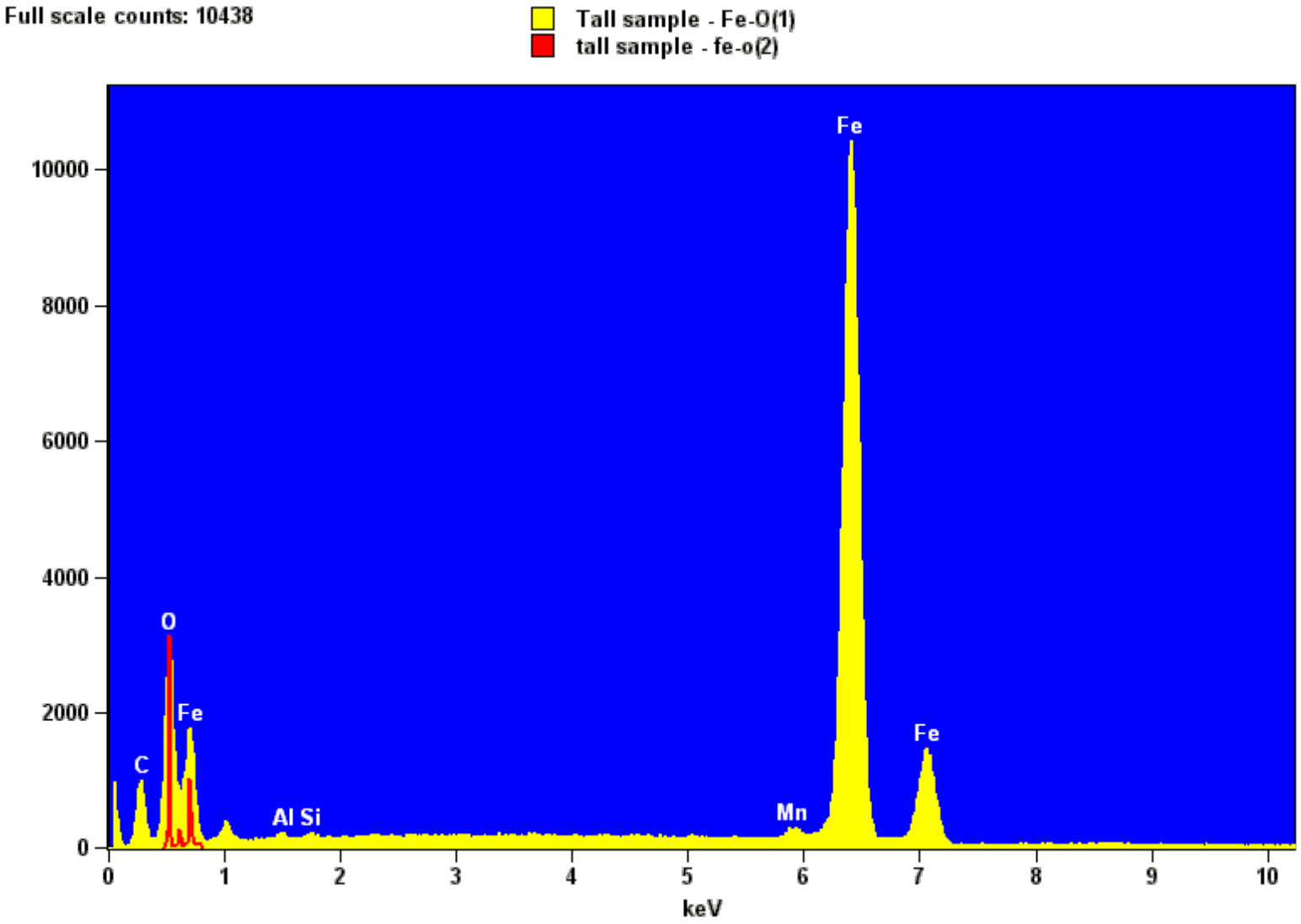
Fe-O Particle – EDS + WDS



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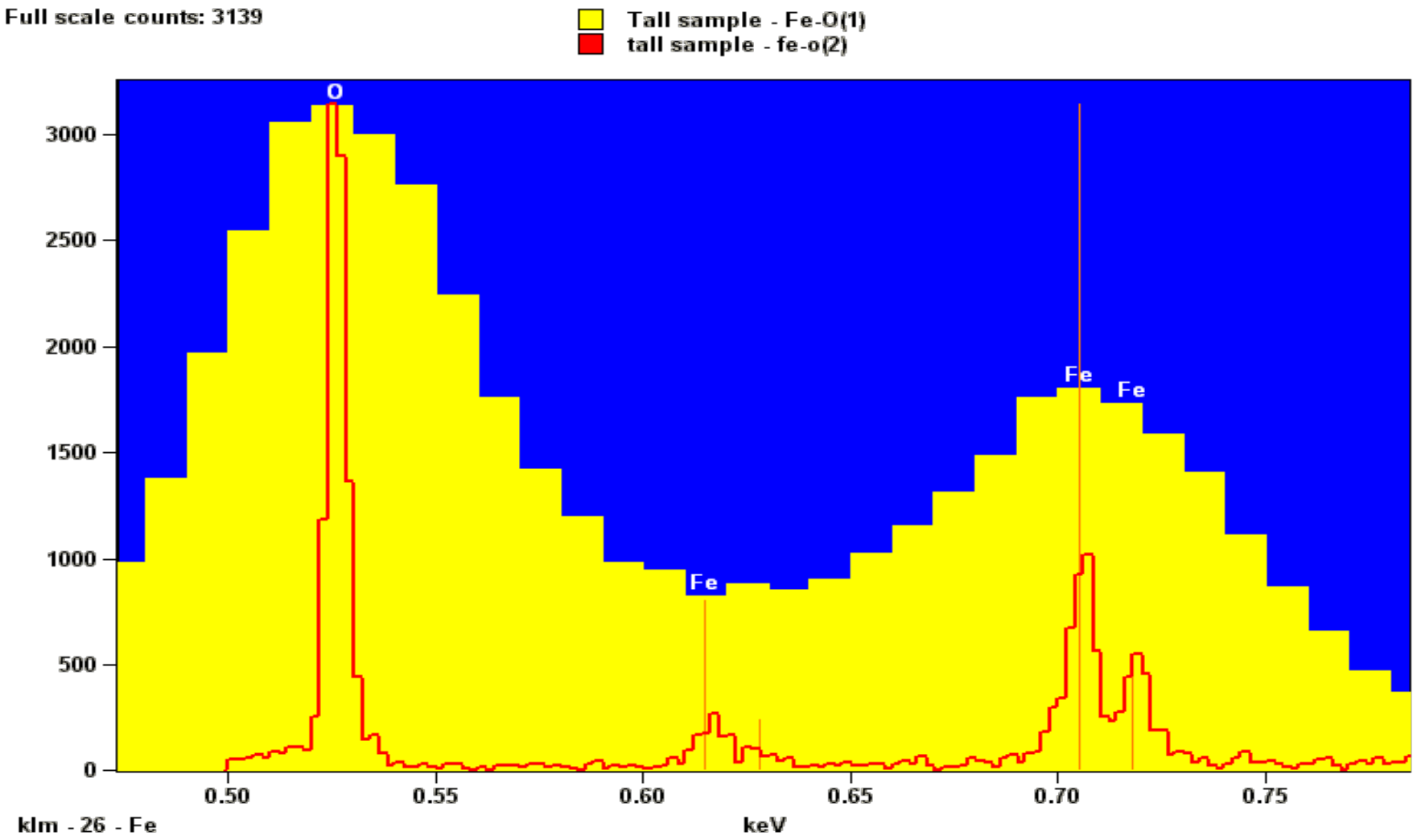
Fe-O Particle – Low-Energy EDS + WDS



