

Scanning Electron Microscopy & Energy Dispersive Spectroscopy and X-ray Diffraction

Brad Kobe

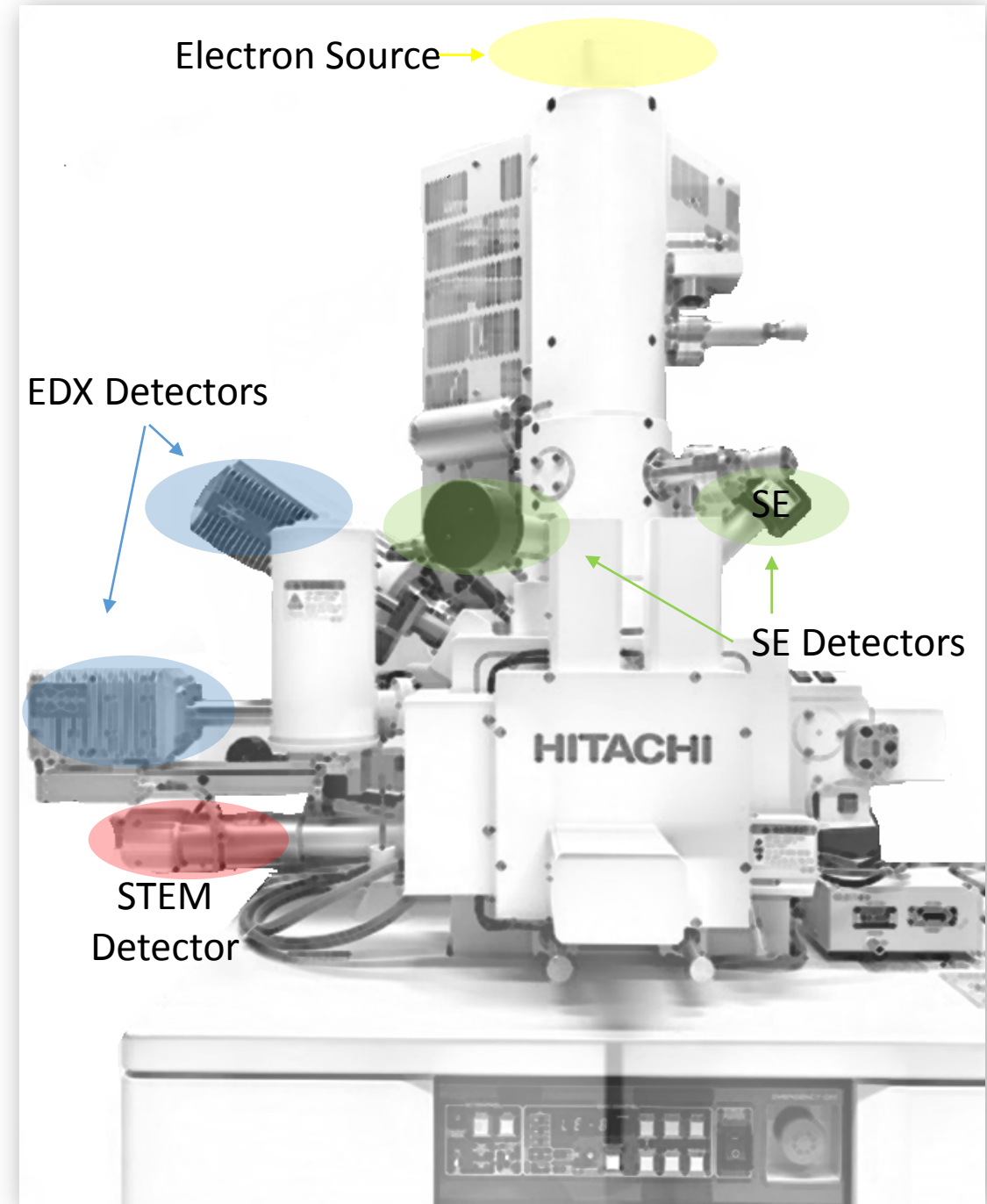
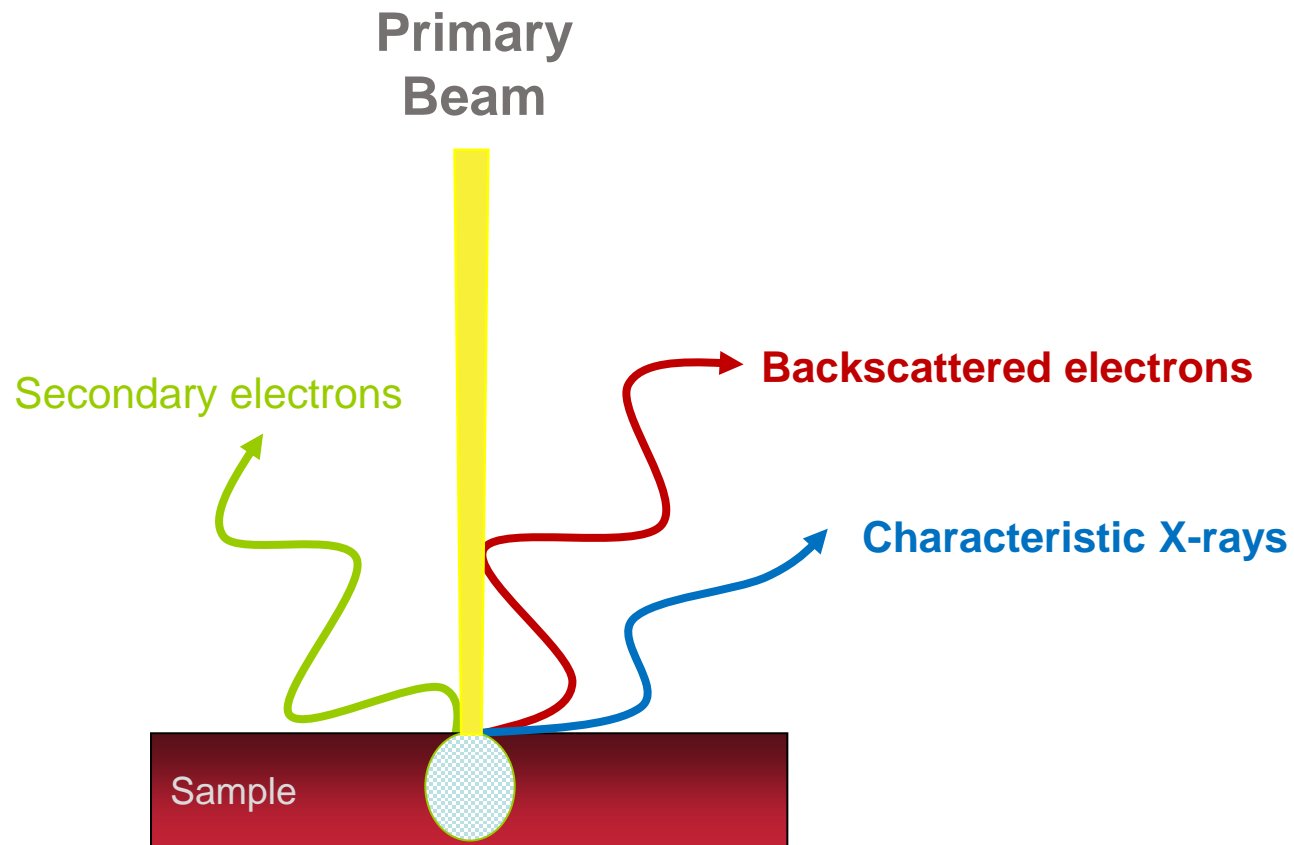
Western University

Surface Science Western



ISO 9001:2008 Registered

How does an SEM work?



