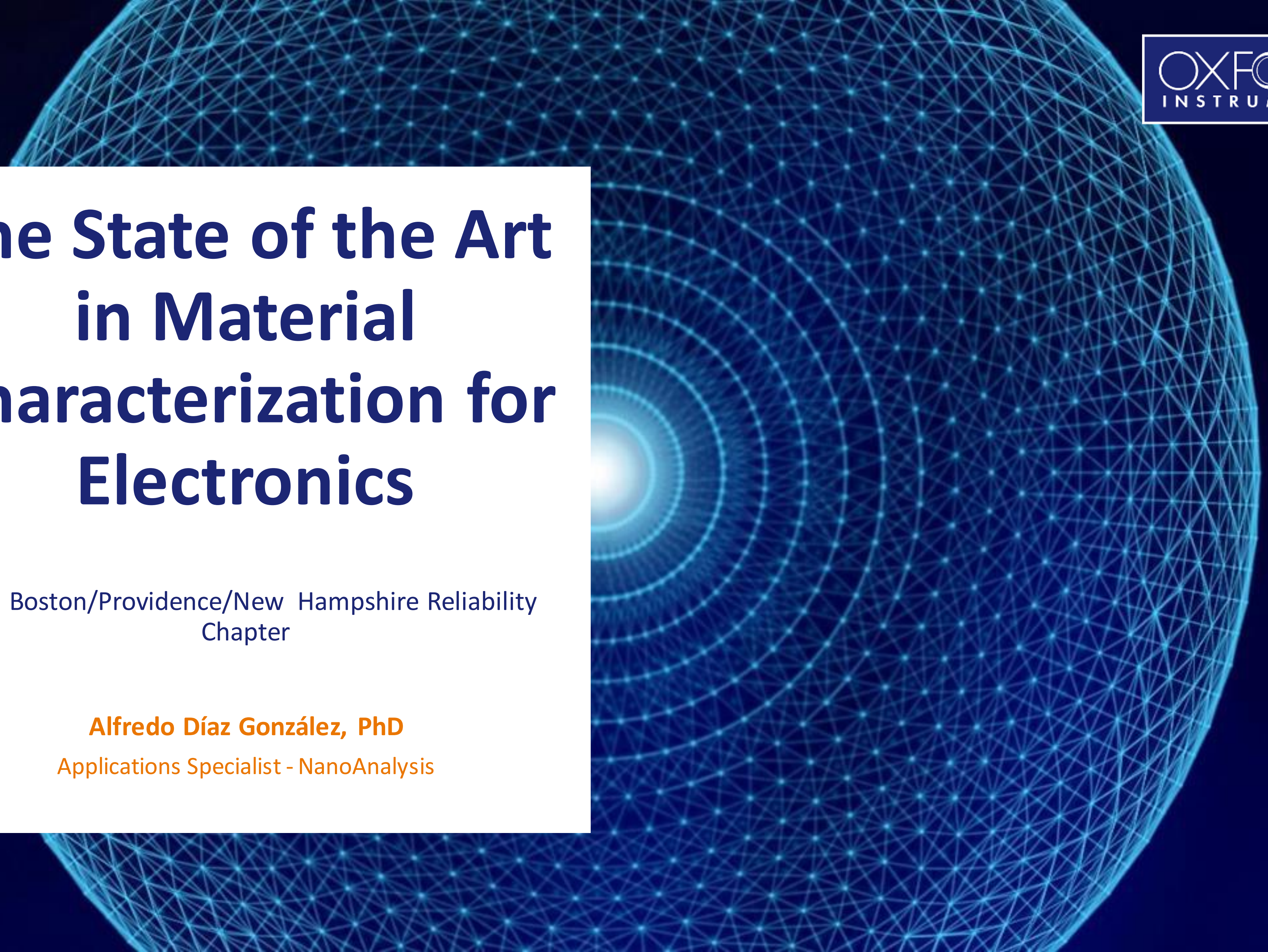


The State of the Art in Material Characterization for Electronics

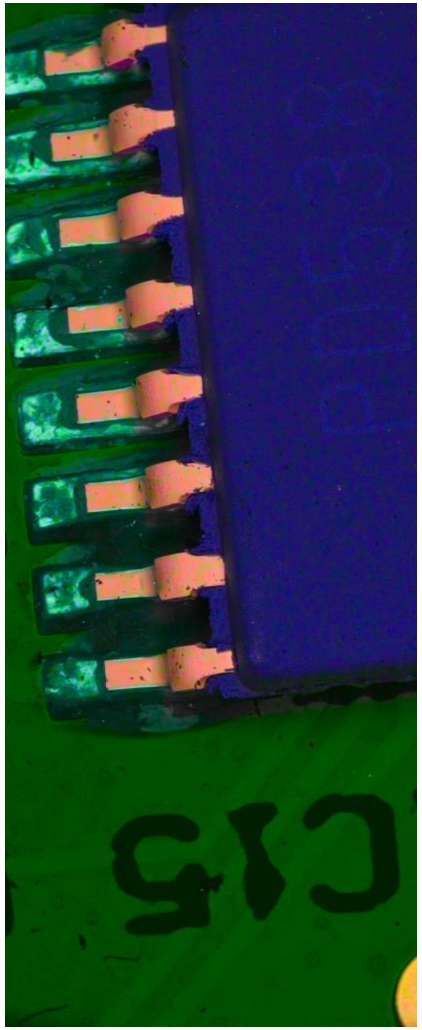
IEEE Boston/Providence/New Hampshire Reliability Chapter

Alfredo Díaz González, PhD

Applications Specialist - NanoAnalysis

Outline

- Introducing Oxford Instruments
- SEM-based Characterization: The Basics
- Specific Techniques: The Basics and Examples
- Non-SEM Characterization: The Basics and Examples
- Questions and Answers



Oxford Instruments: who we are

Founded in 1959 as the first commercial spin out from Oxford University, we are a global provider of high technology products and services to the world's leading companies and foremost scientific research communities. Our products enable customers to image, analyse, control and manipulate materials down to the atomic and molecular level.

Our purpose is to enable a greener, healthier, more connected advanced society

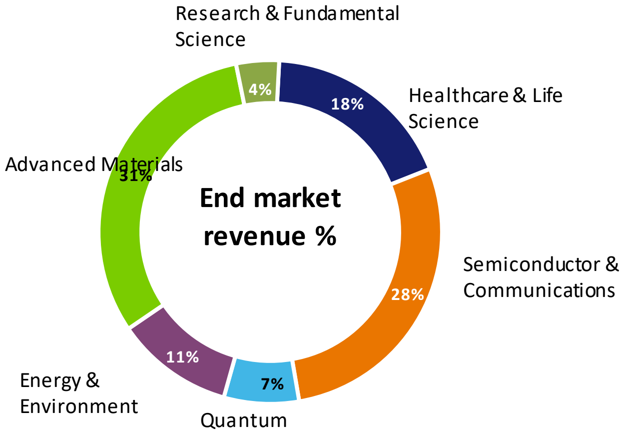
We enable our customers to

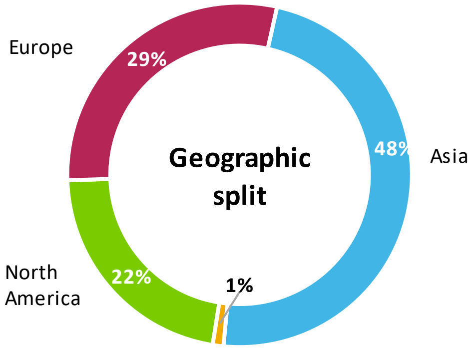
Make new scientific discoveries

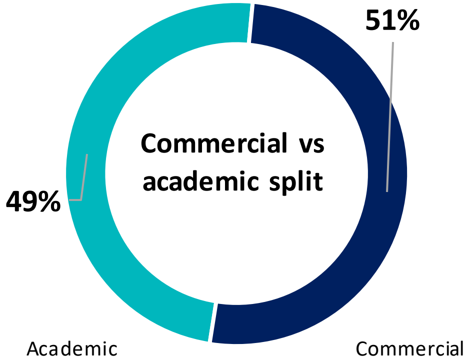
Accelerate applied R&D

Increase productivity in high-tech manufacturing

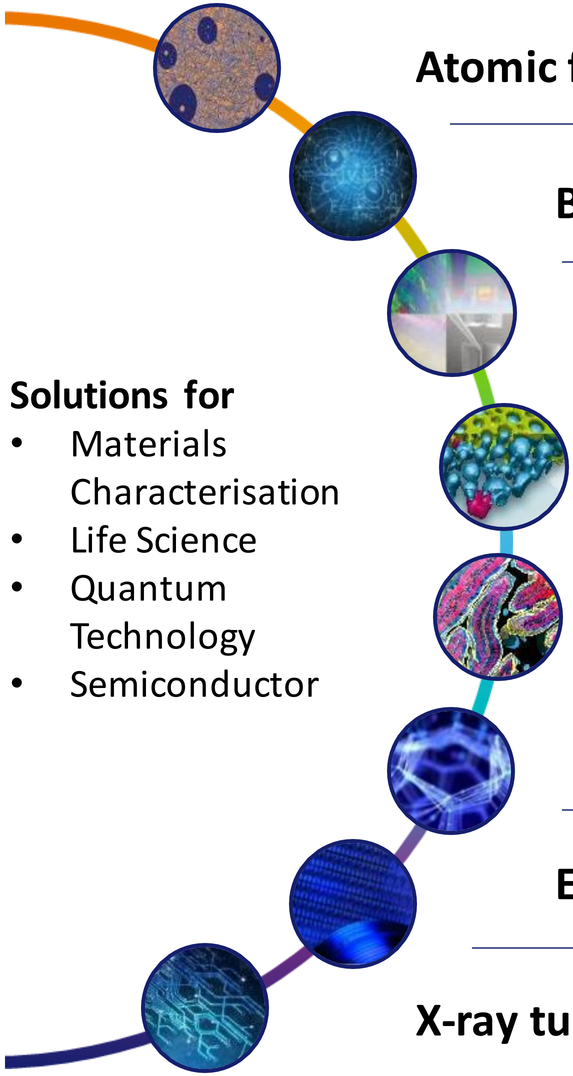
Accelerating global progress in key areas including healthcare, energy and environment, and advanced materials - the building blocks of modern society