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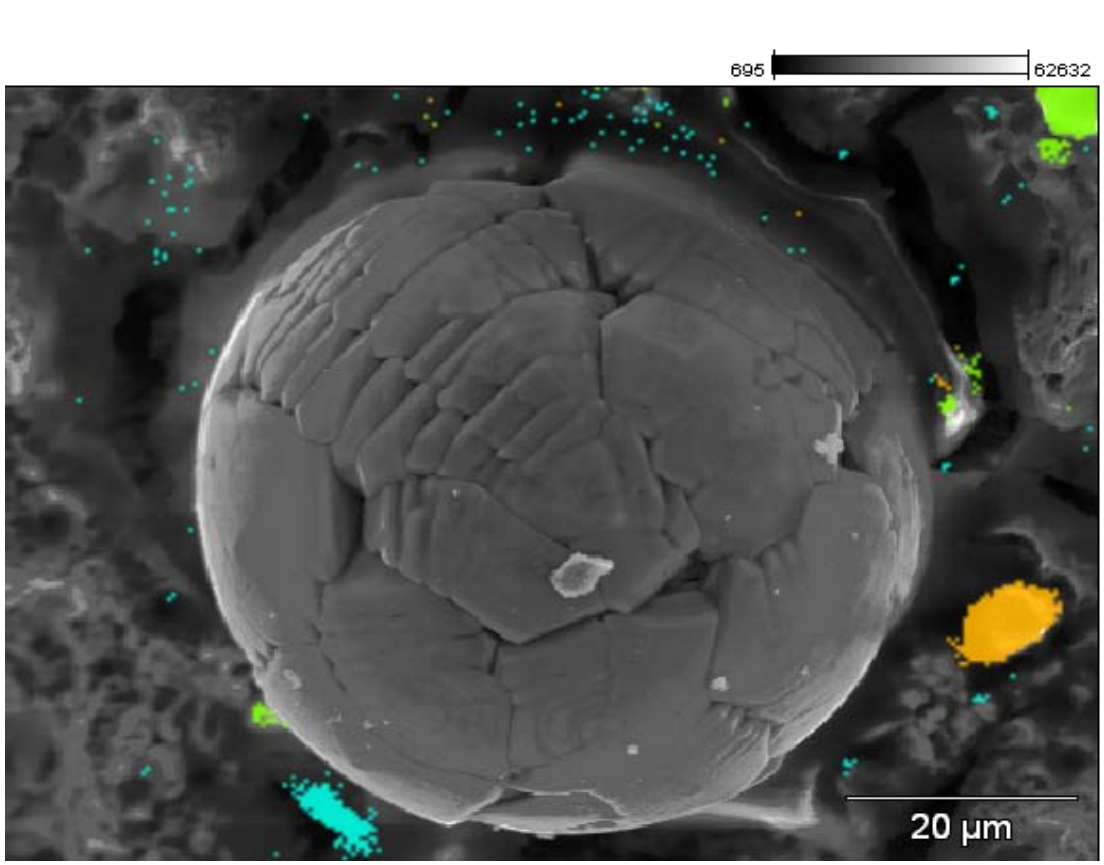
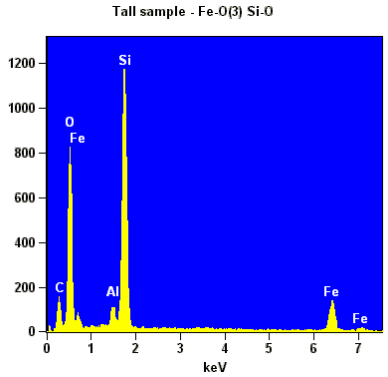
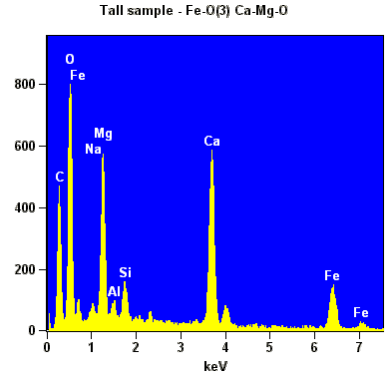
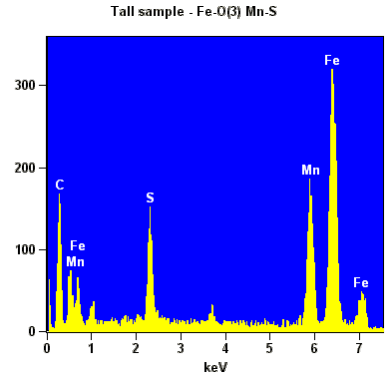
Fe-O Particle – Other Phases