System Capabilities

One of the first analytical techniques used to characterize samples

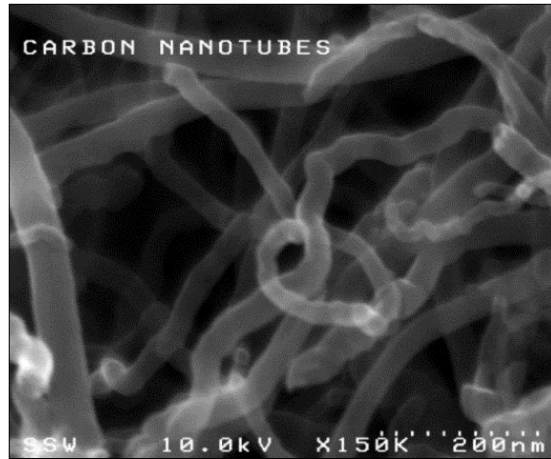
Scanning Electron Microscope

- Magnification range:
5 X to over 1,000,000 X
- Resolution less than 1 nm
- High resolution images
- Atomic number contrast

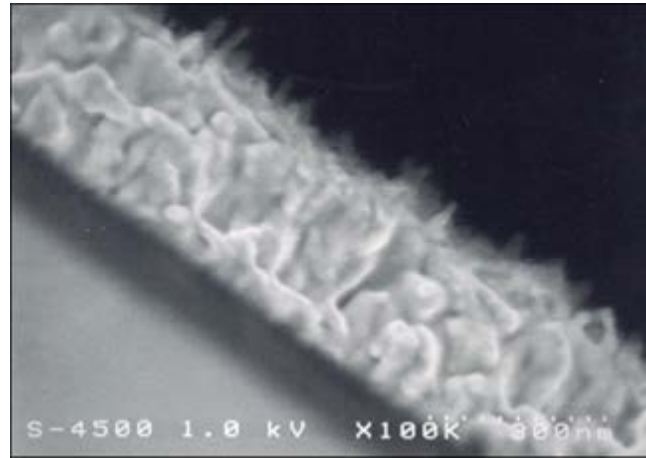
Energy Dispersive Spectrometry

- Beryllium to Uranium
- Detection limit ~ 0.1 weight %
- Analysis depth typically several microns
- Semi-quantitative
- Spot analysis, line scans and mapping

Fast and Versatile Technique



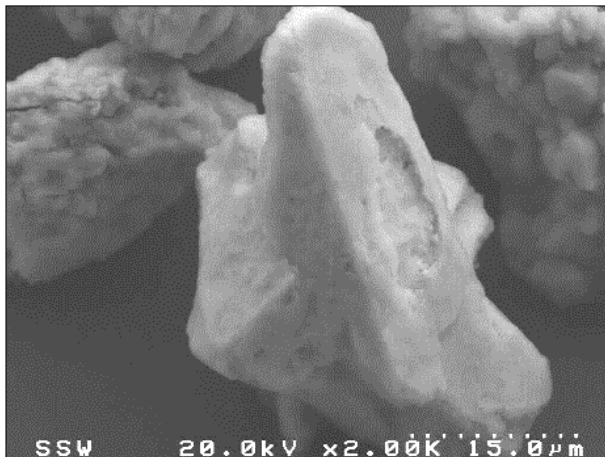
Nano-materials



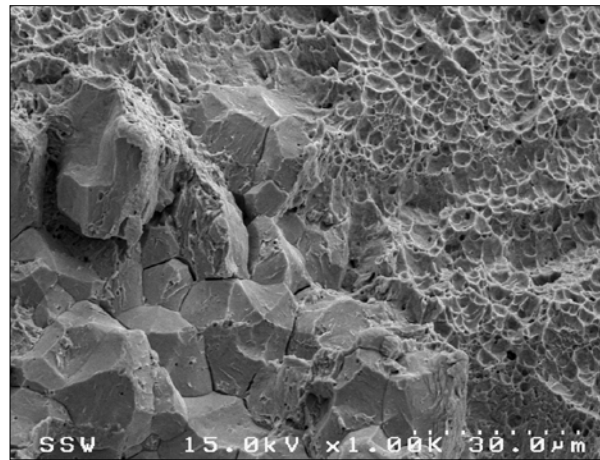
Coatings



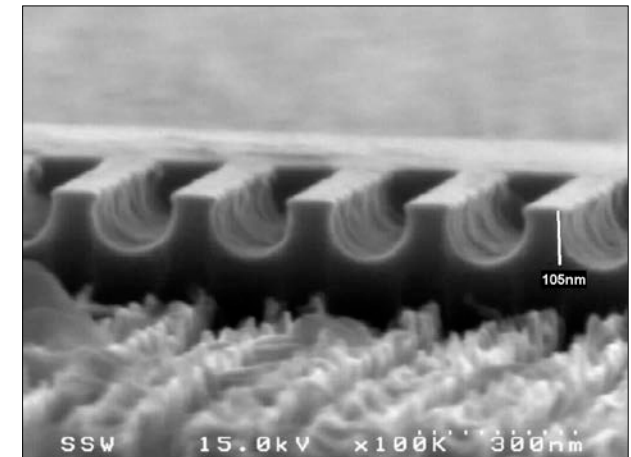
Biological / Medical



Mineralogy

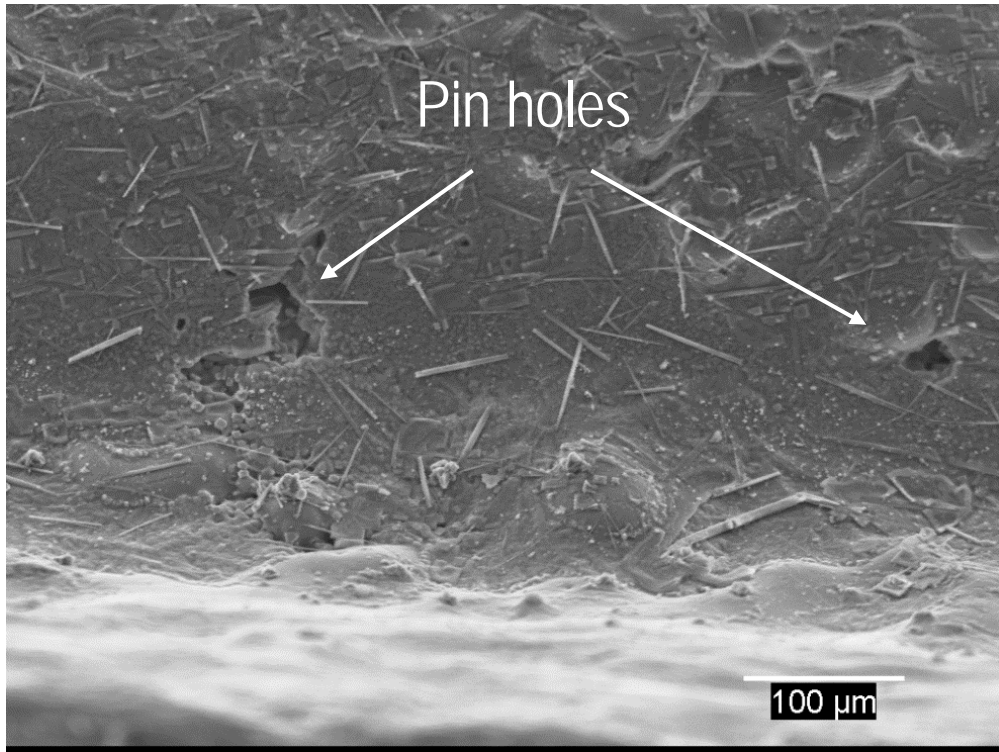


Failure Analysis

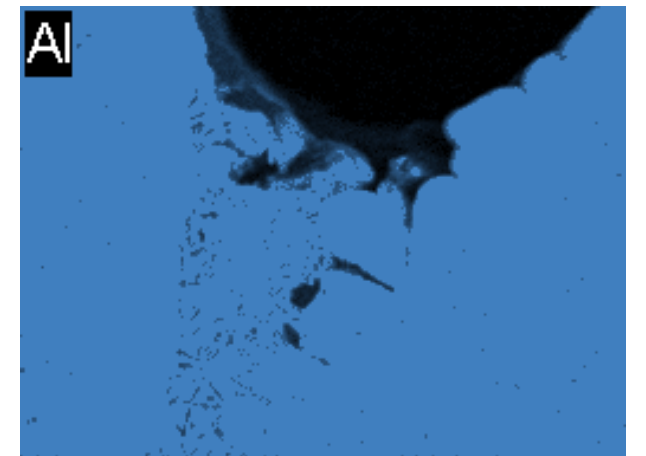
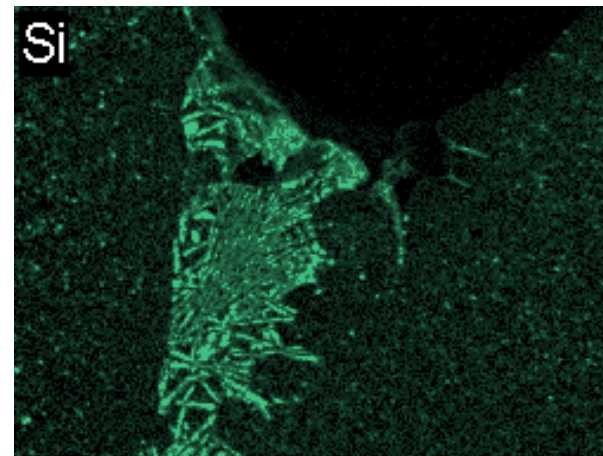
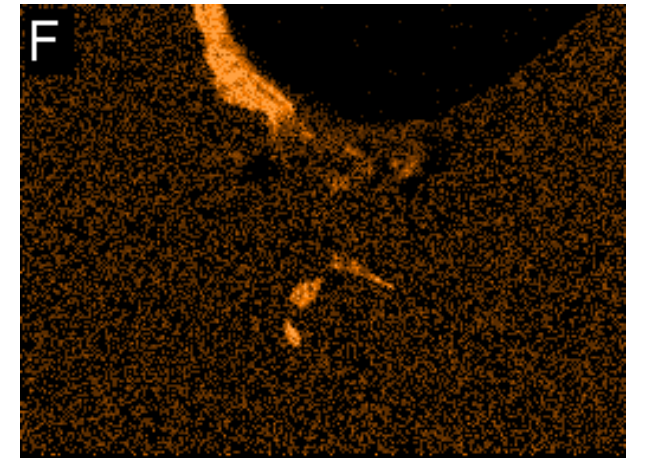
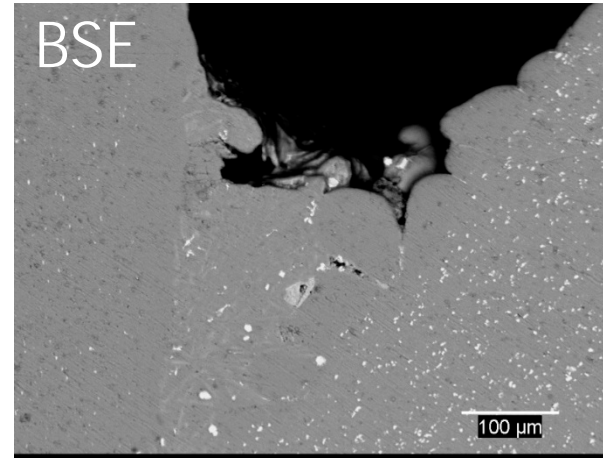