Power your research with our solutions



Solutions for
- Materials Characterisation
- Life Science
- Quantum Technology
- Semiconductor

Atomic force microscopy (AFM) Asylum Research

Benchtop nuclear magnetic resonance (NMR) spectrometers Magnetic Resonance

Analytics for electron microscopy NanoAnalysis

3D Raman & correlative microscopes WITec

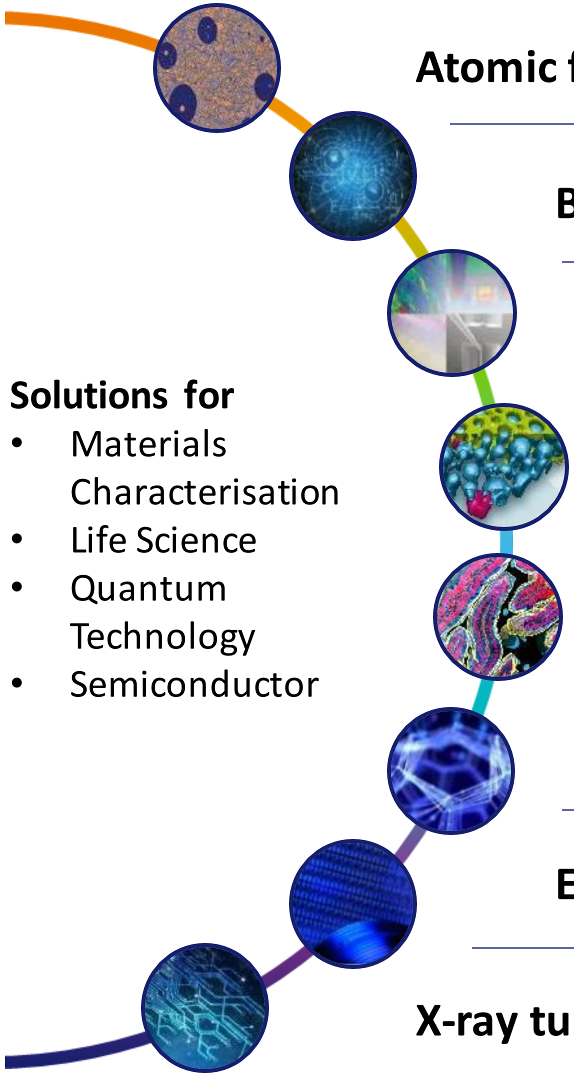
Scientific cameras, microscopy solutions, spectrographs & cryostats Andor

Dilution refrigeration and superconducting magnets NanoScience

Etch & deposition processing equipment, solutions & recipes Plasma Technology

X-ray tubes, power supplies & integrated X-ray sources X-ray Technology

Power your research with our solutions



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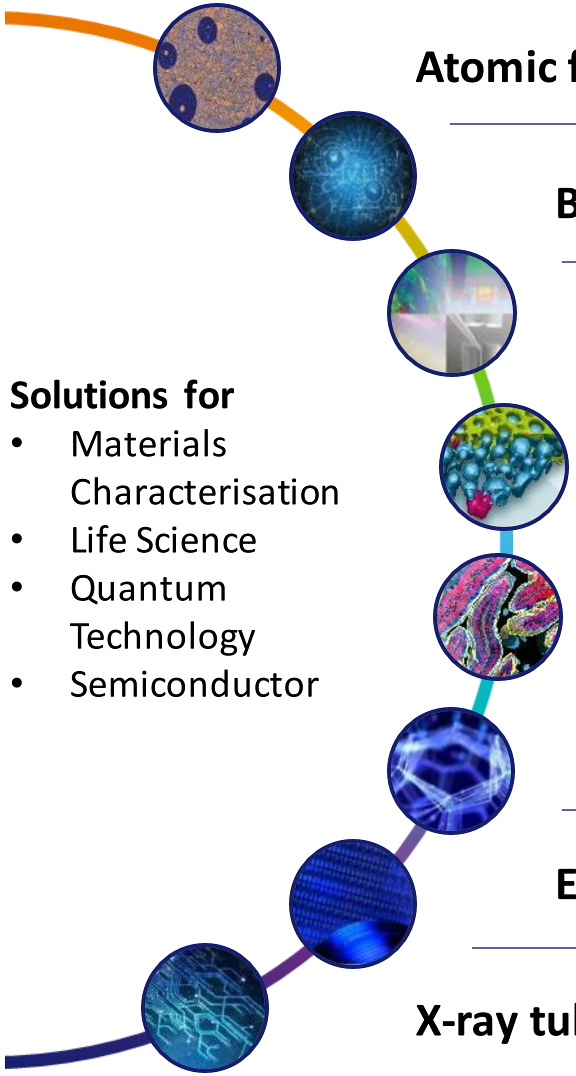

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Solutions for

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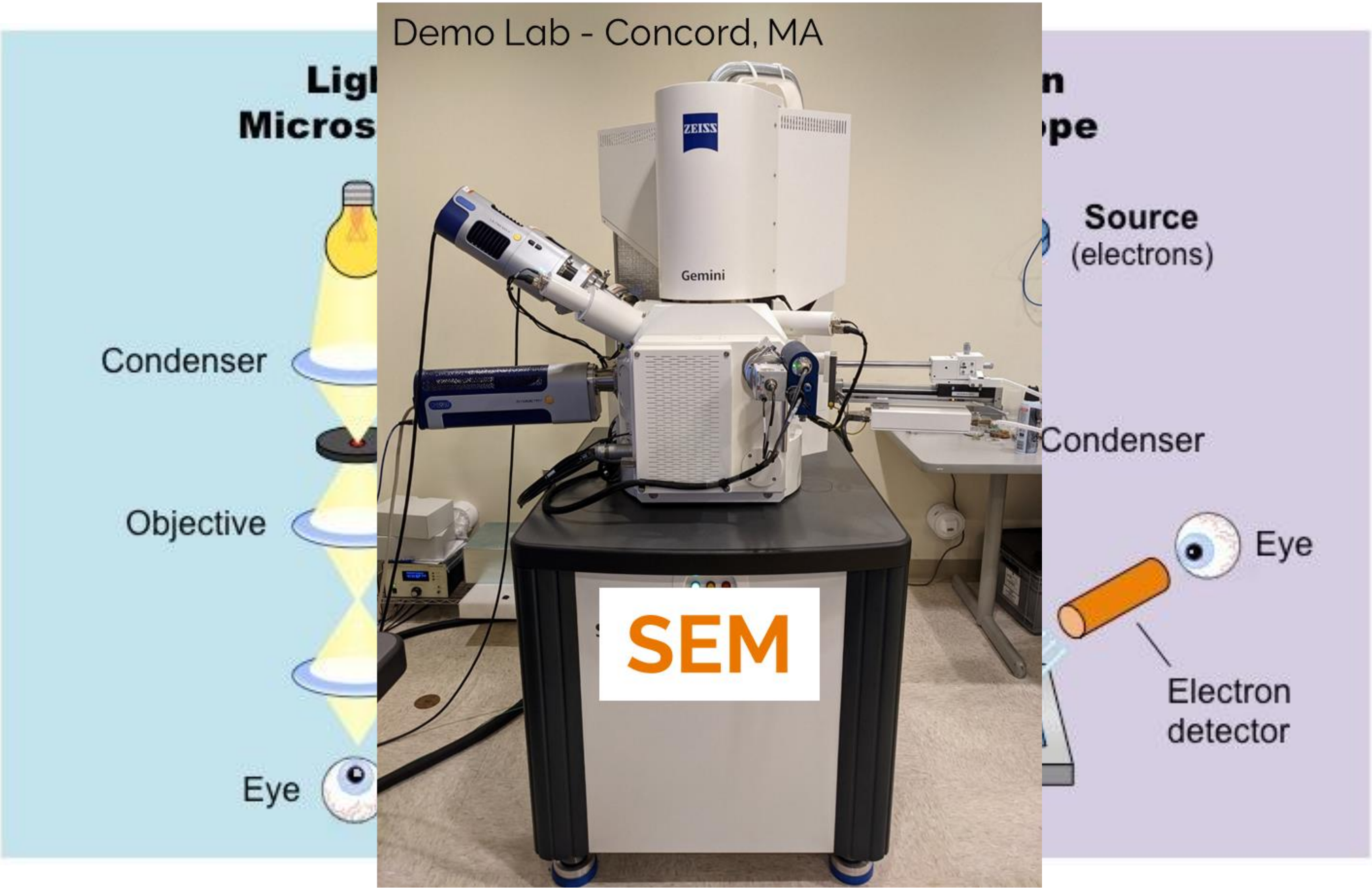
- Life Science
- Quantum Technology
- Semiconductor