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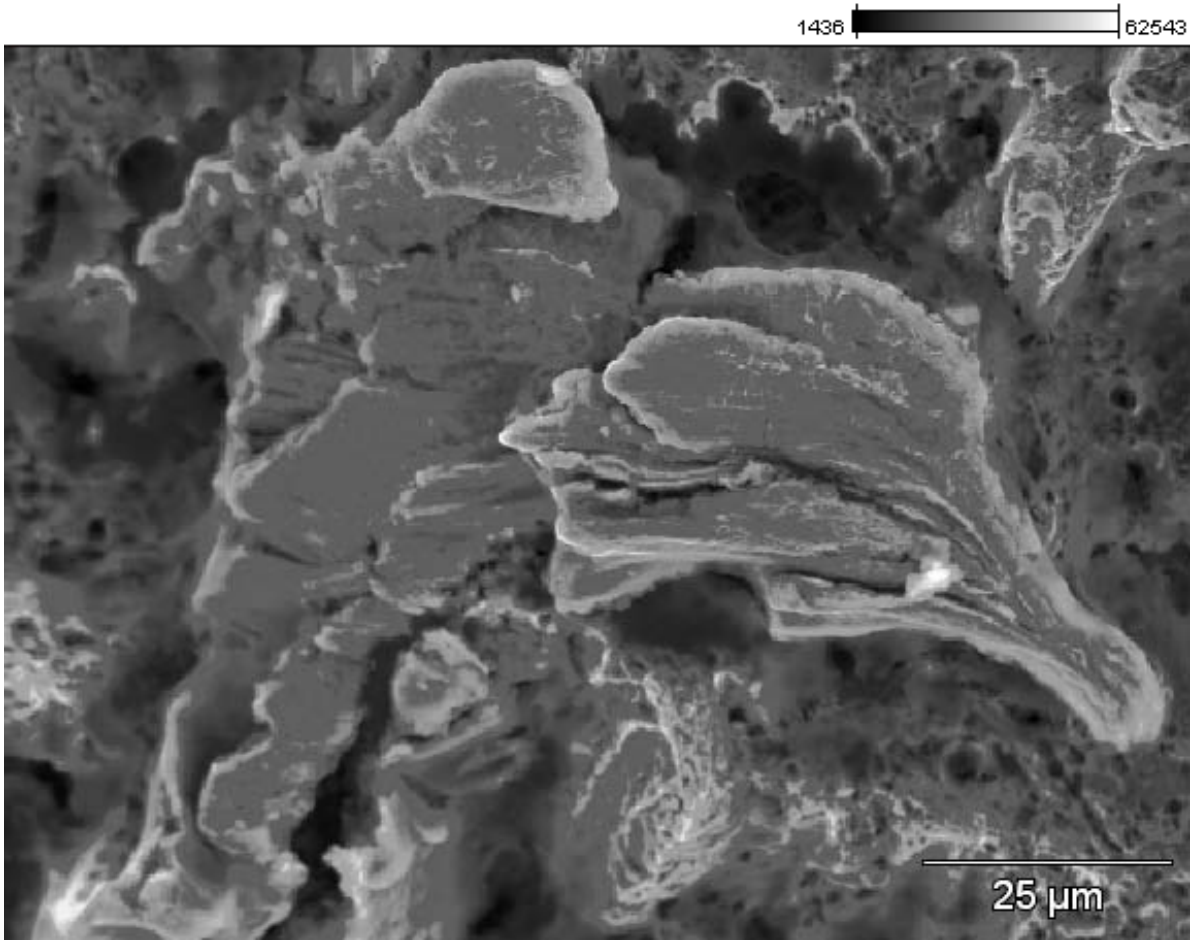
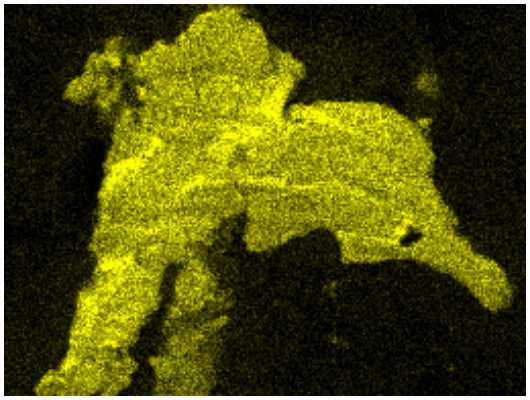
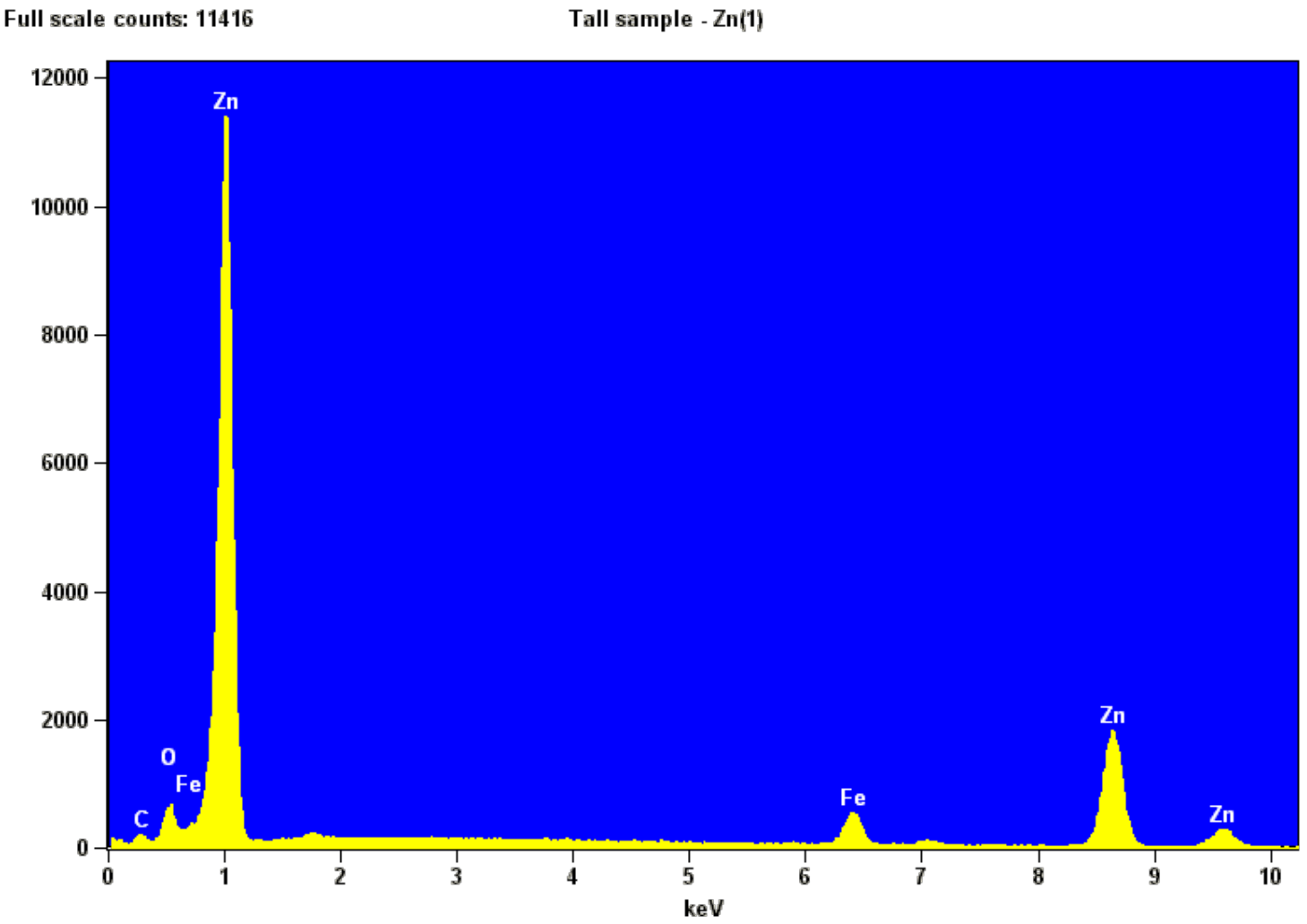
Zn Particle Analysis