Semiconductors

Leaking Automotive Radiator

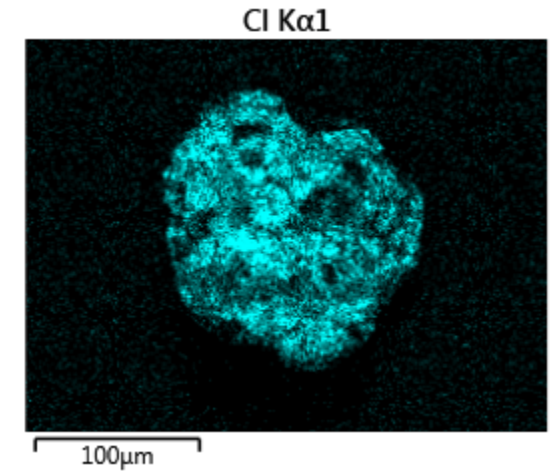
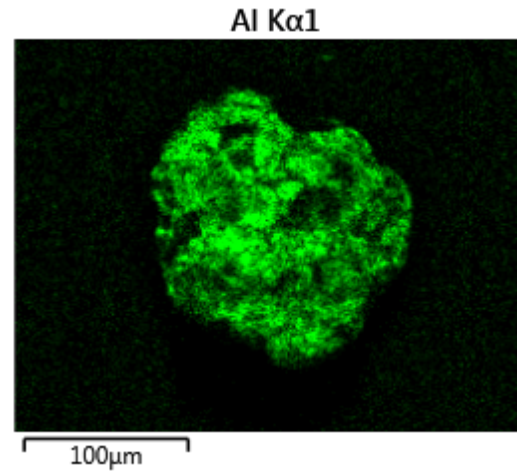
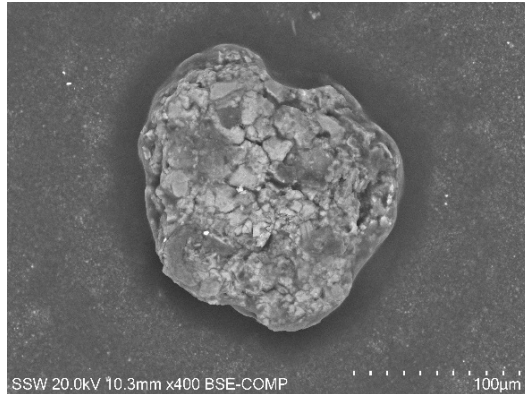
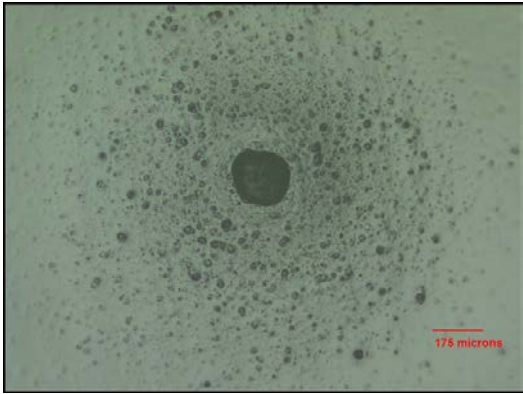


- Where are the leaks occurring?
- Result of manufacturing defect, cracking or corrosion issue?

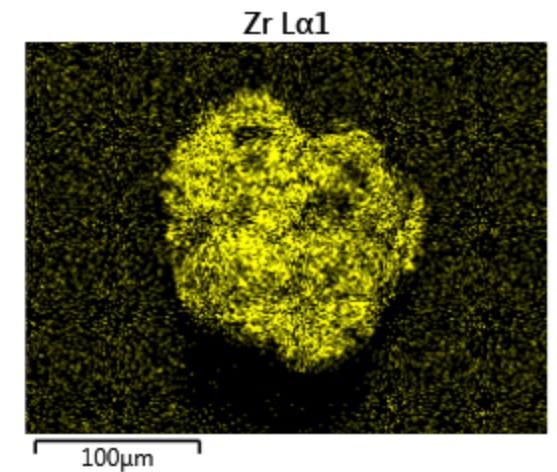
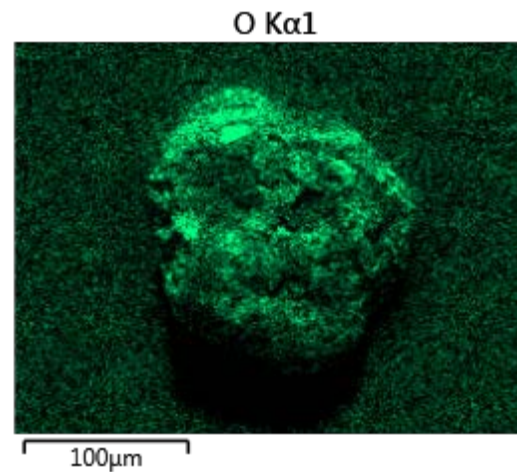


BSE image and elemental X-ray intensity maps of defect in cross-section

Automotive Paint Defects: Antiperspirant



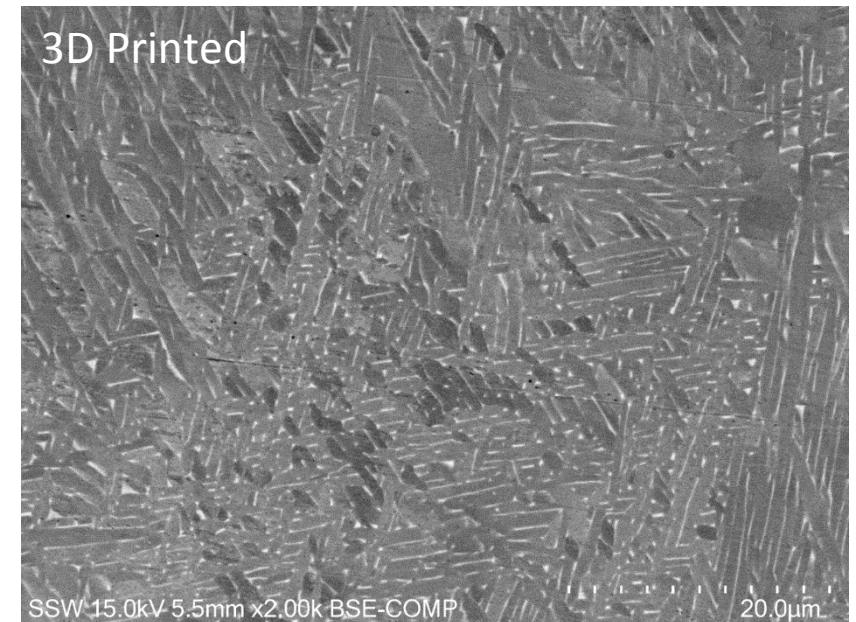
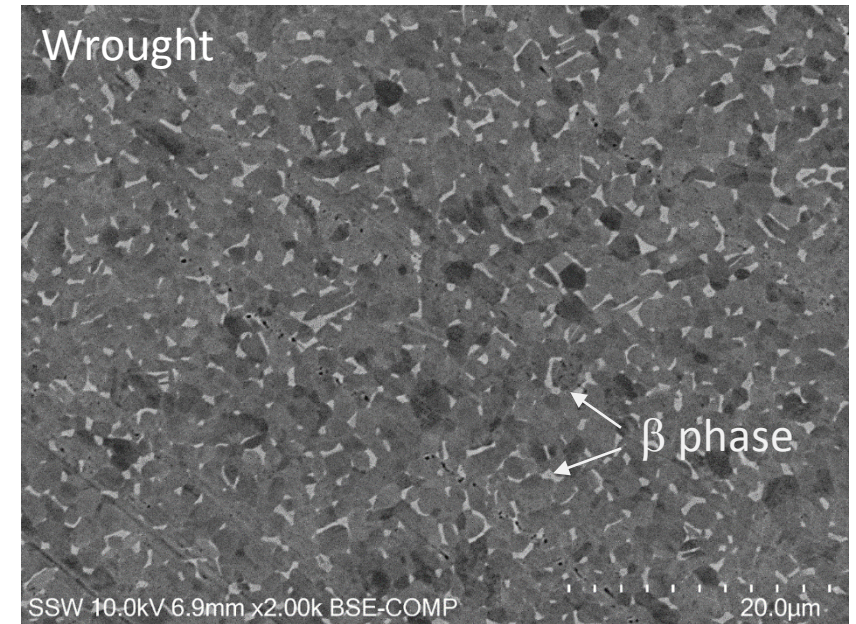
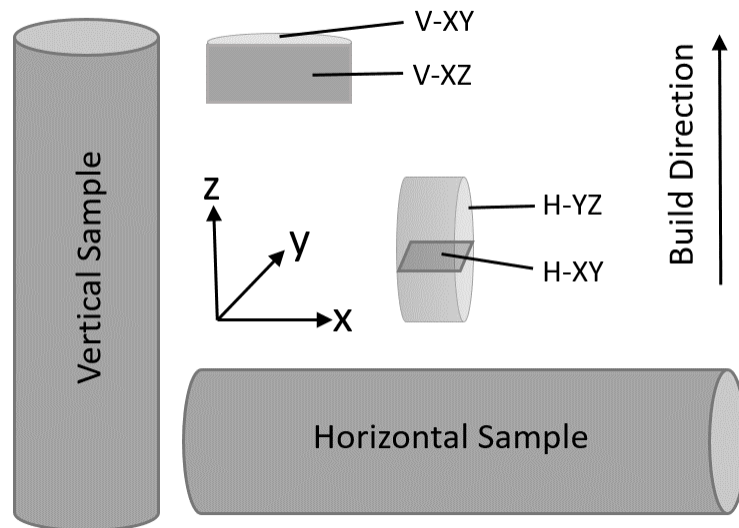
- Numerous sources responsible for paint defects:
 - Silicone, grease, oil, fibres and dirt
- SEM/EDX can quickly identify contaminants