Scanning Electron Microscopy

Scanning Electron Microscope

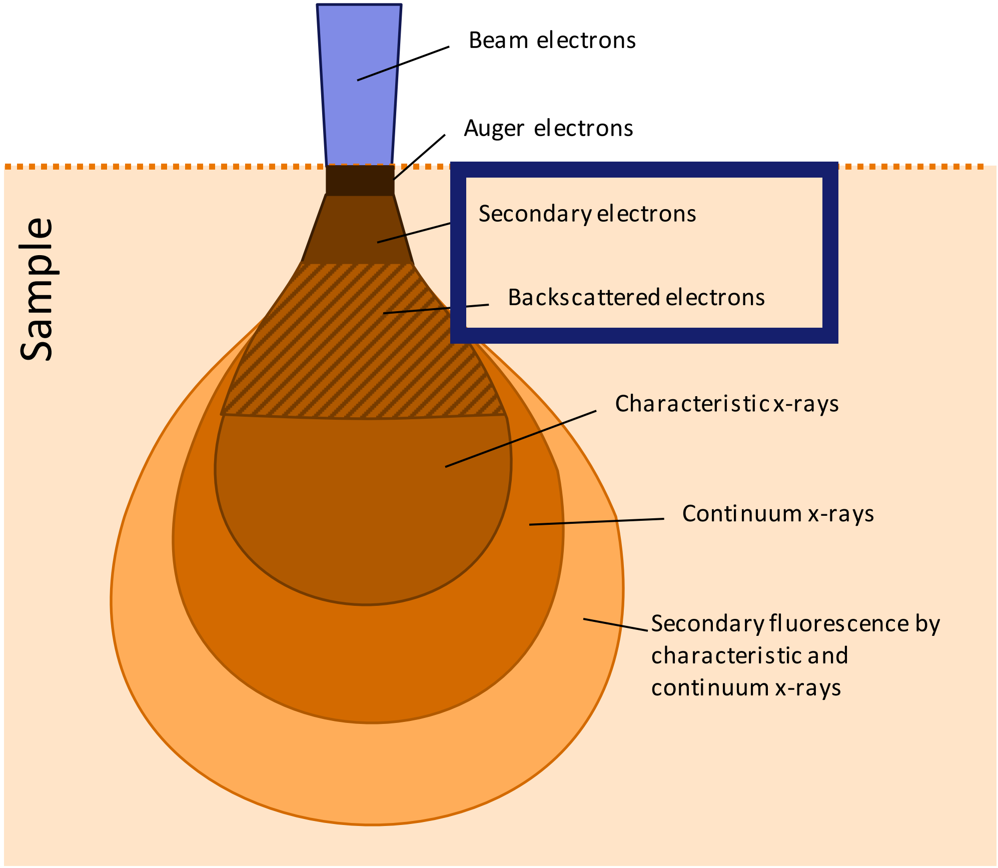


Demo Lab - Concord, MA

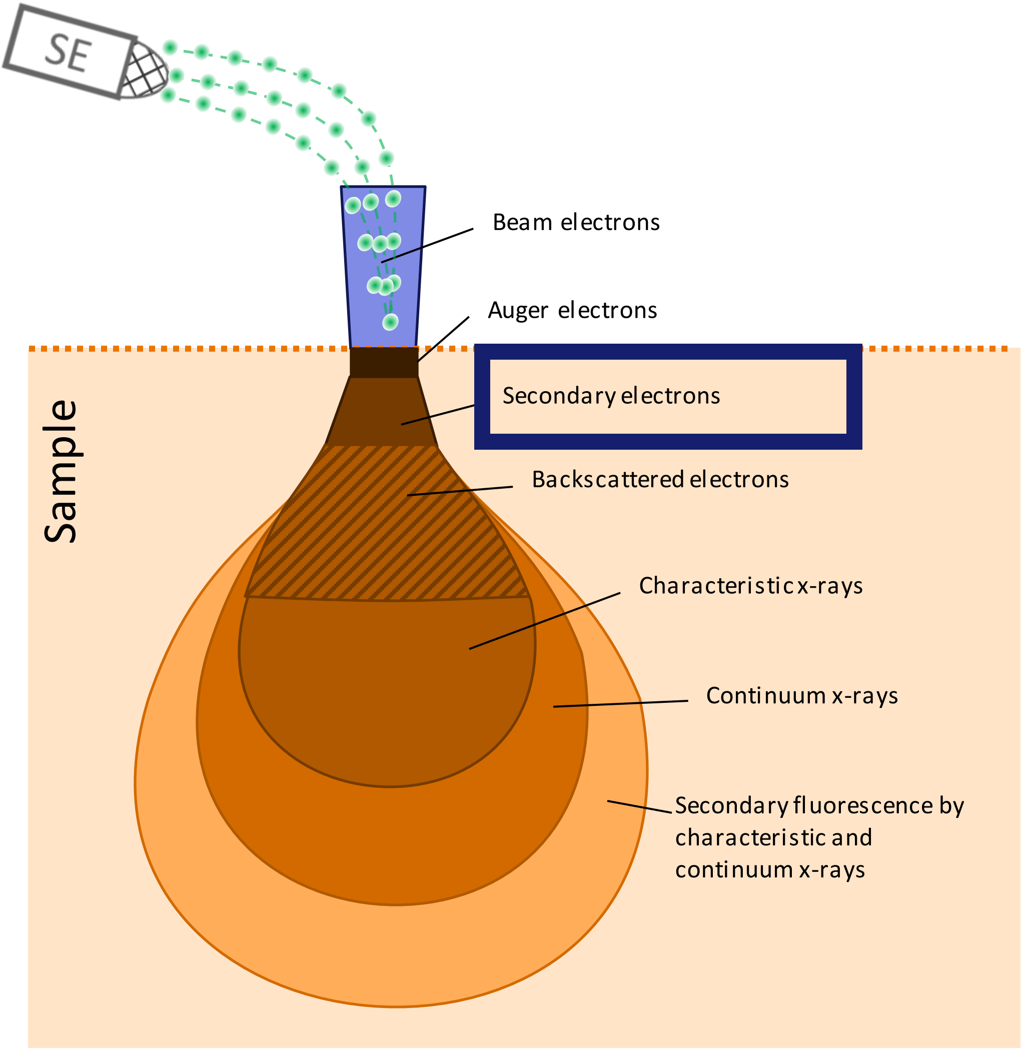
SEM

Scanning Electron Microscope

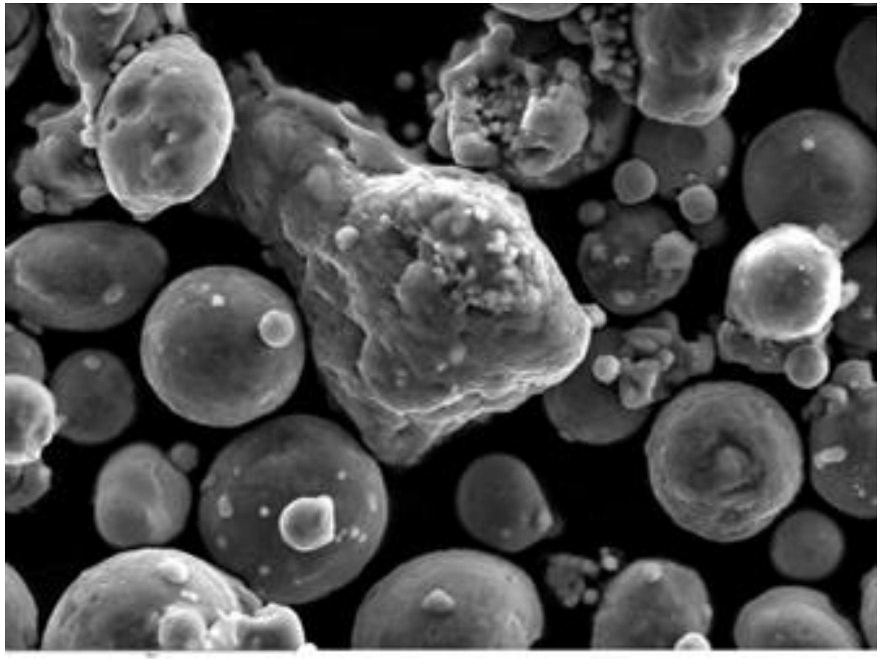
Electron Beam – Sample Interaction



Scanning Electron Microscope: Imaging



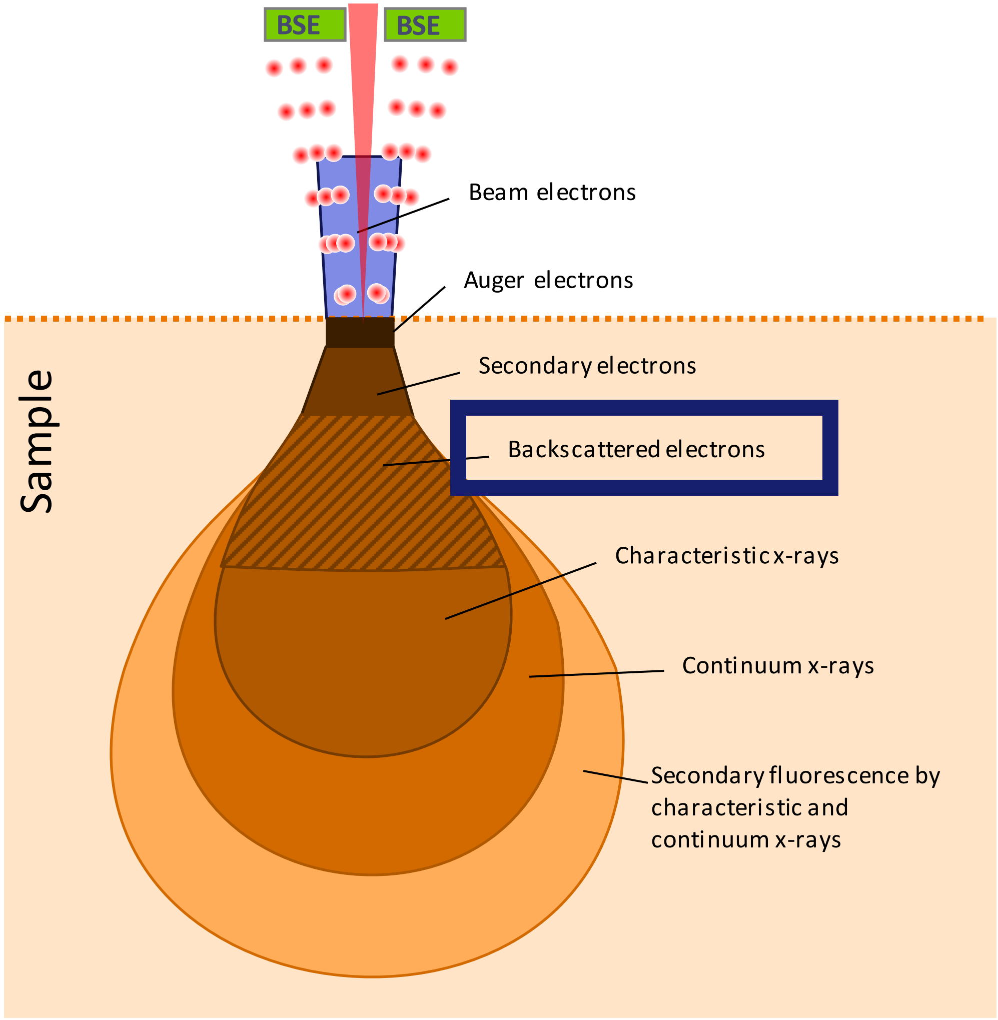
Secondary Electron (SE) Detectors



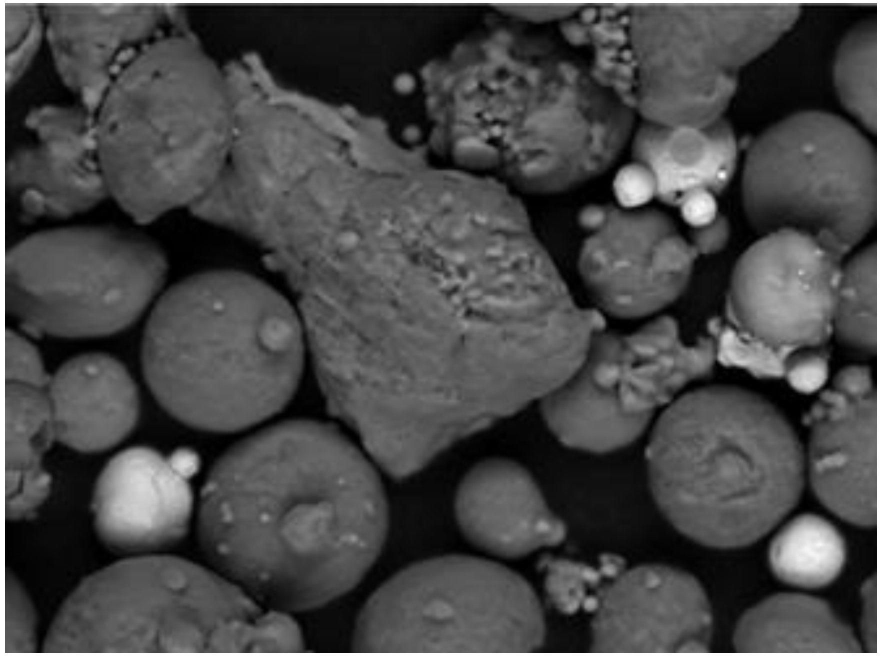
SE Image
Greyscale
Topography contrast

- Detects low energy (50eV) SEs
- Signal originates from the near-surface regions
- Provides useful topographical information about the surface

Scanning Electron Microscope: Imaging



Backscattered Electron (BSE) Detectors