Zn-K Map



Zn Particle – EDS



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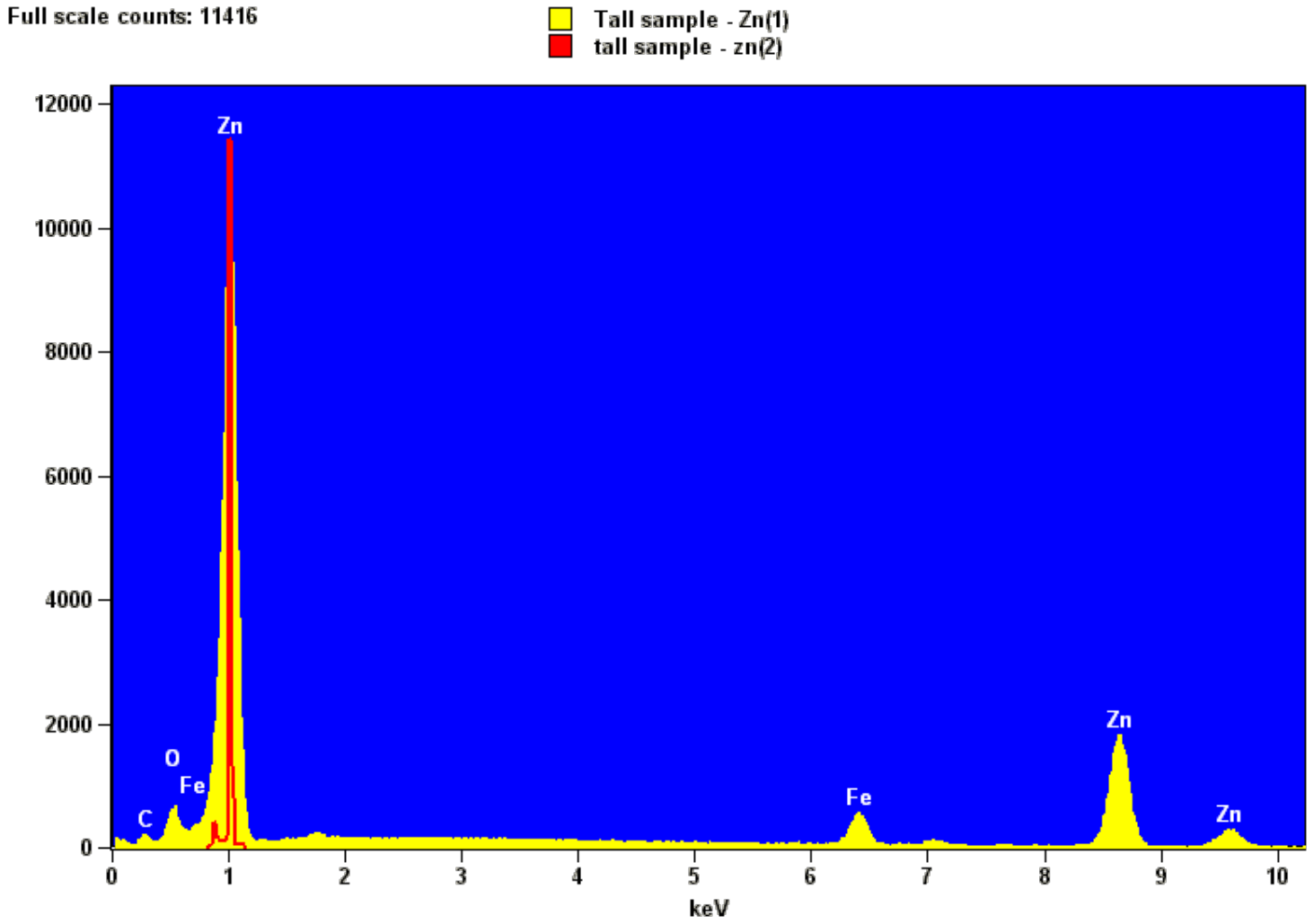
Zn Particle – EDS + WDS



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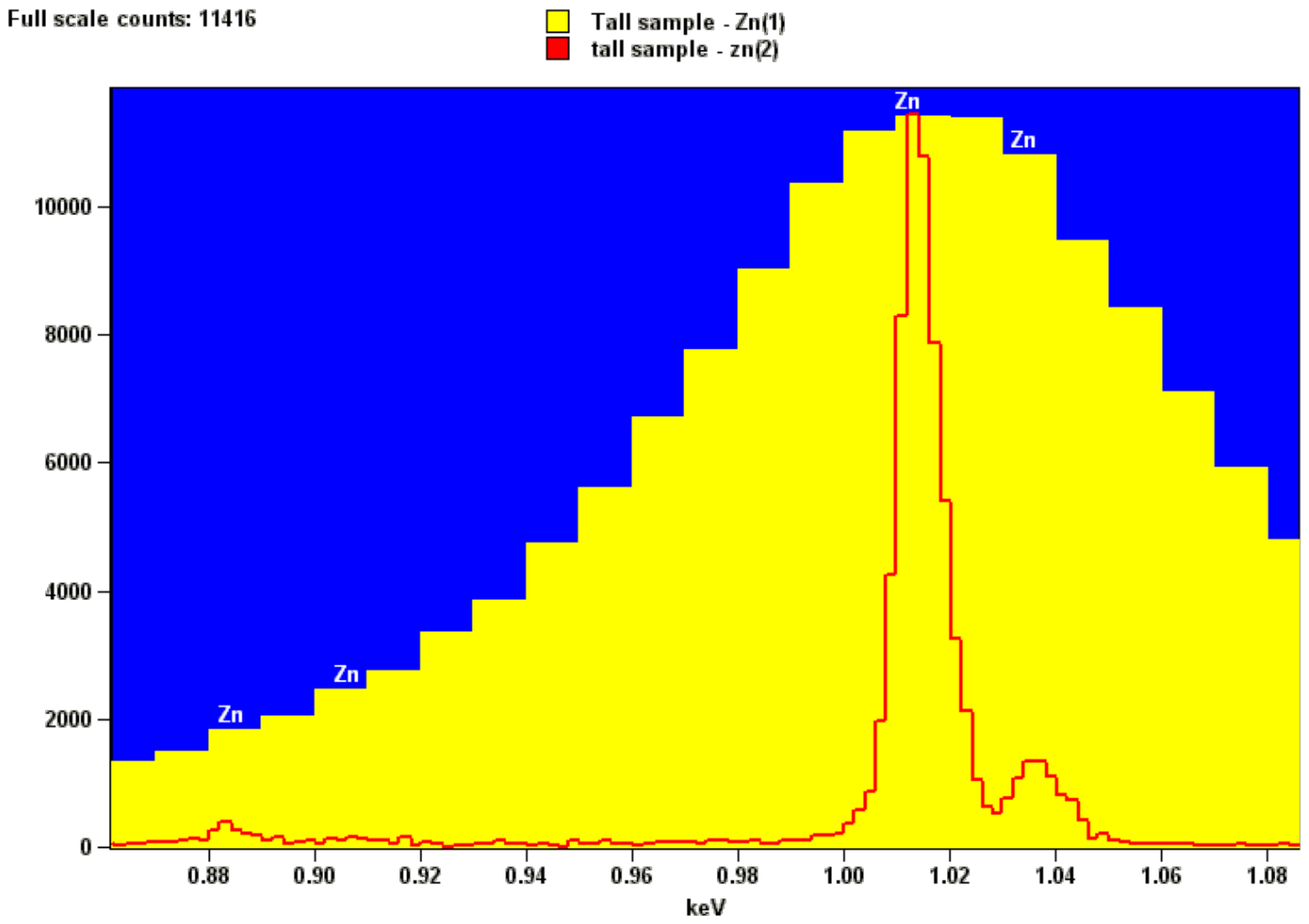
Zn Particle – Low Energy EDS + WDS



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Spring Sample Summary

- Fracture surfaces cause shadowing effects
  - Dual EDS detectors can be useful
- Many particles with a variety of chemistries
  - Most are multi-element compounds
- Many have the same contrast as the background
  - Mapping is more useful and time efficient than point analyses
- Any of these particles could have caused crack initiation

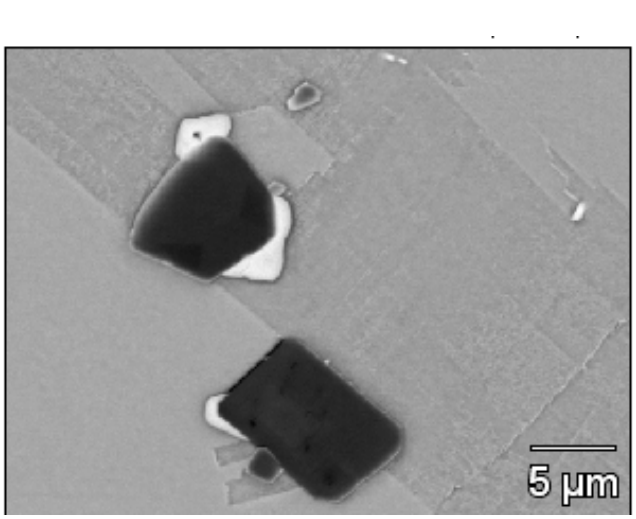
Microanalysis of a Failed Aerospace Component