Aluminium zirconium tetrachlorohydrate

3D Printed Titanium alloys

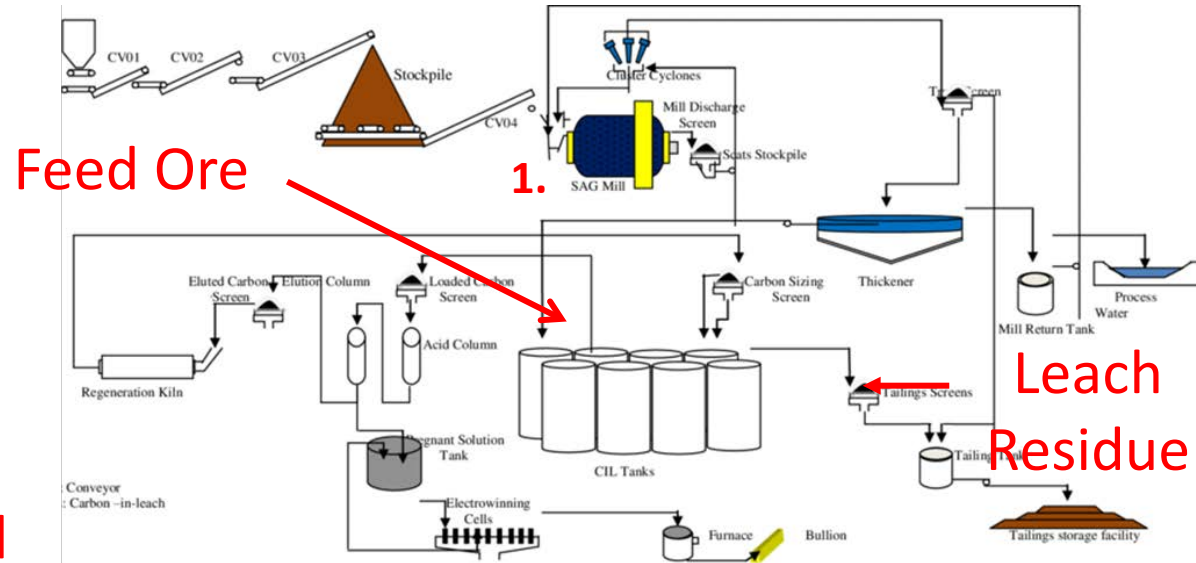
- Comparing the microstructure of wrought and 3D printed parts
- Microstructure influences the mechanical properties



BSE images showing difference in the microstructure between wrought and 3D printed titanium alloys

Gold Recovery

- Gold Recovery Process:
 - liberate the gold grains by grinding
 - extract the gold in a series of leach tanks
 - simultaneously adsorb the leached gold on added activated carbon particles
 - strip the carbon particles of the adsorbed gold
 - stripped gold is then smelted
- Operation was reporting ~25% loss of Gold
- Comparative analysis between feed and leach residue using Feature Analysis
- Automated gold grain search
 - Evaluate degree of gold grain liberation
 - Evaluate compositional characteristics of gold grains



Ground Gold Ore

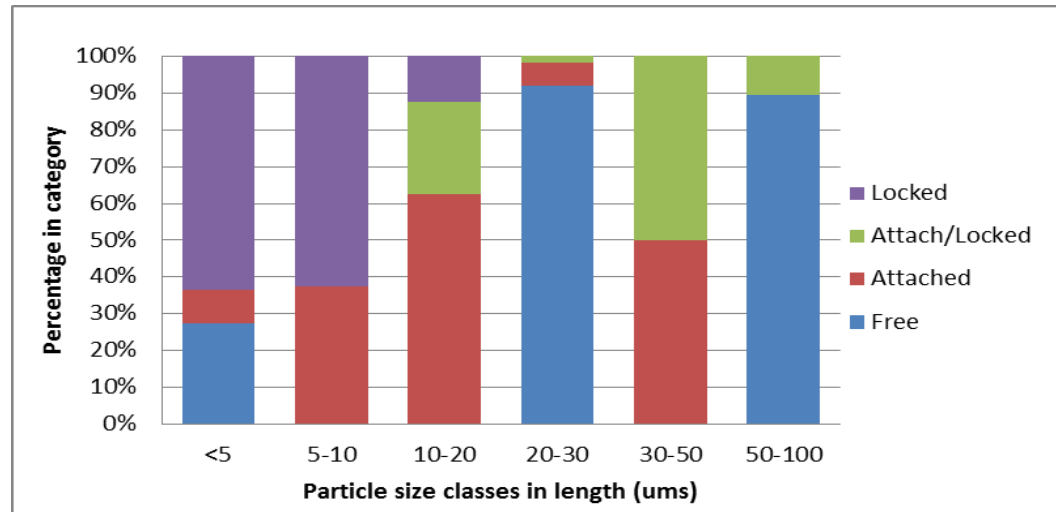
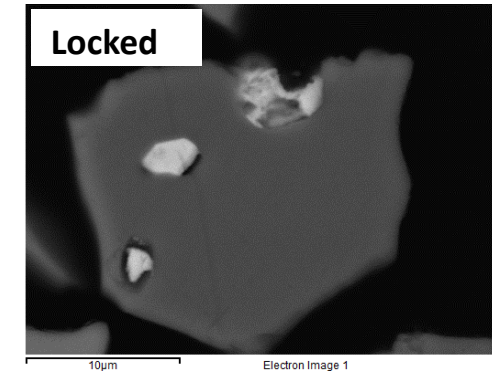
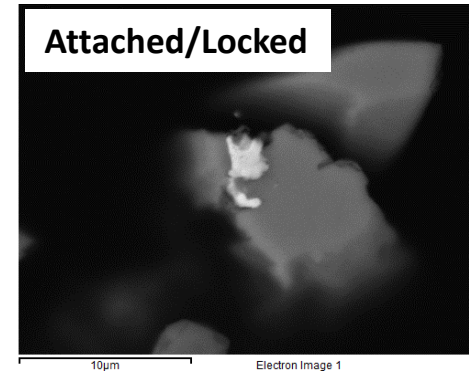
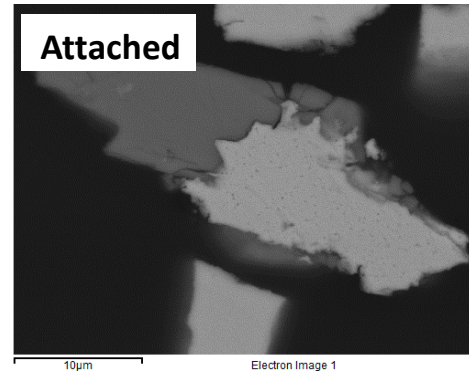
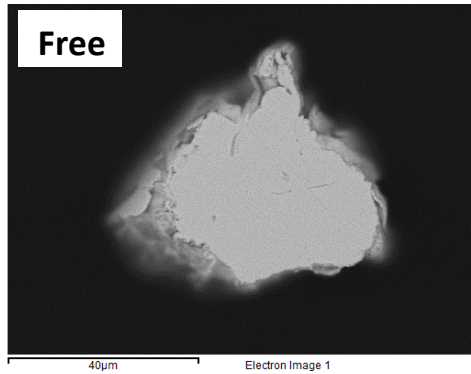
Leaching
Stripping
Smelting