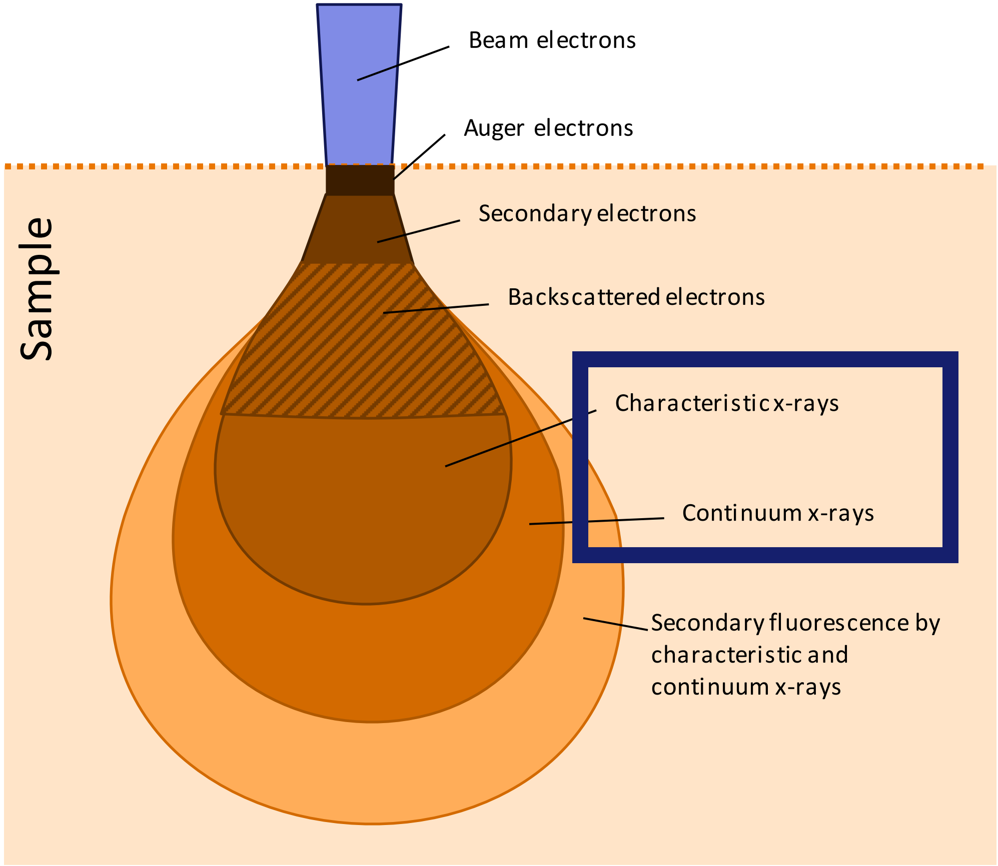


BSE Image
Greyscale
Z-number contrast
Topography contrast

- High energy electrons (Originate from deeper and larger regions)
- Provides indicative information on atomic number (Z)
- Segmented detectors can show topography

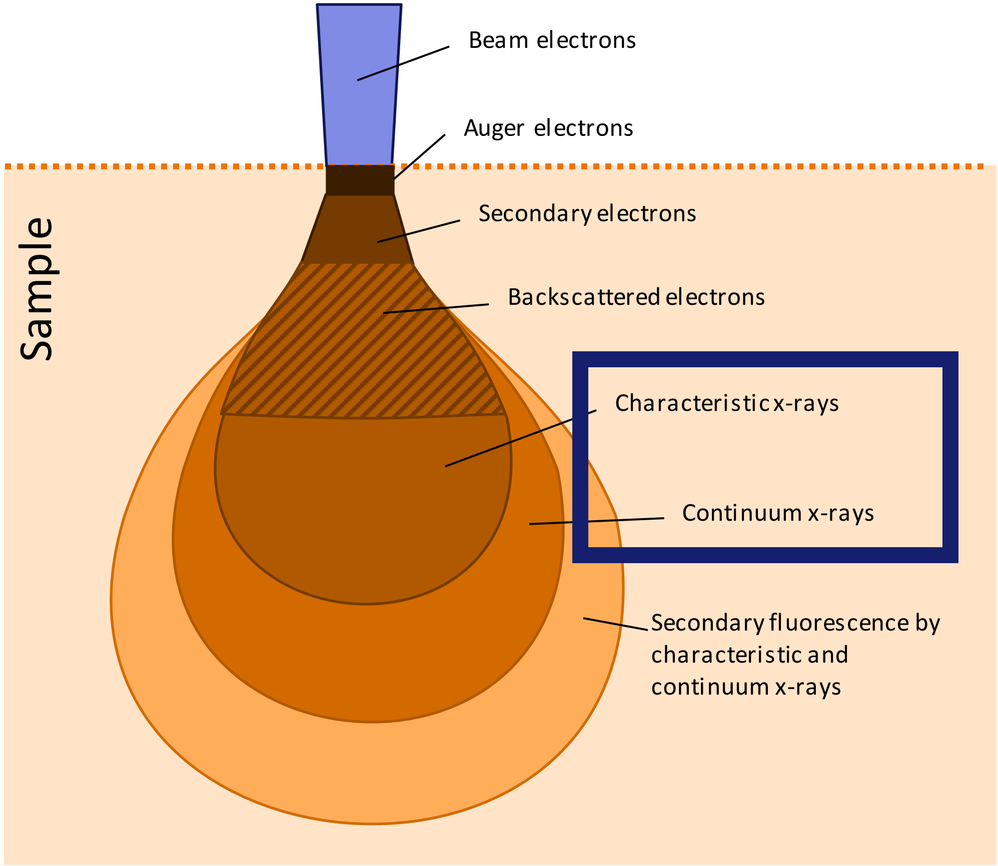
Scanning Electron Microscope

Electron Beam – Sample Interaction

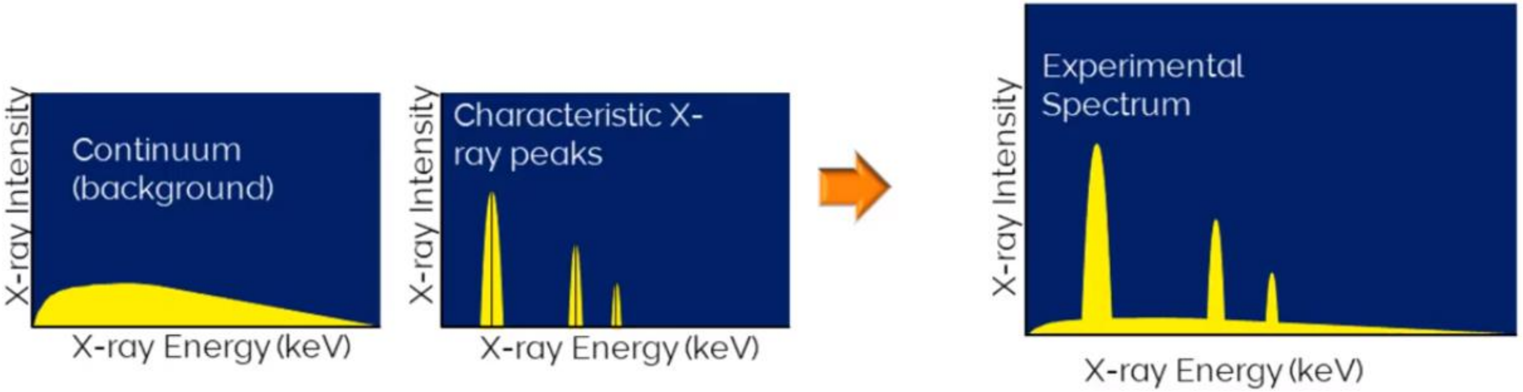


Scanning Electron Microscope: X-rays

Electron Beam – Sample Interaction

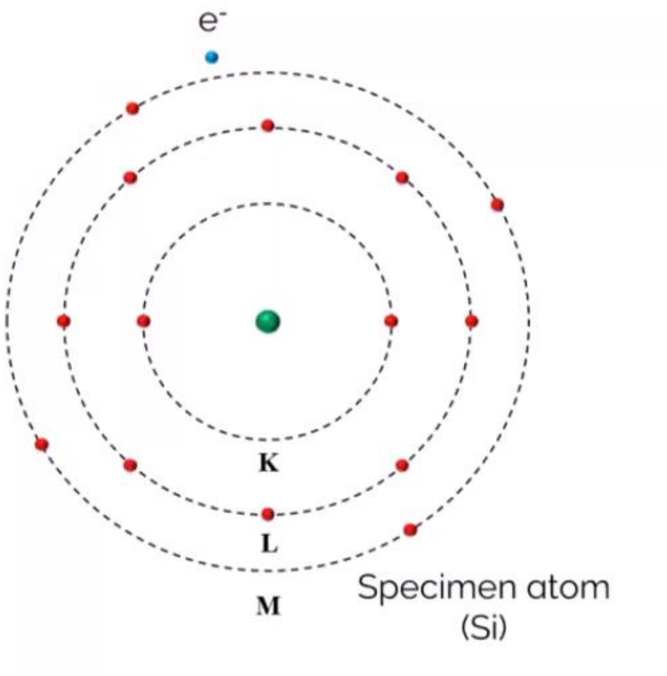


- X-rays are emitted from a material when it is ionized by an electron
- The x-rays are acquired and characterized
- An x-ray spectrum consist of two components:

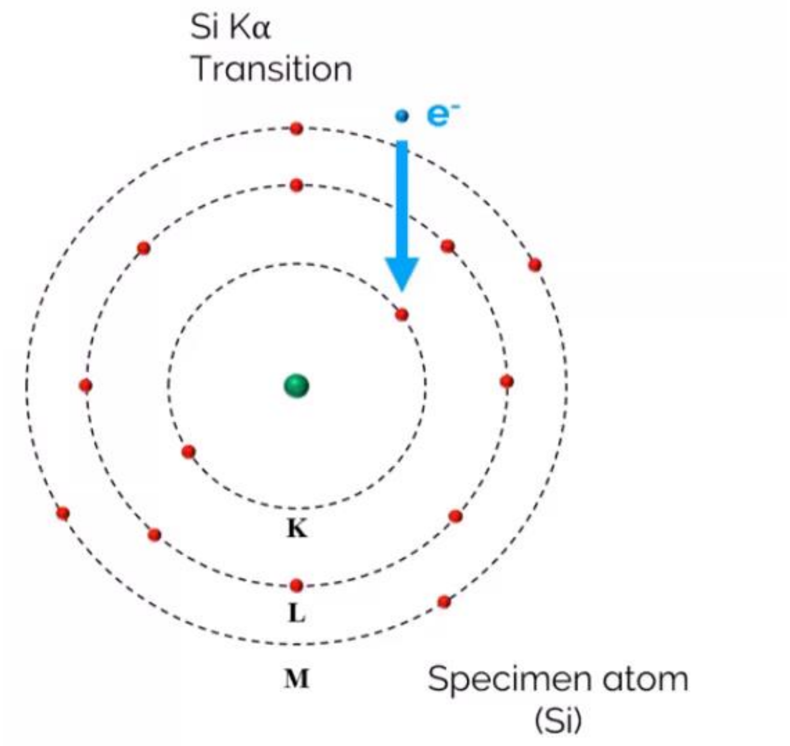


Fundamentals: e$^-$ Beam and Sample Interaction

Continuum or background
(Bremsstrahlung radiation)



Characteristic X-rays



Fundamentals: e^- Beam and Sample Interaction

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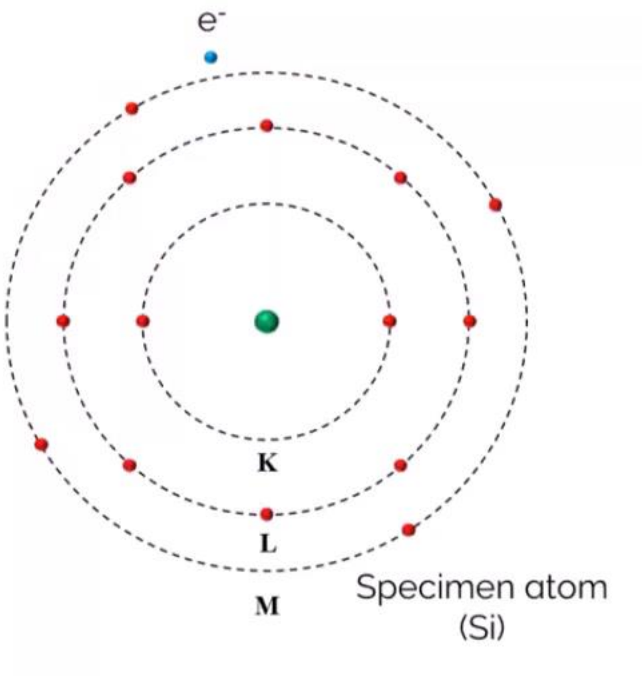
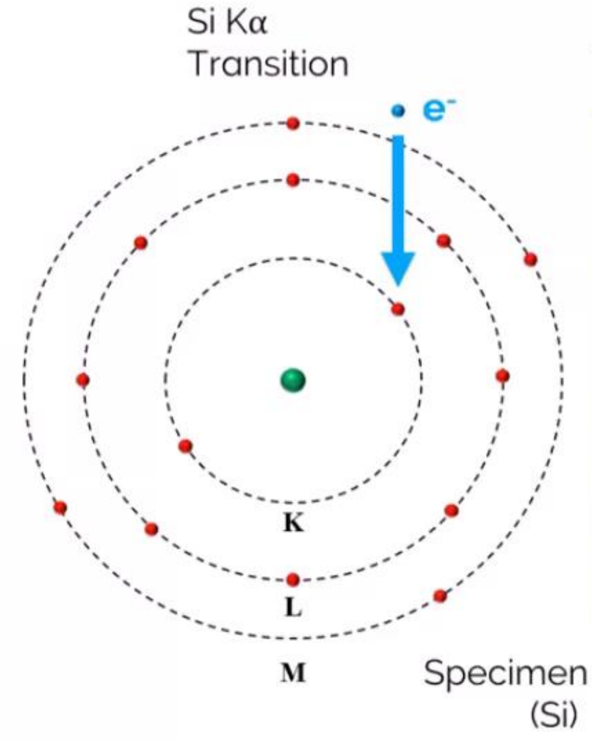
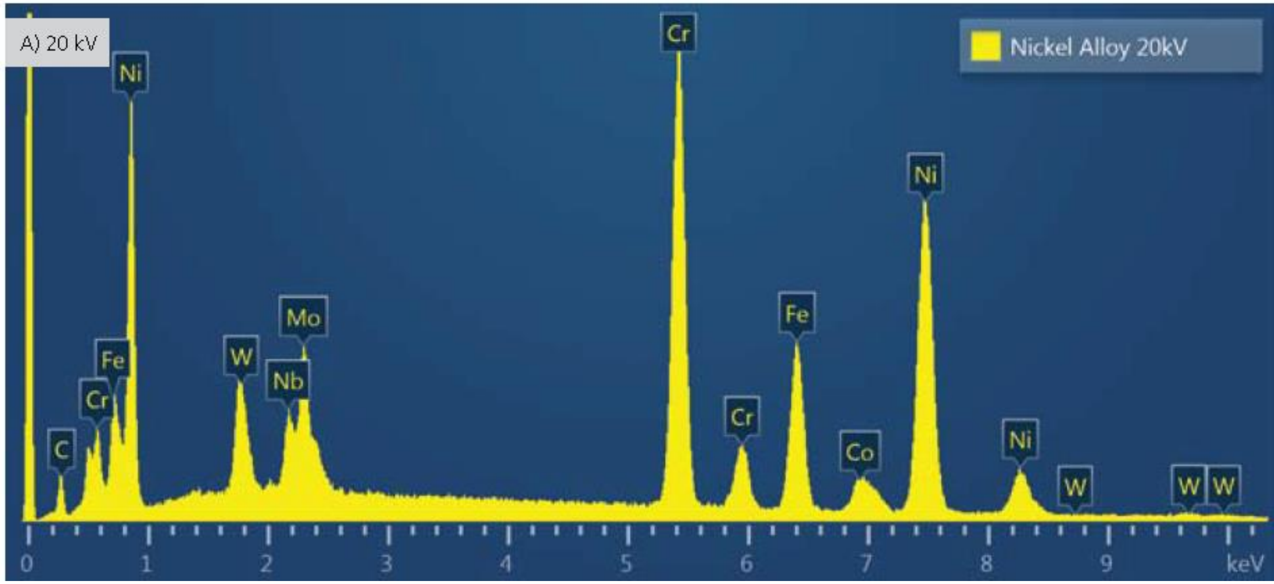
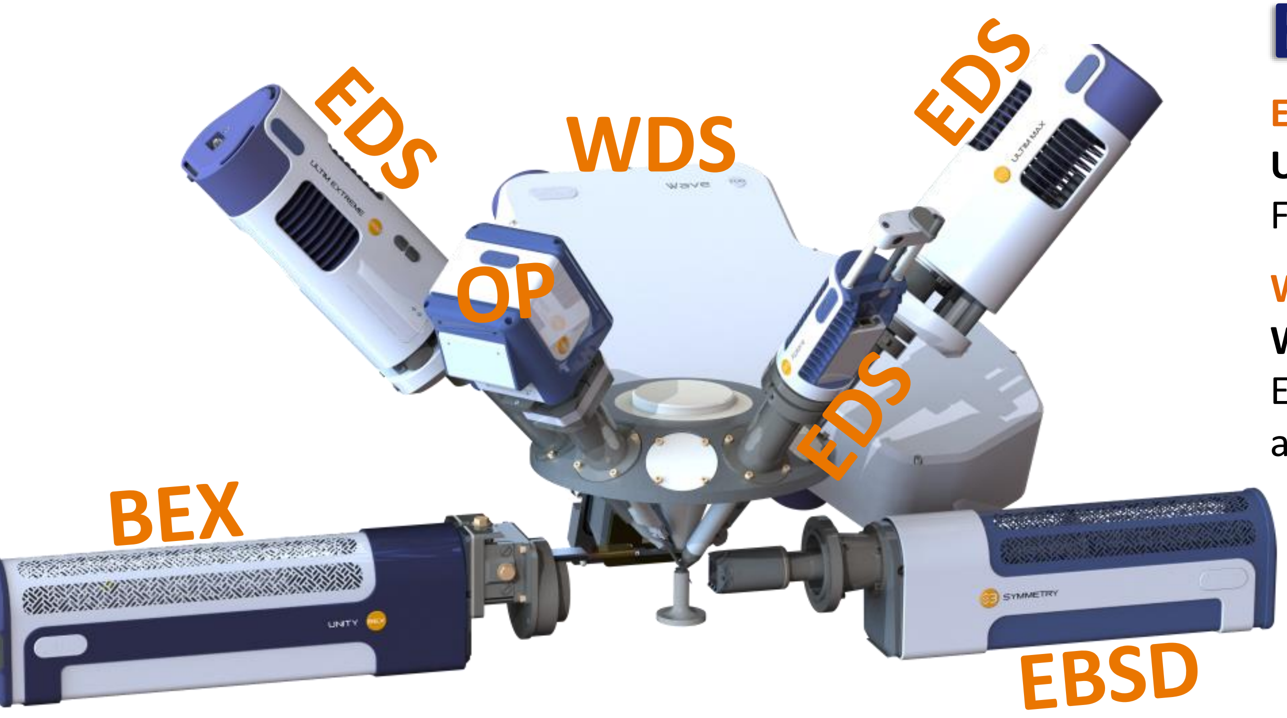