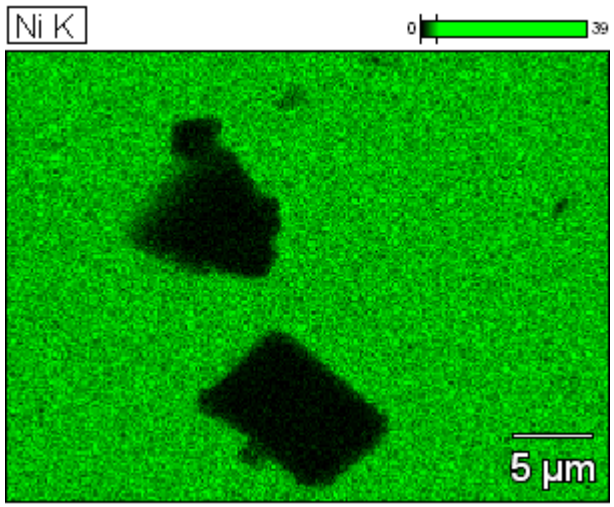
Experimental Conditions

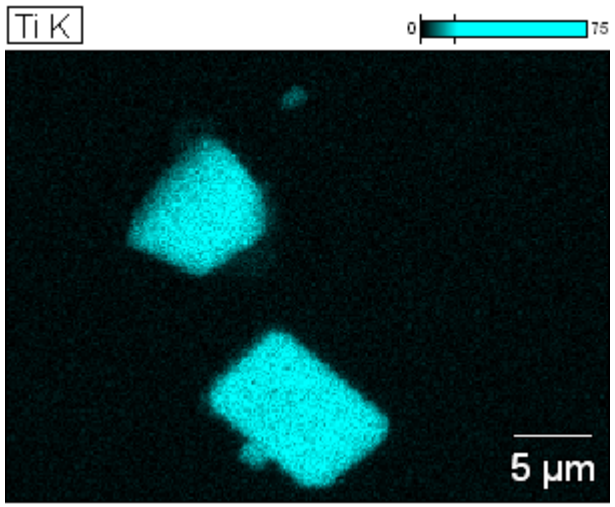
- 20 kV beam voltage
- Unmounted polished sample
- 30 nA beam current

- Acquisition types, termination criteria and times
  - EDS
    - 256 pixels
    - 100 counts per pixel termination
  - EBSD Points
    - 1 second exposure
    - Concurrent EDS spectrum
  - EBSD Mapping
    - 128 pixels
    - 40 patterns per second, ~ 5 minute acquisition
    - Concurrent EDS SI

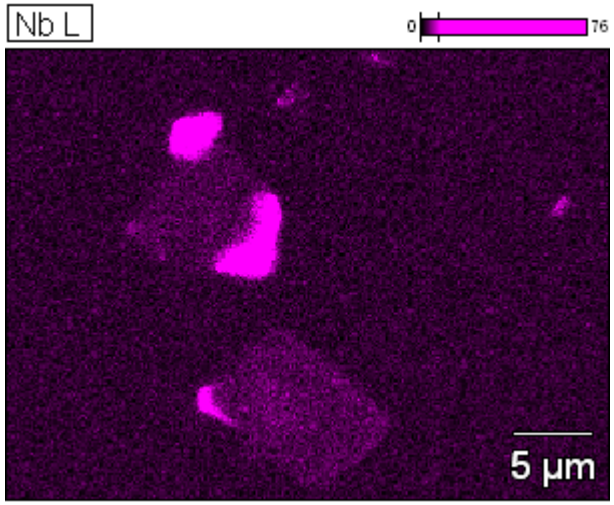
Elemental Distributions – High Magnification