Gold



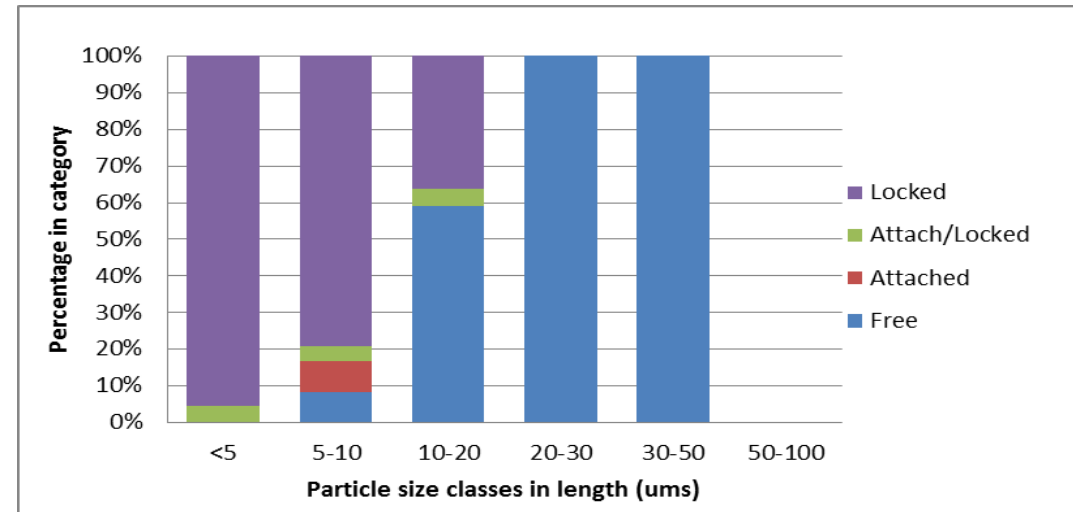
Evaluation and comparison of Au grains: degree of liberation



Size classified Au grains in the Feed

Feed results:

- *Au grains found in all size classes*
- *Liberation: variable across size classes*



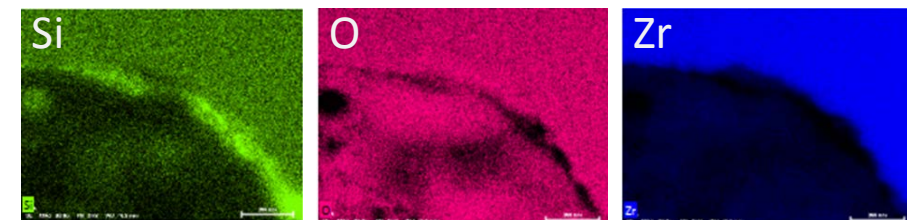
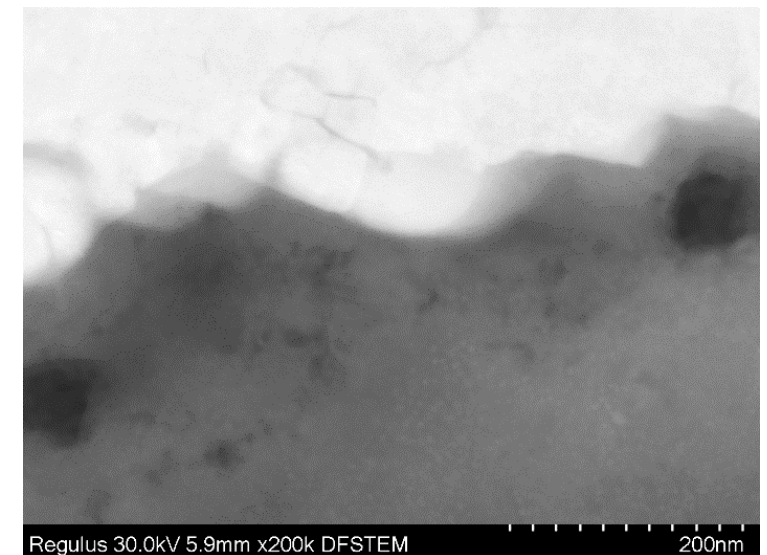
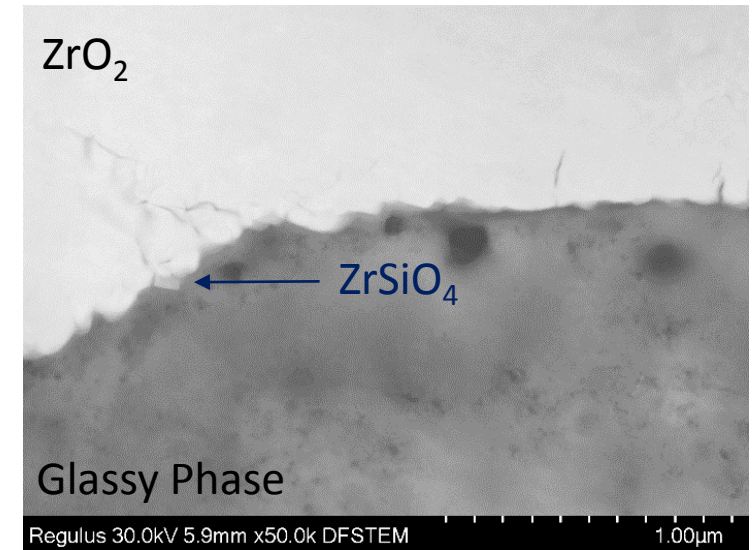
Size classified Au grains in the Leach residue

Leach residue results:

- *Au grains confined to size classes < 50 µm*
- *Liberation: fine sizes are locked; coarse sizes are free*
- *Composition of Au grains show high Bi and Sb*

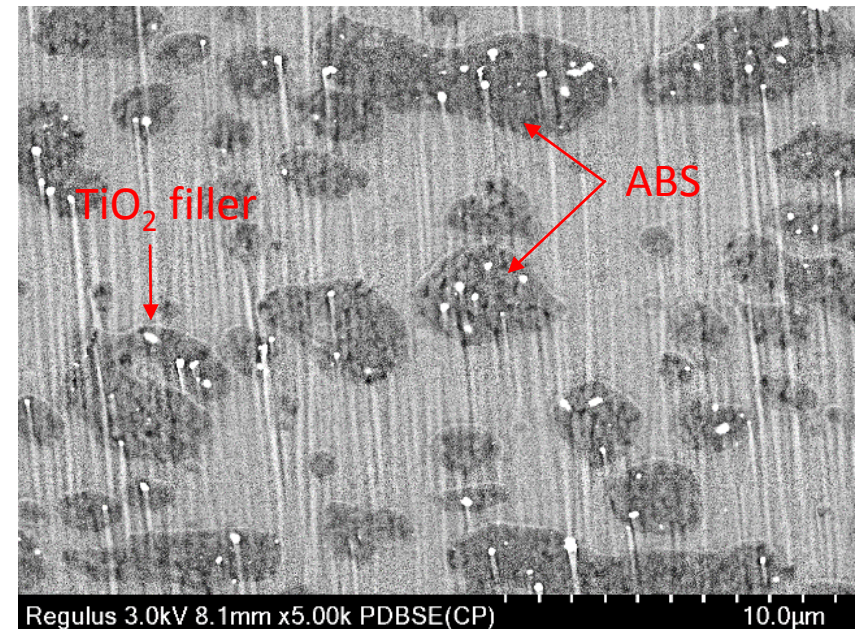
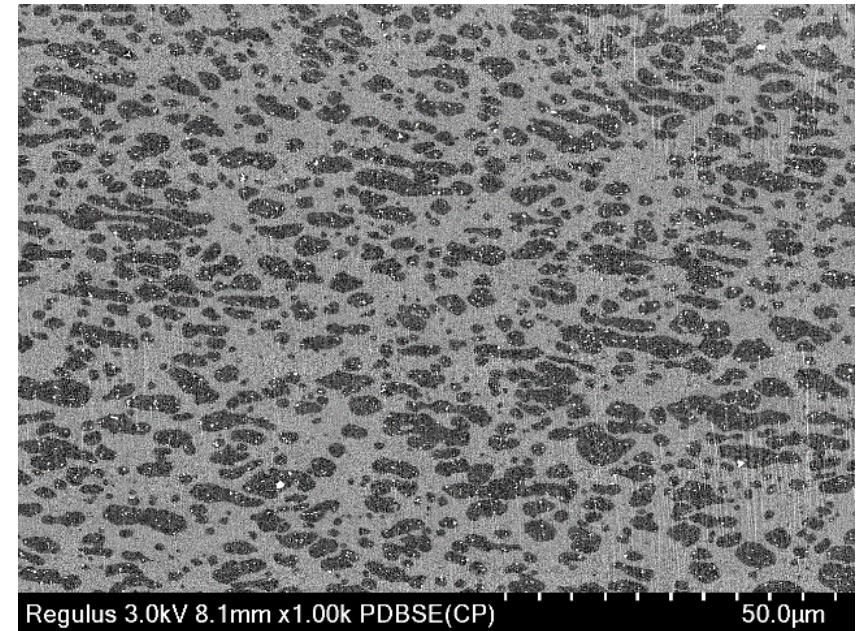
STEM Imaging: Mars Meteorite