Characteristic X-rays





Electron Microscopy Solutions



EDS & WDS

Energy Dispersive X-ray Spectroscopy

Ultim Max & Ultim Extreme silicon drift detectors

Fast and accurate elemental characterization

Wavelength Dispersive X-ray Spectroscopy

Wave

Elemental characterization with ultimate spectral resolution for accurate quantification of trace elements

BEX

Backscattered Electron & X-ray Imaging

Unity

High-definition color imaging embedded with elemental data as you navigate your sample

Nanomanipulation

OmniProbe

Sample manipulation and lift-out

EBSD

Electron Backscatter Diffraction

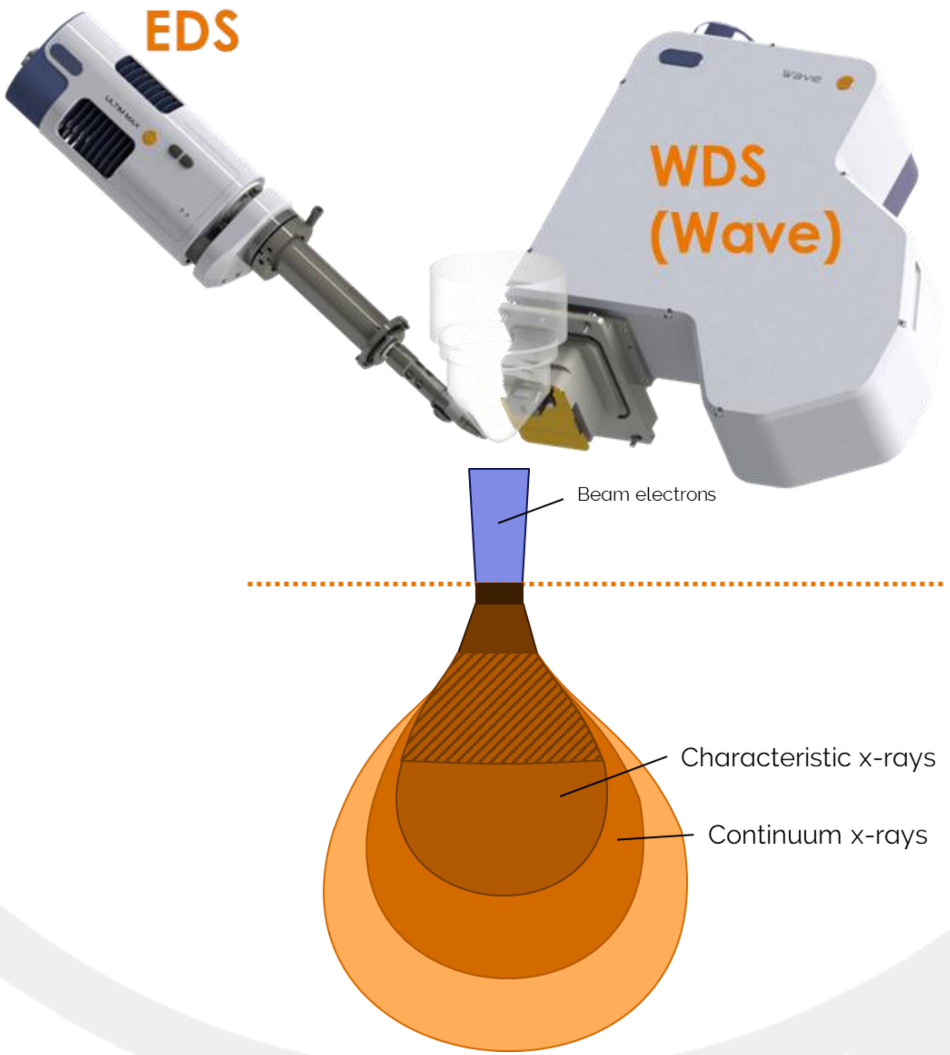
Symmetry CMOS camera

Structural analysis and phase identification

EDS & WDS

Energy Dispersive X-ray Spectroscopy

Wavelength Dispersive X-ray Spectroscopy



High Spatial Resolution Analysis: Bulk SRAM device on SEM

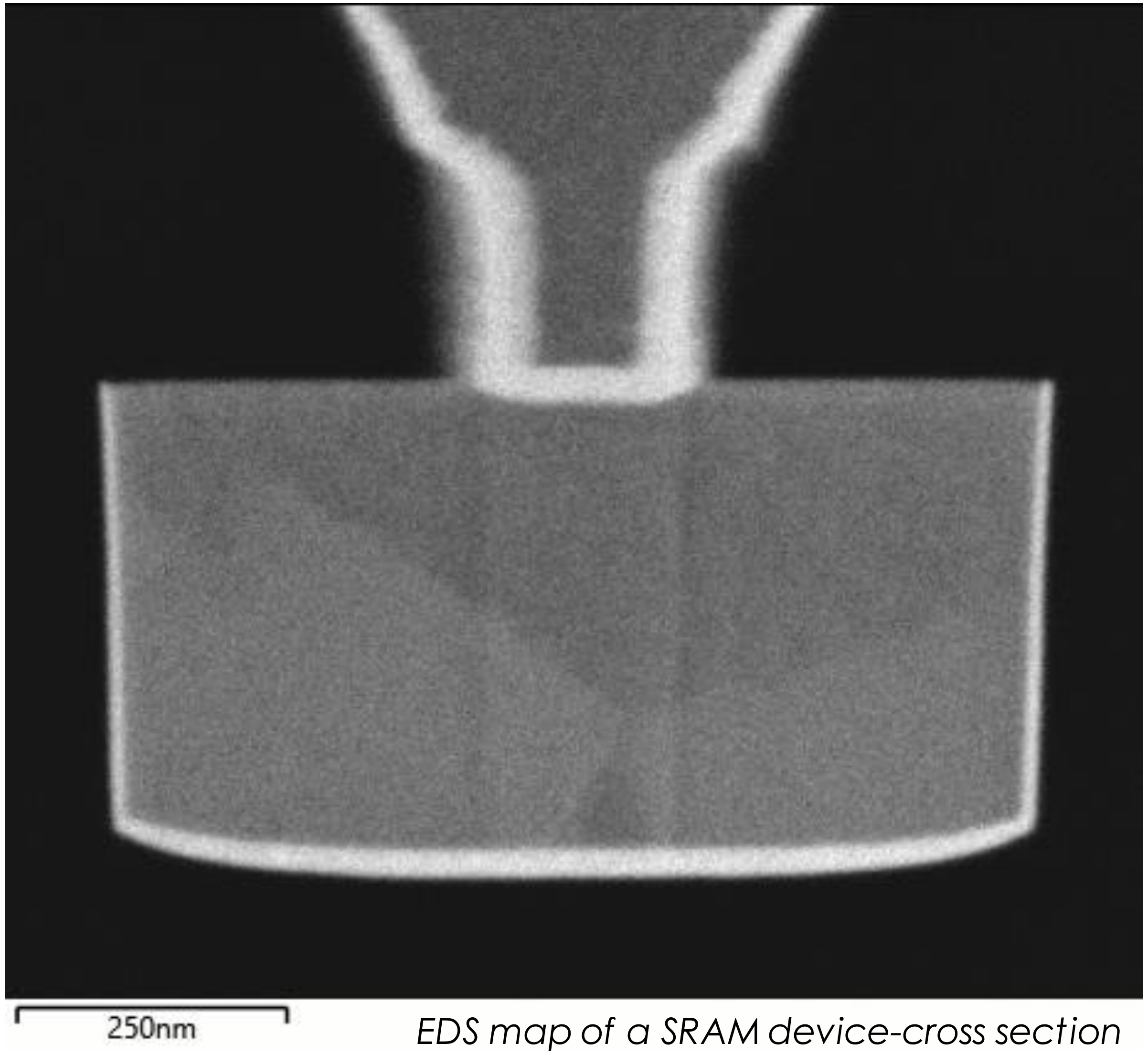
Problem:

- Chemically characterize structures in bulk devices
- Structures range in size from µm's to nm's
- Imaging ~5 nm scale or surface features in the SEM requires low kV and short working distance

Solution:

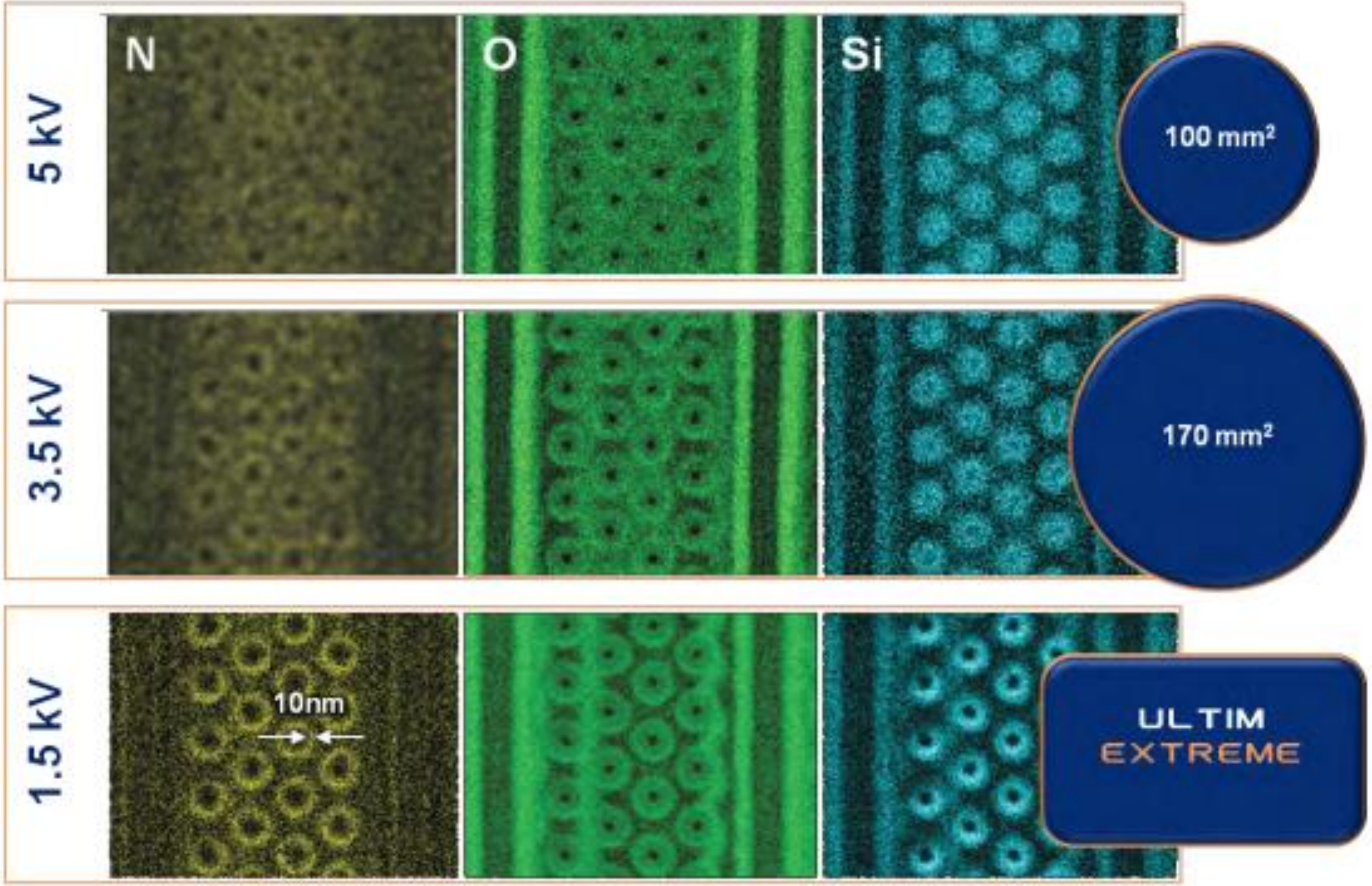
- Working at lower kV improves spatial resolution
- Ultim Extreme large area SDD:
  - Designed for low kV operation
  - Optimized geometry for short working distance
  - Windowless construction
  - Maximum X-ray collection efficiency
- TruMap processing ensures that you see the real distribution of elements