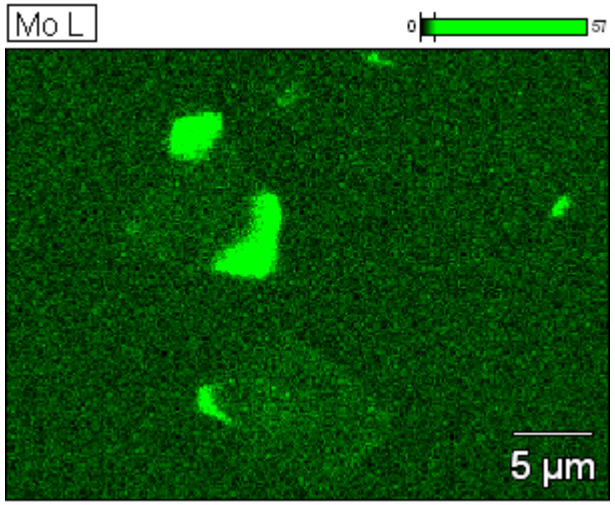




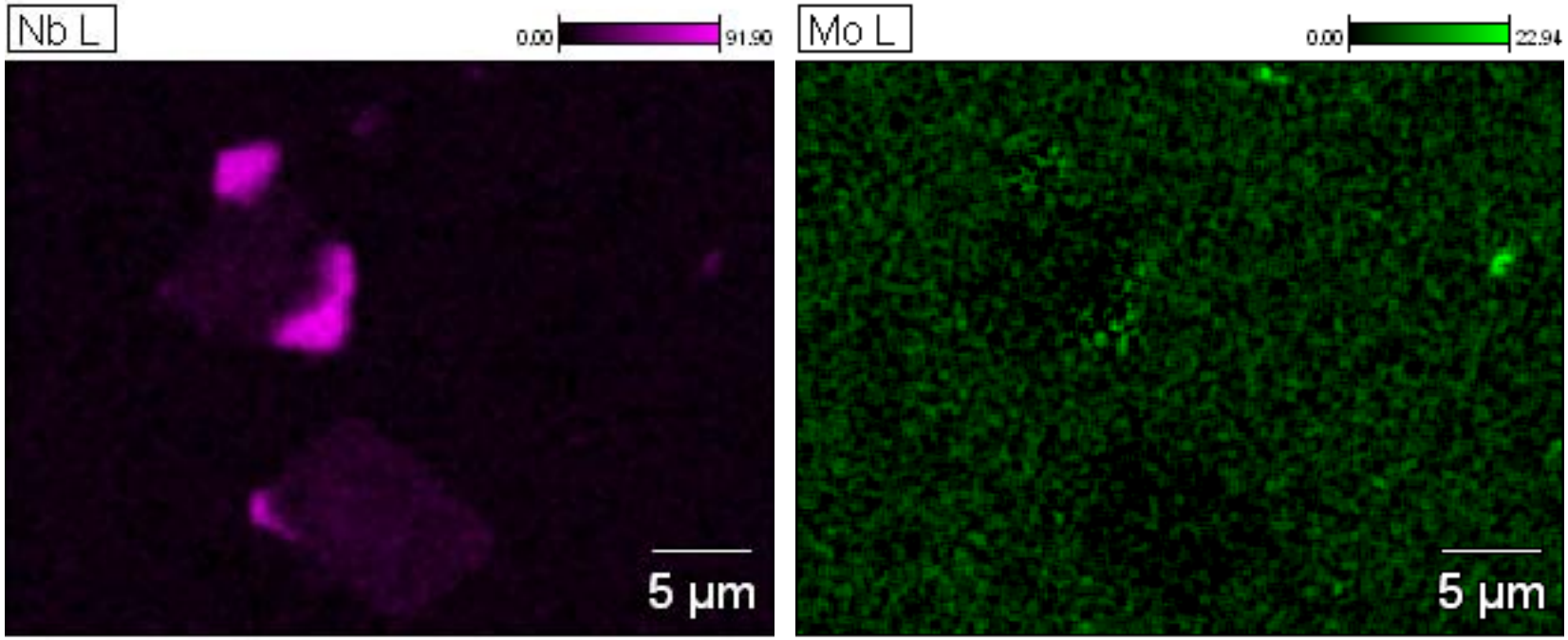


- Bright and dark particles
- Bright particles seem to be Nb and Mo





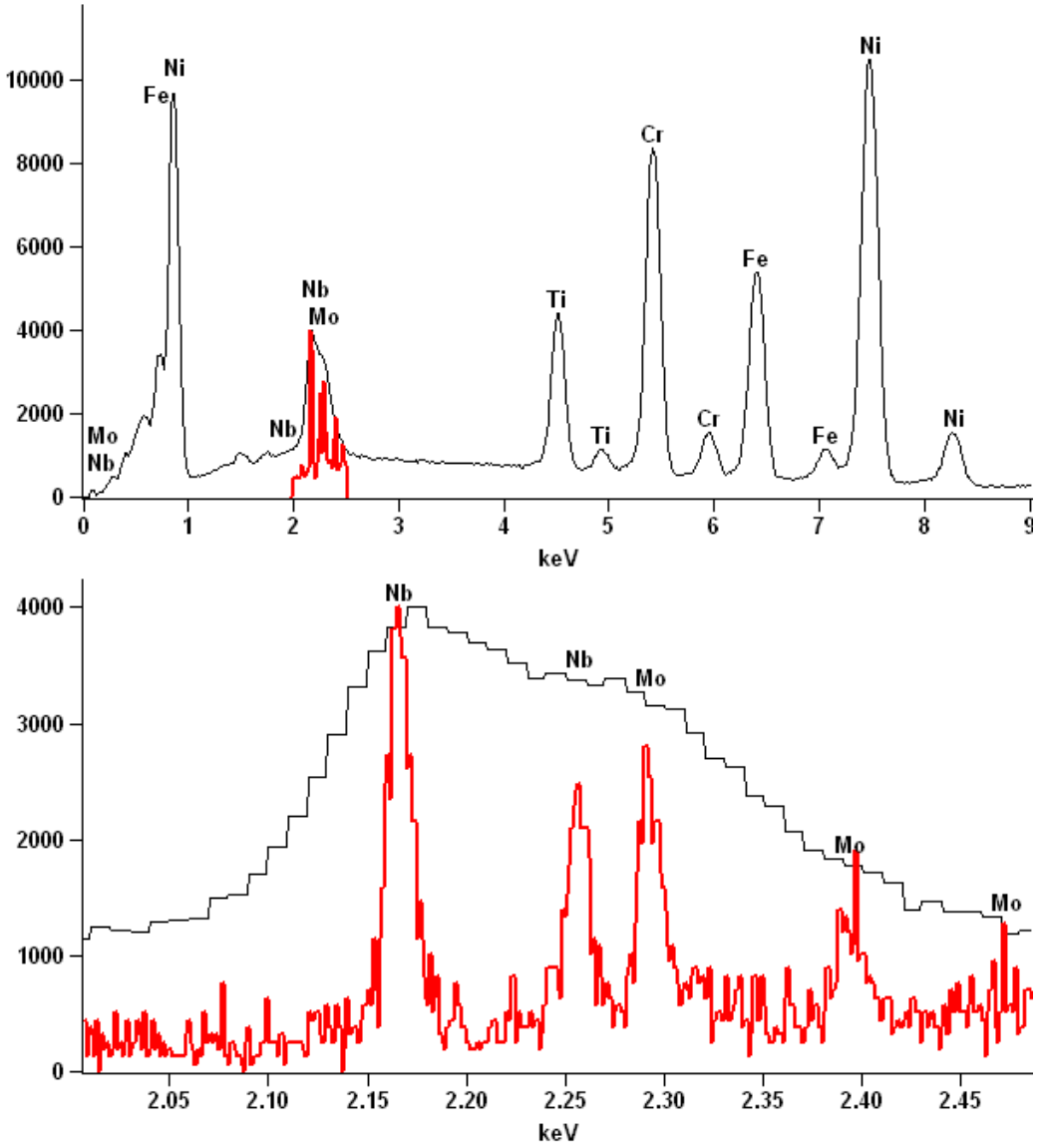
True Elemental Distributions – Quantitative Mapping



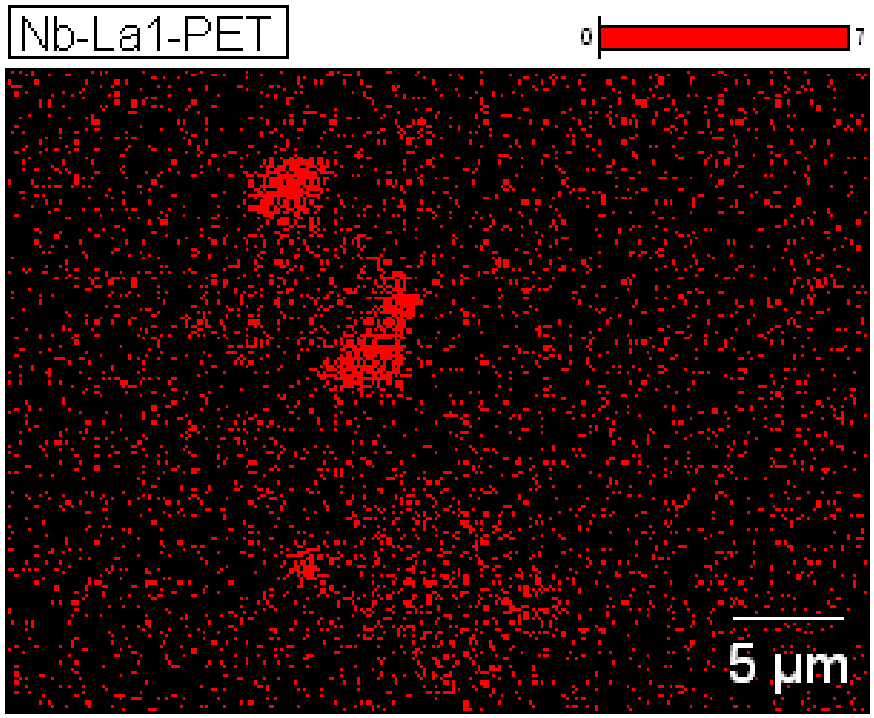
- Bright particles are Nb rich and contain no Mo !
  - Quantitative maps are REQUIRED for maps when elemental peaks overlap.