- Ejected from Mars ~ 22 million years ago
- Sample experienced extreme pressure
- Interested in shock wave generated zircon interface
- Prepared thin section using focus Ion beam
- High angle transmitted electrons (DF-STEM)
- Dark field imaging shows contrast between phases (atomic number)

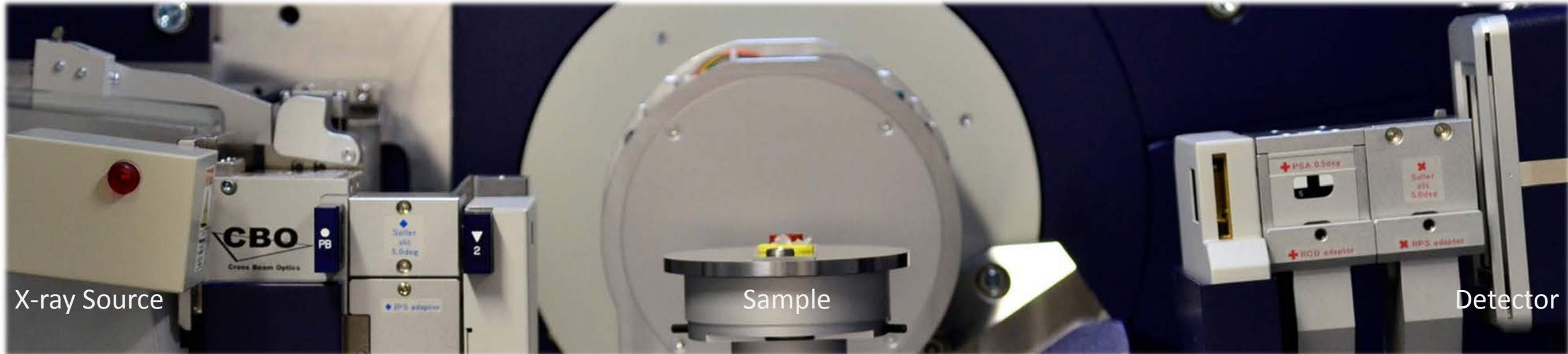


Molded PC-ABS Aircraft Parts

- Blend of three polymers
Polycarbonate + (acrylonitrile + butadiene)
- What to know the shape, size and distribution of domains
- Domains change the bulk and surface properties
- Prepared a cross-section using argon ion mill



X-ray Diffraction

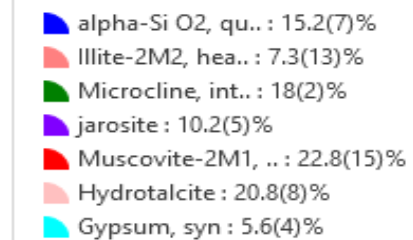
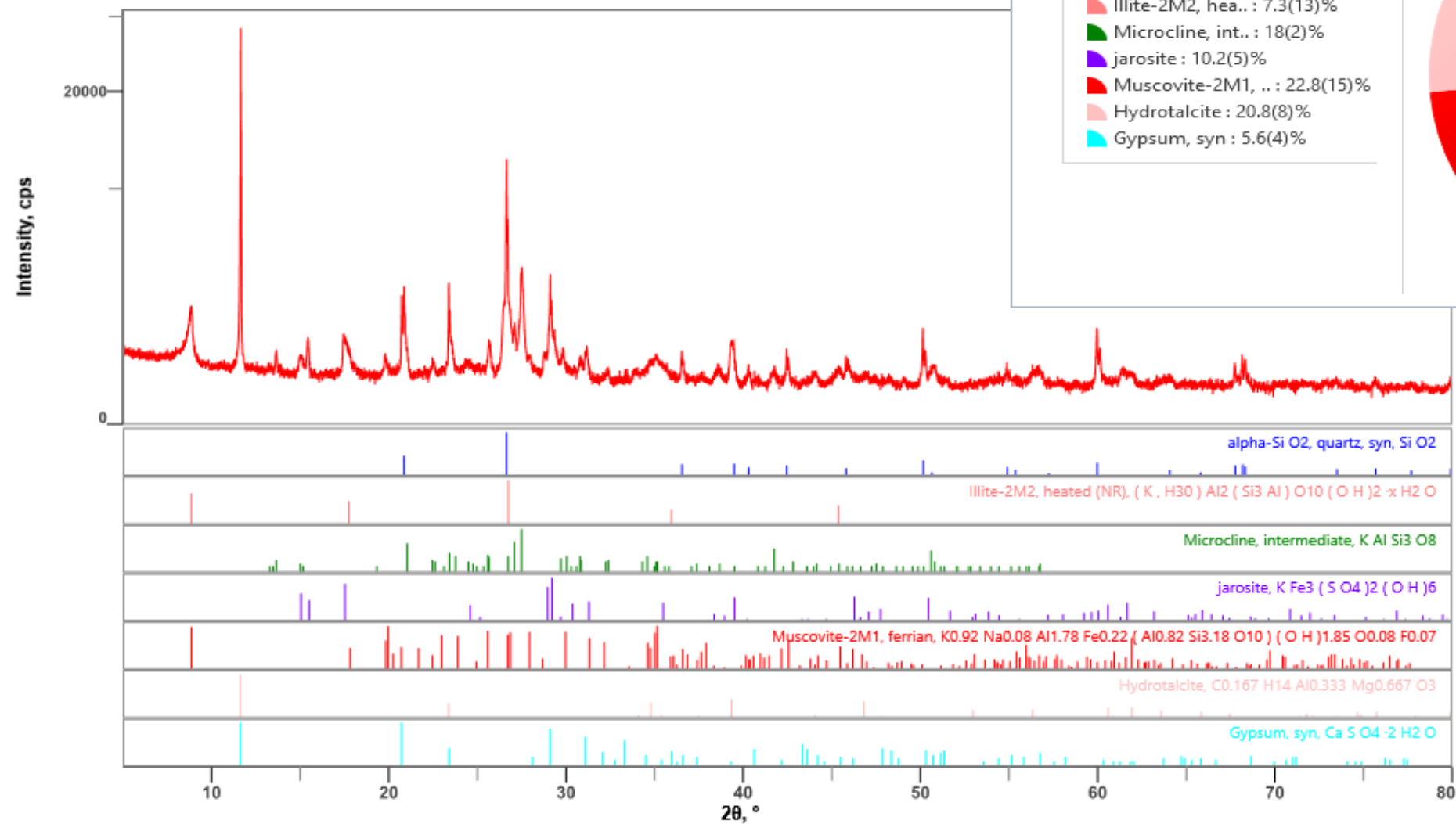


- Typically examine crystalline materials with repeating crystal structures
- Constructive interference between X-rays and crystal planes (Bragg's Law)
- Relatively fast technique to identify phases in materials