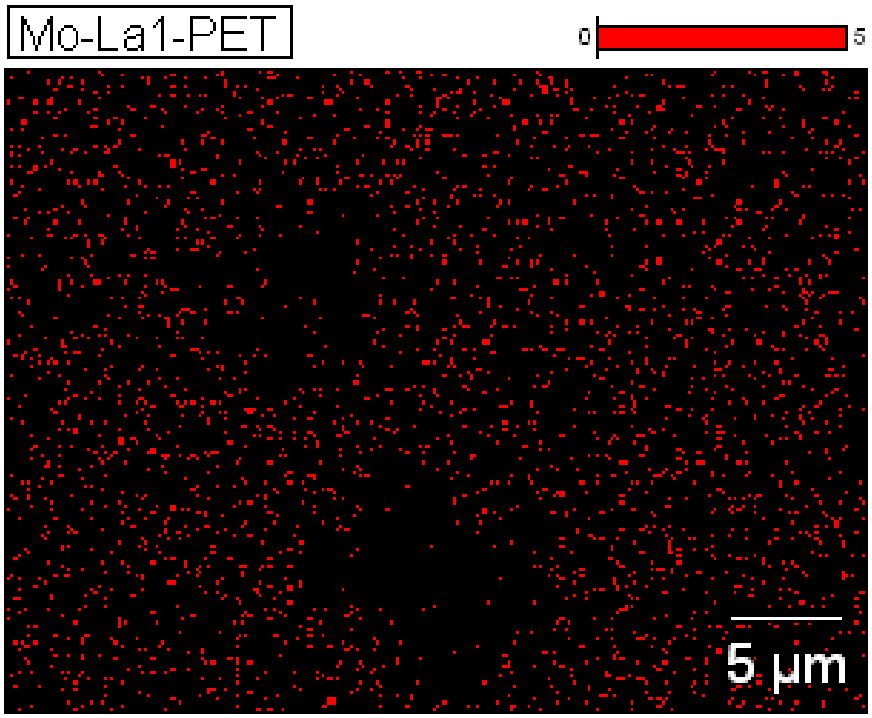
WDS Peak Deconvolution

- WDS spectral resolution is significantly better than EDS spectral resolution.
- Elemental confirmation of Nb-L and Mo-L spectral peak overlap.



WDS Mapping

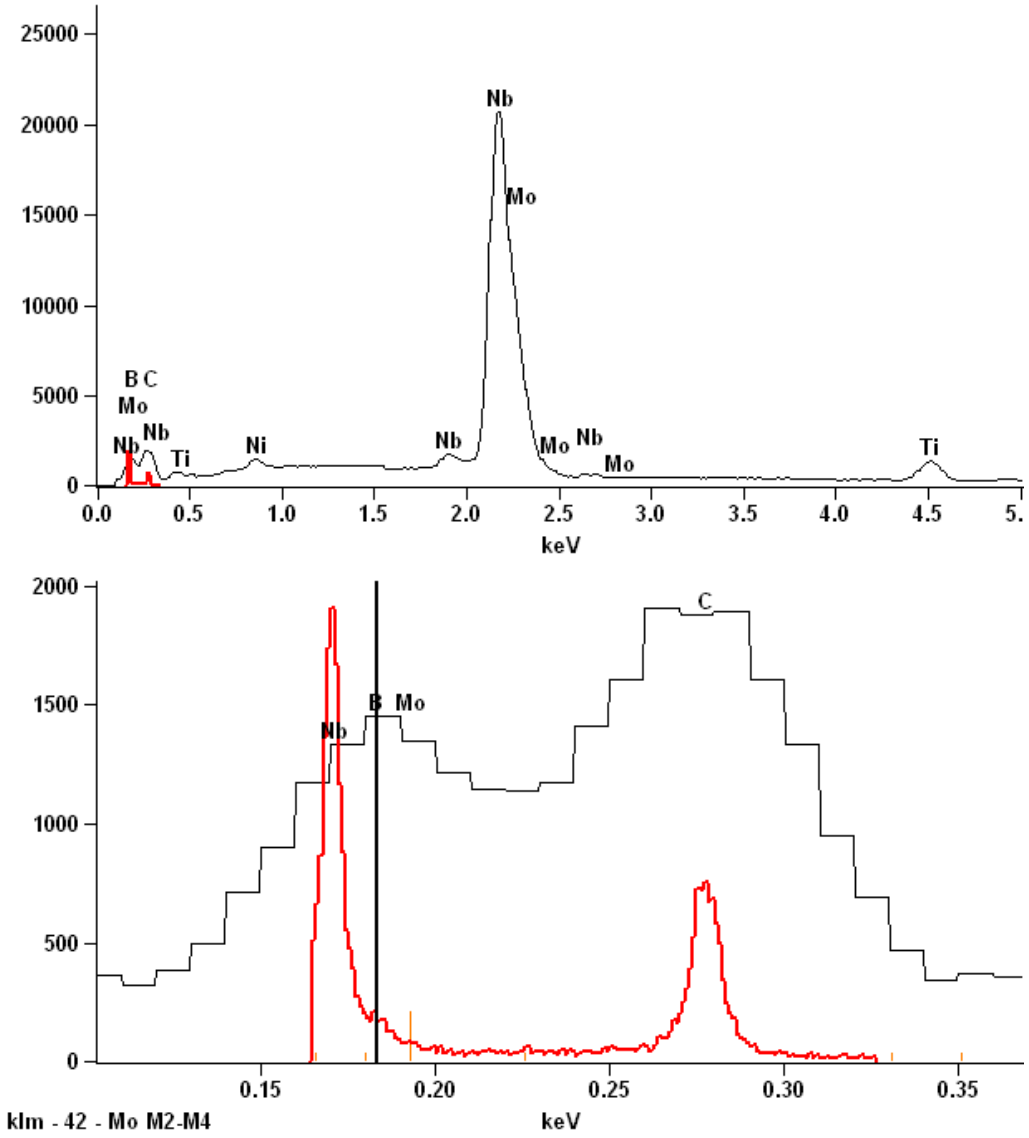




- WDS Spectral resolution provides true elemental distribution of peak-overlapped elements.

Low Energy Peak Verification of Bright Particles

- EDS peak identification implies presence of B and C.
- WDS spectral resolution confirms:
  - EDS peak near 180 eV is NOT B-K or Mo-M but is Nb-M.
  - EDS peak near 280 eV is C-K.
- The bright particles are Nb-C.