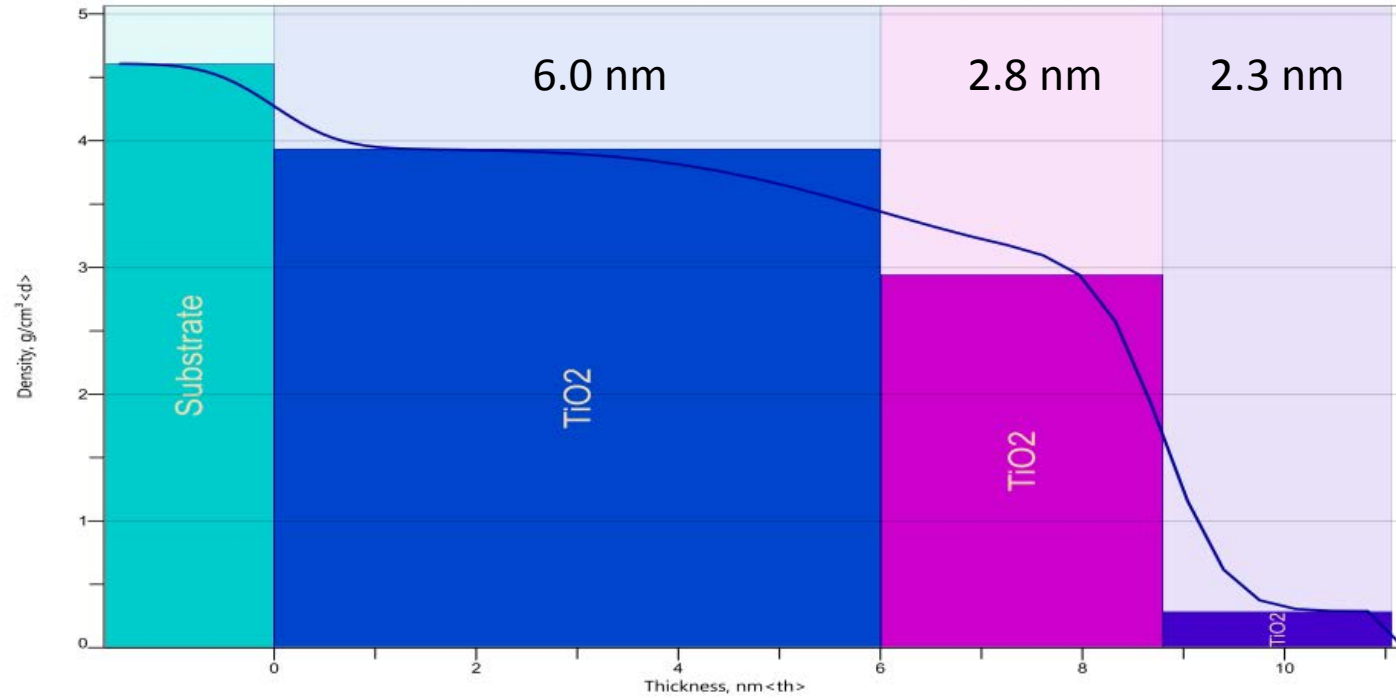
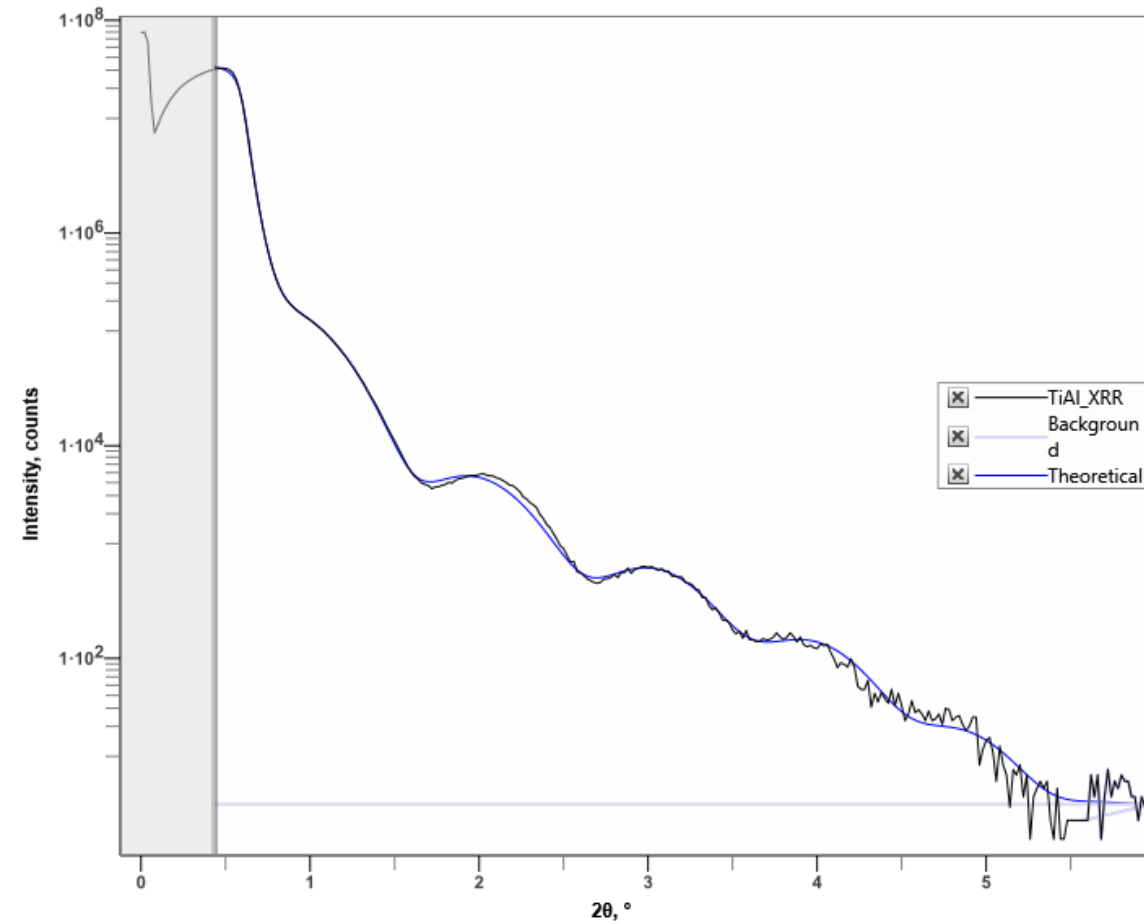
X-ray Diffraction

- Phase identification of powder and bulk samples
 - Qualitative & quantitative analysis
- Reflectometry
 - Thin film structure analysis (thickness and roughness)
- Grazing incidence XRD
 - Phase identification of coating while minimizing substrate contribution
- SAXS (Small Angle X-ray Scattering)
 - Determine particle size distribution (1 to 100 nm)
- Residual stress analysis

Powder XRD (Minerals)



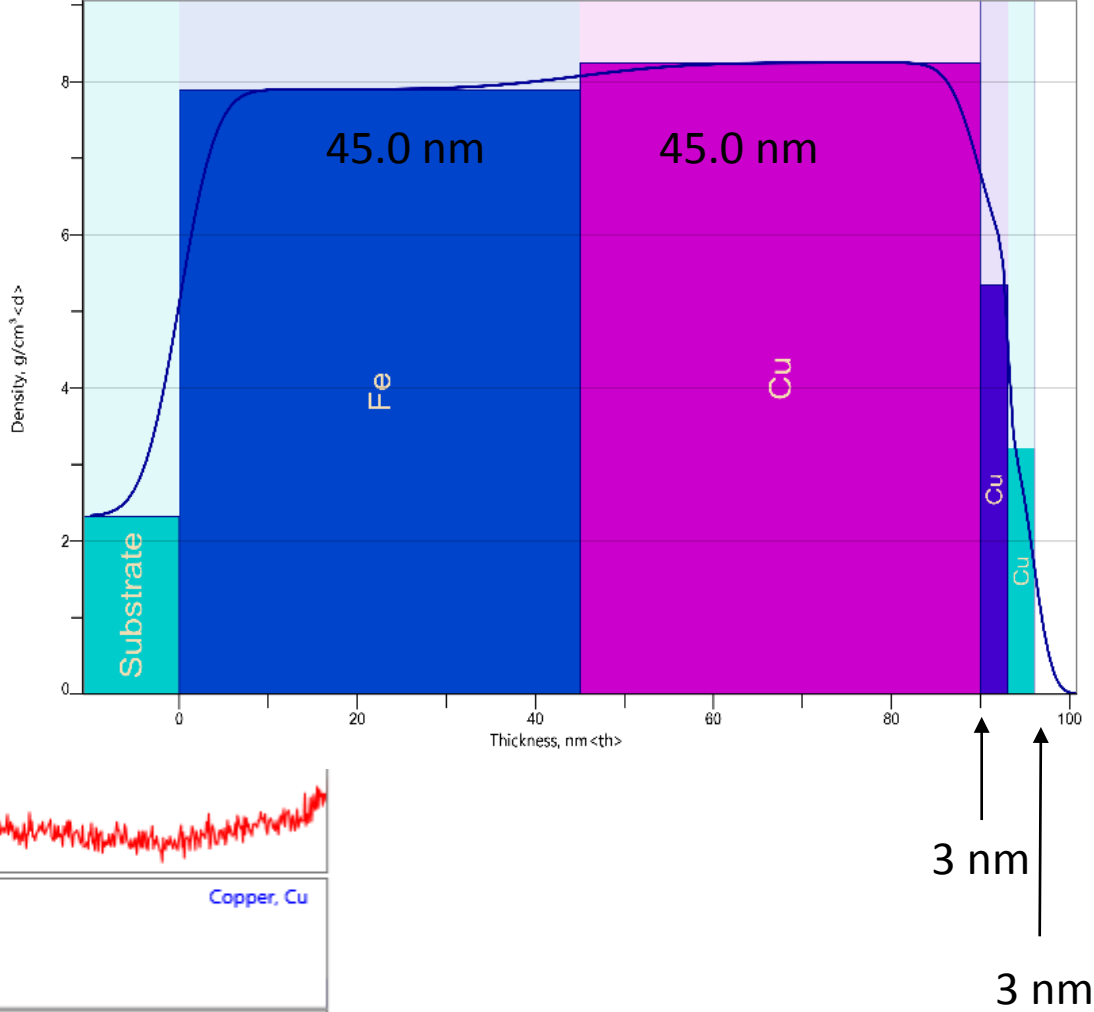
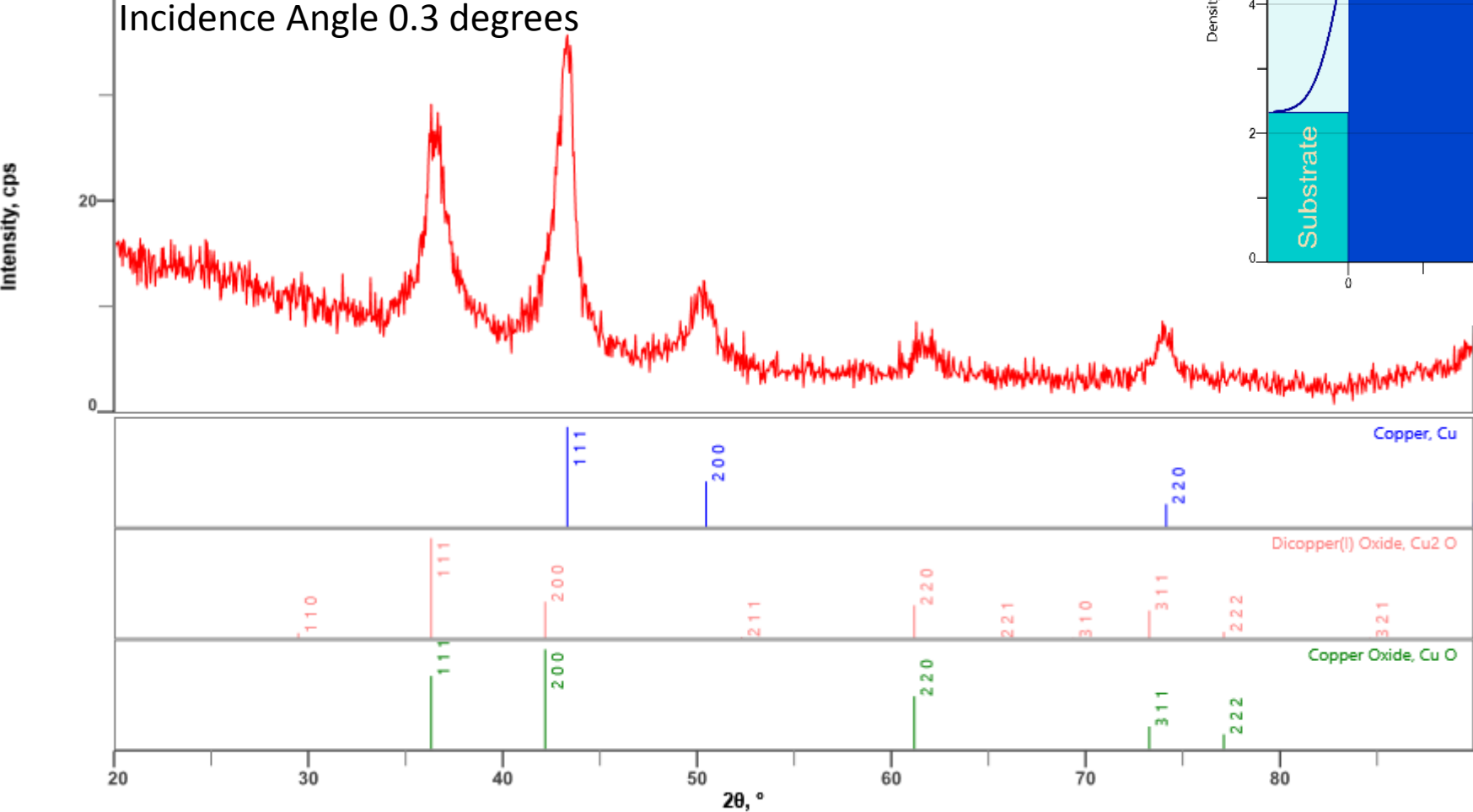
Thin Film Analysis (Reflectometry)



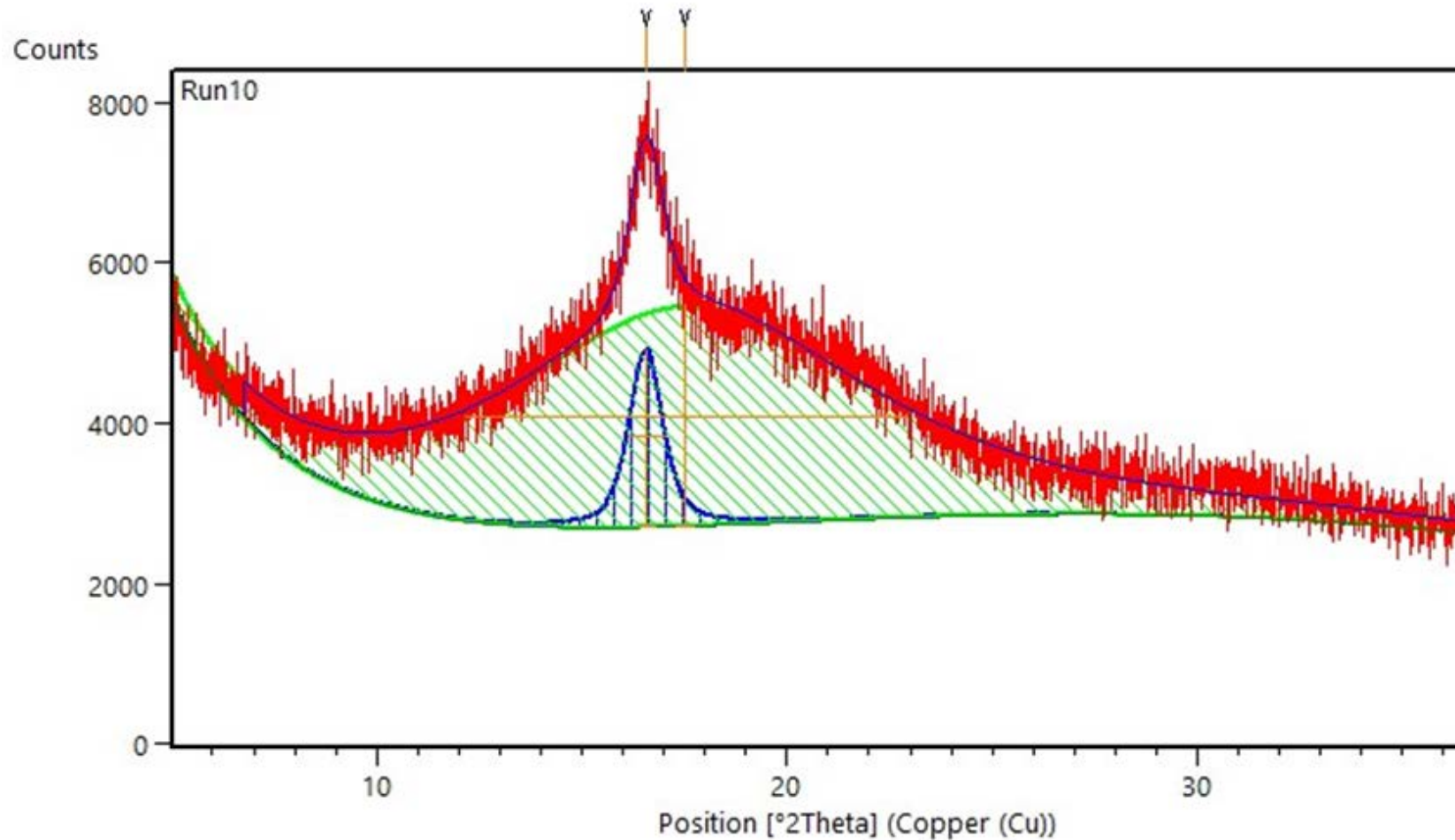
Native oxide on titanium alloy

Thin Film Analysis

Incidence Angle 0.3 degrees



Degree of Crystallinity in a Polymer



Summary

- These techniques can be used to solve manufacturing issues from many sectors including aerospace, automotive, medical and semiconductor etc.
- **SEM/EDX spectroscopy fast technique to examine materials**
 - High resolution images
 - Atomic number contrast
 - Elemental analysis (Be to U)
 - Semi-quantitative
 - detection limit 0.1 weight %
- **XRD quickly identifies phases in a variety of materials**
 - Thin film thickness measurements
 - Particle / void size measurements
 - Stress analysis
 - Preferred orientation