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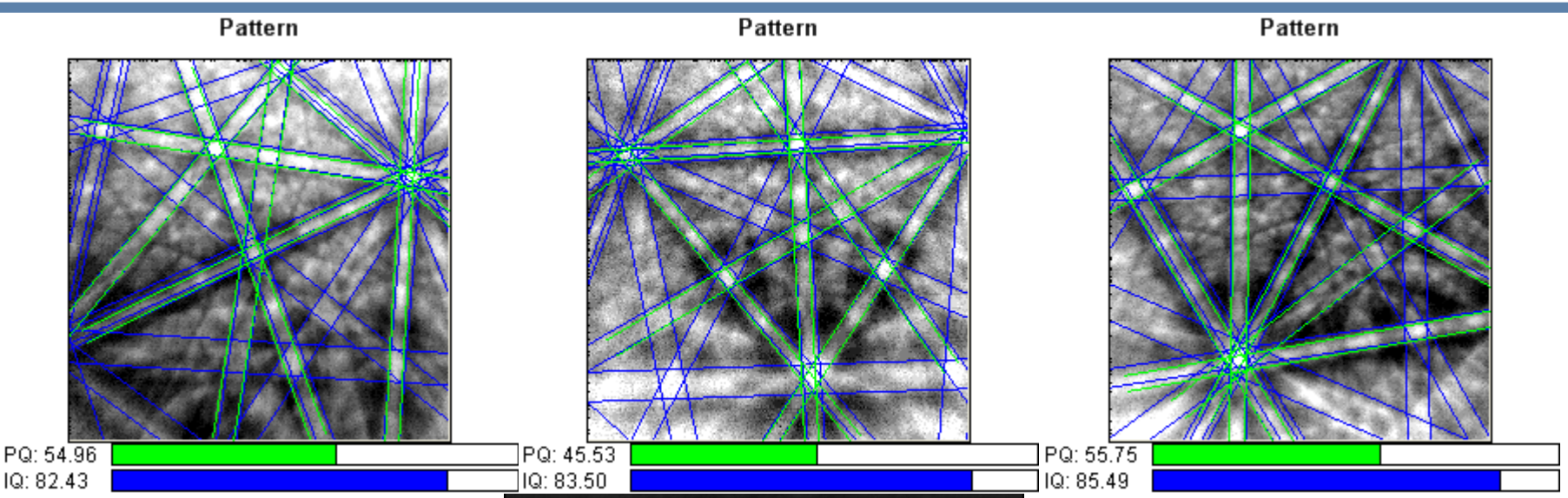
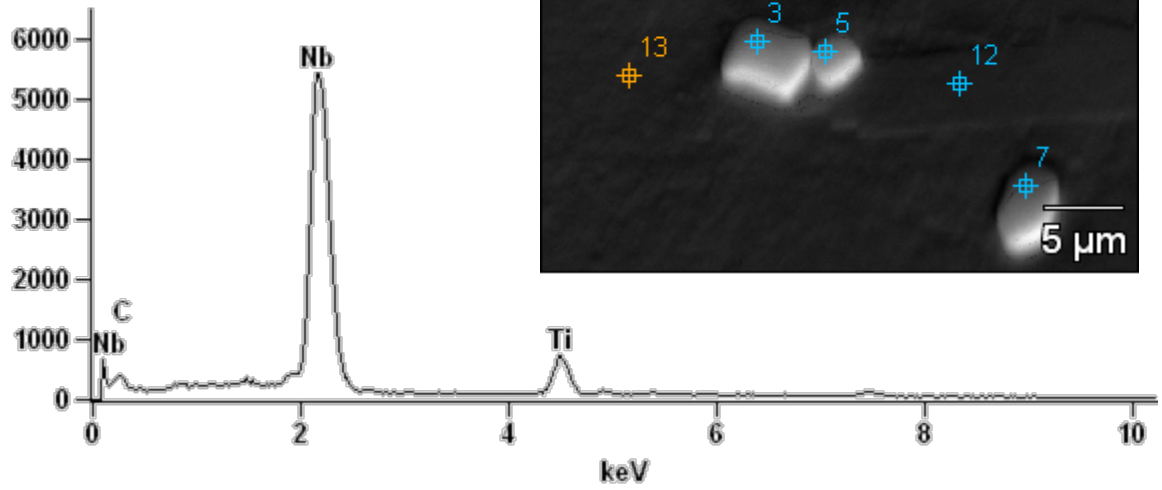
EBSD Particle Analyses





- Using EDS and WDS elemental results, EBSD identifies bright particles as NbC.

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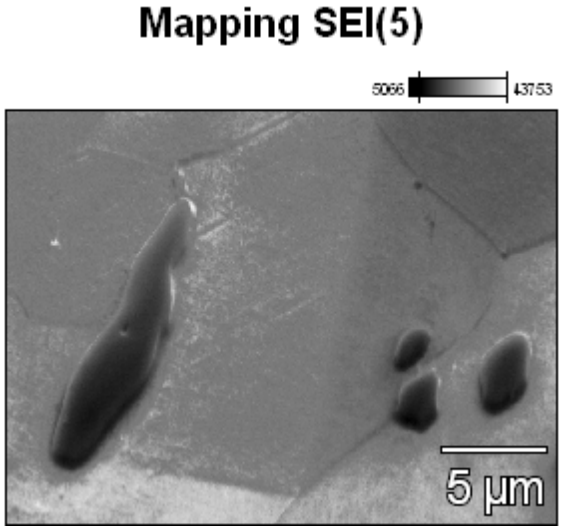
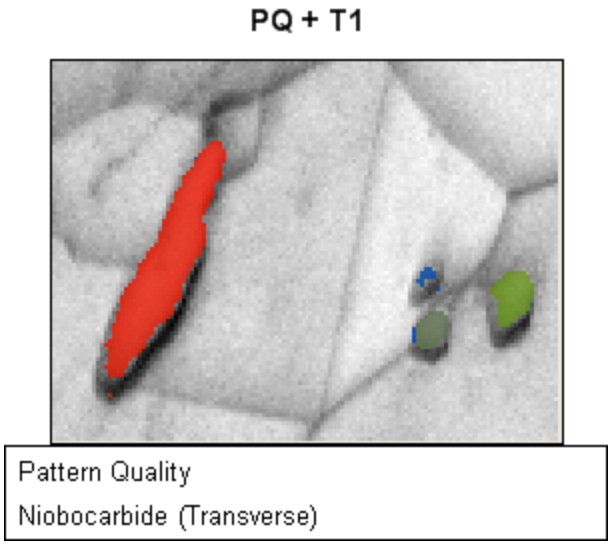
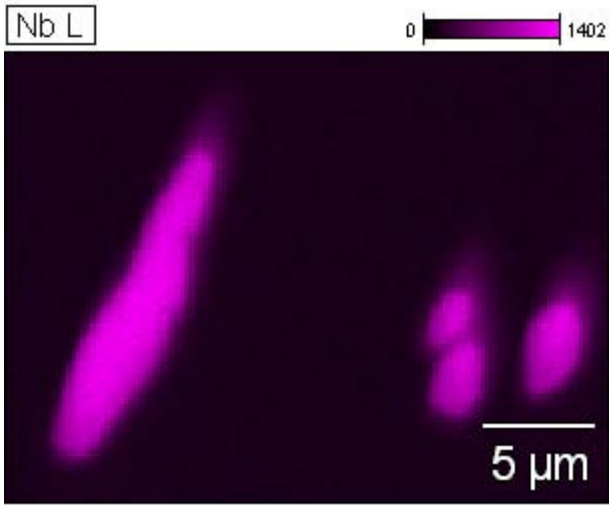
EBSD + SI Mapping







- Matrix is indexed as Ni (FCC).
- Particles are indexed as NbC (FCC).
- Orientations are different for each particle.
- EDS and EBSD results are self-consistent.

Aerospace Sample Summary

- Brightly imaging particles (in BSE)
- Particles contain Nb and C
- Confirmed to not contain Mo and B
- Crystallographically confirmed as NbC

- These may be a reason for unintended properties.

Summary

- Dual-detectors help smooth the x-ray intensity from rough sample surfaces
- EDS Spectral Imaging analyses
  - Acquisition in less than 3 minutes
  - Elemental distributions are found that are not visually apparent in electron image
  - Phase analyses provide a wealth of compound information, both spatial and chemical
- WDS analyses
  - Can be challenging on fracture surfaces
  - Provide elemental confirmation in spectra
  - Provide true elemental distribution maps
- EBSD analyses
  - Identify crystal structure
  - Map distribution of crystal phases

Conclusions

- NORAN System 7 microanalysis system provides all of the necessary tools in electron-beam instruments to fully understand the distribution of alloys, minerals, and compounds in samples for failure analysis.

Questions

- Thank you for attending today's webinar.

- Ask any questions using the chat function.