Many 'traditional' TEM analyses can be done on SEM

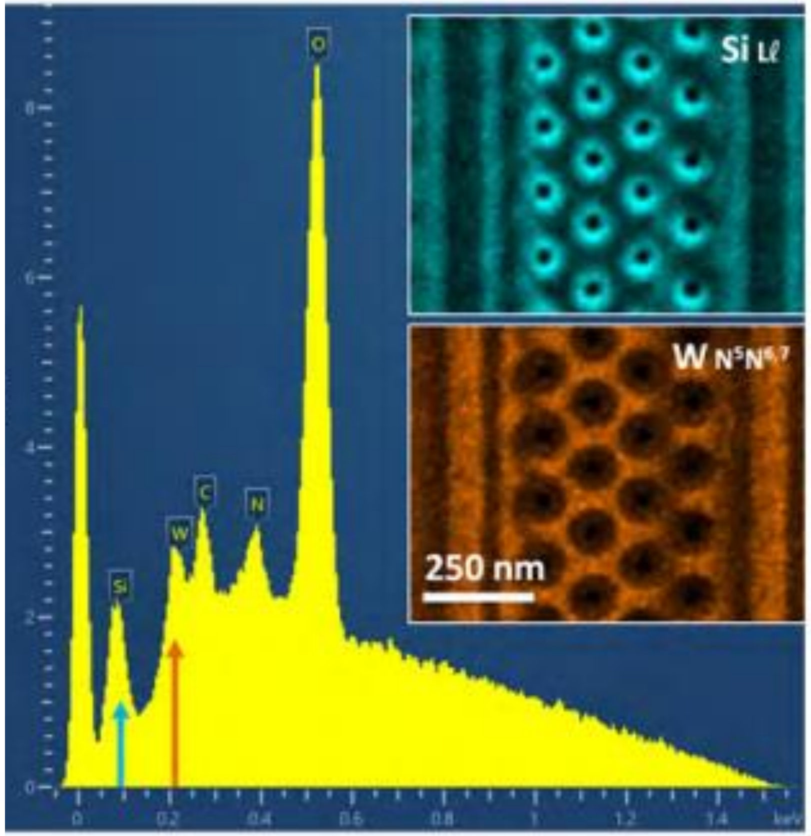


EDS map of a SRAM device-cross section

High Spatial Resolution Analysis: Flash device mapping on SEM at low kV

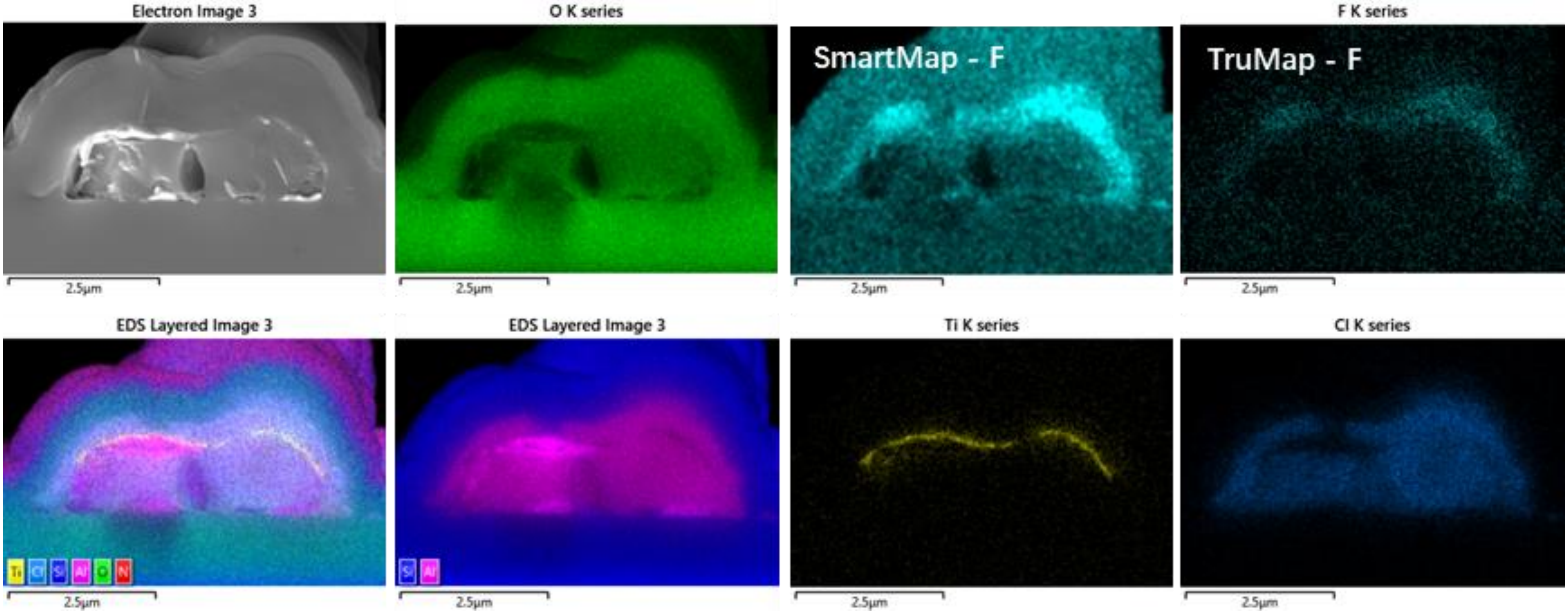


EDS maps of a NAND flash memory device



- Low kV EDS mapping with Ultim Extreme, AZtecTruMap

High Spatial Resolution Analysis: Power Device Failure



Electron Image 3

O K series

SmartMap - F

F K series

TruMap - F

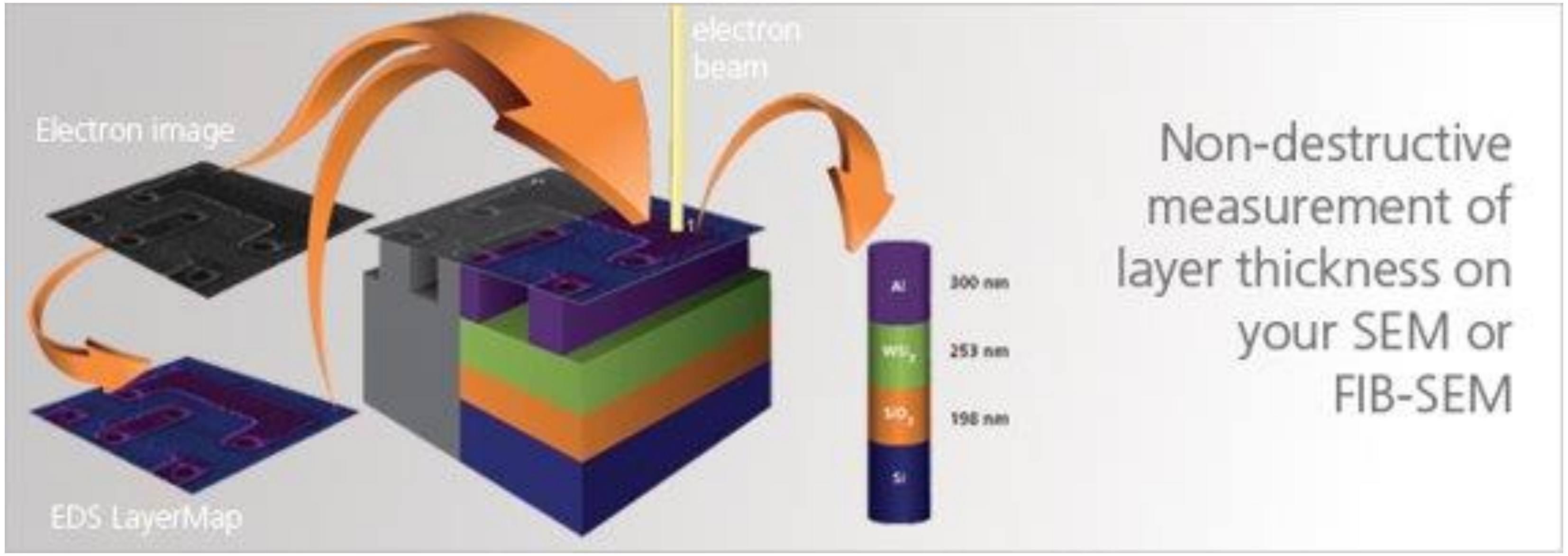
EDS Layered Image 3

EDS Layered Image 3

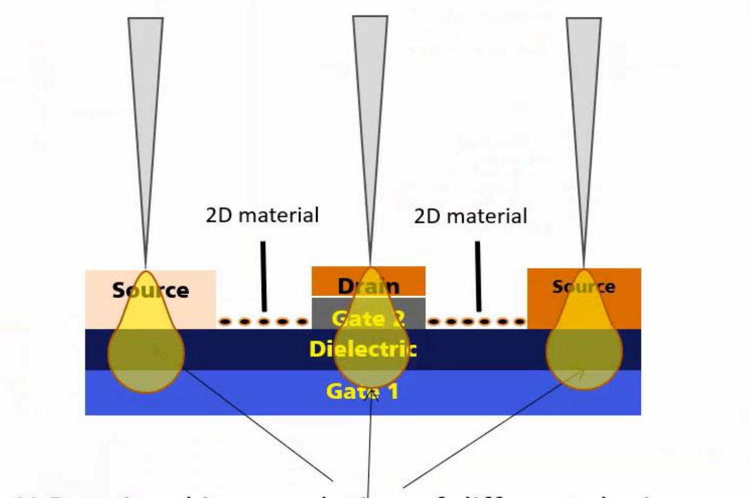
Ti K series

Cl K series

EDS Layer Thickness Measurement
AZtec LayerProbe Technique Comparison



Non-destructive measurement of layer thickness on your SEM or FIB-SEM

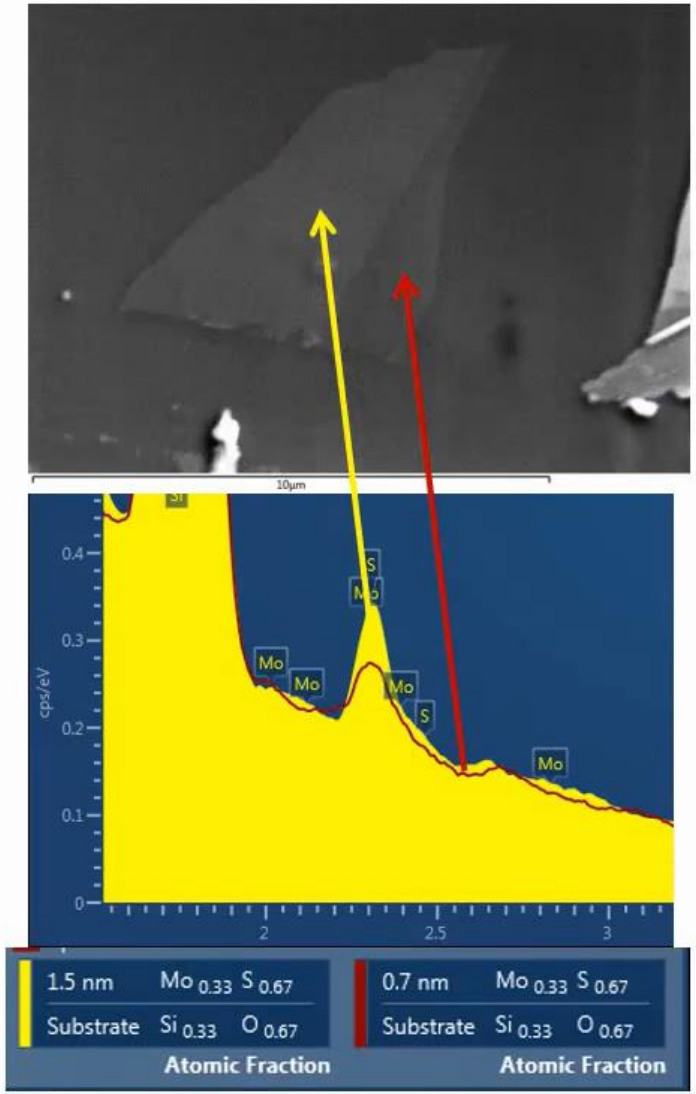


X-Ray signal is convolution of different device regions

	LayerProbe	Ellipsometry	XRF	FIB/TEM
Non-destructive	Yes	Yes	Yes	No
High spatial resolution	Yes	No (>>1 micron)	No (>1 micron)	Yes
Rapid analysis	Yes (5s)	Yes	Yes	No (hours)
Cost	Relatively inexpensive	Expensive	Expensive	Very expensive

- Process control
- Deposition and etching
- PECVD, ICPCVD, ALD
- Uniformity and compositional measurements

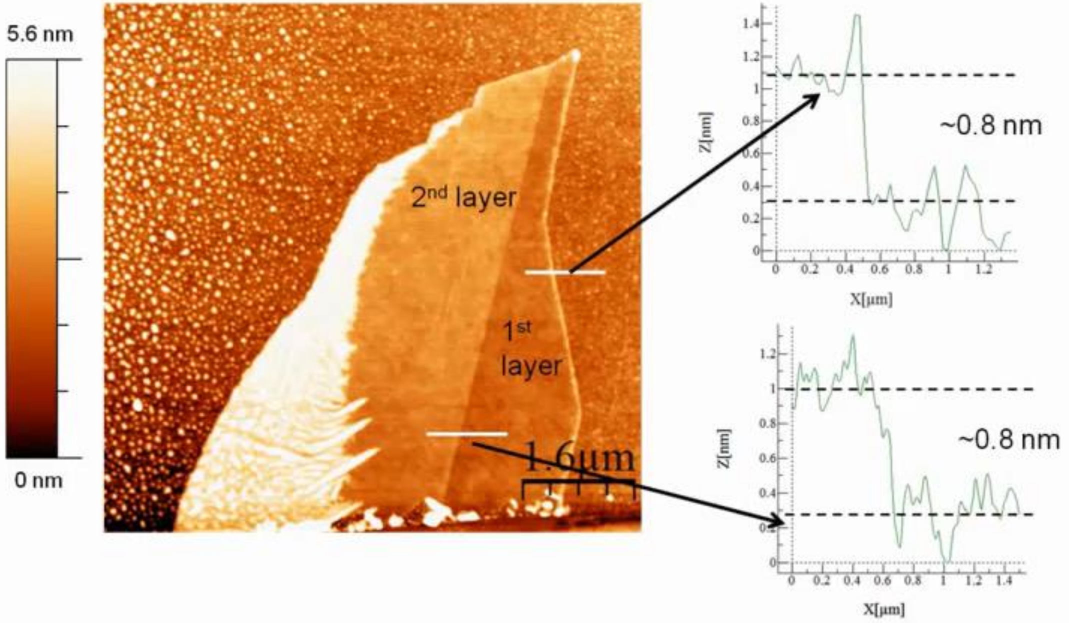
EDS Layer Thickness Measurement: Correlation to AFM



1.5 nm $Mo_{0.33}$ $S_{0.67}$
Substrate $Si_{0.33}$ $O_{0.67}$
Atomic Fraction

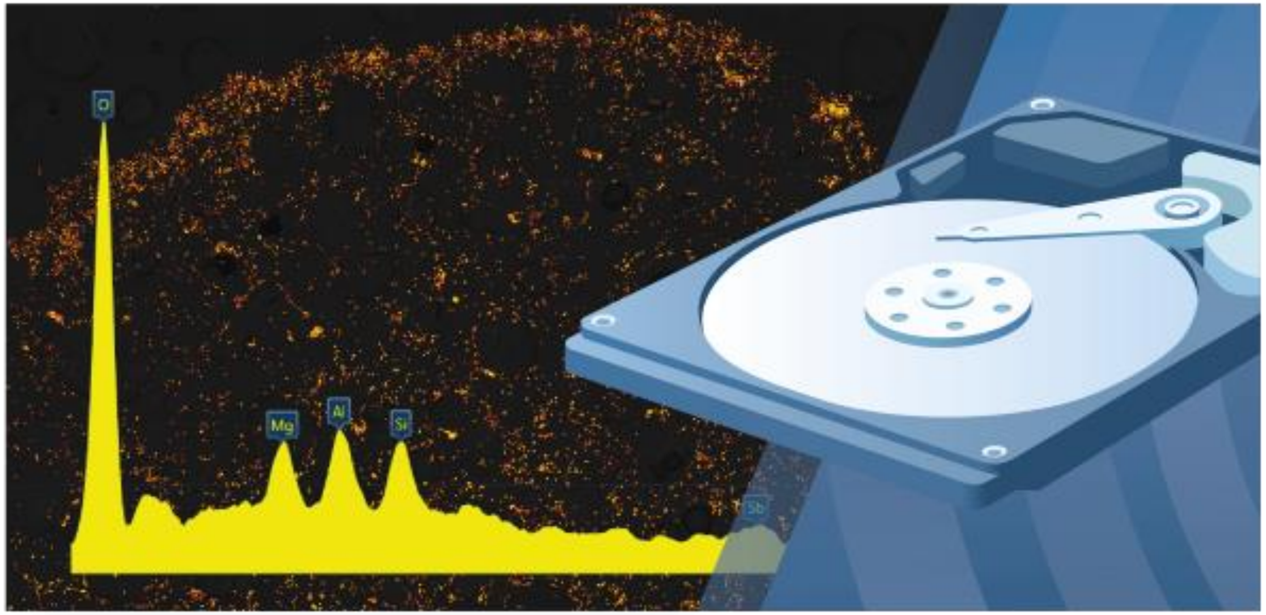
0.7 nm $Mo_{0.33}$ $S_{0.67}$
Substrate $Si_{0.33}$ $O_{0.67}$
Atomic Fraction

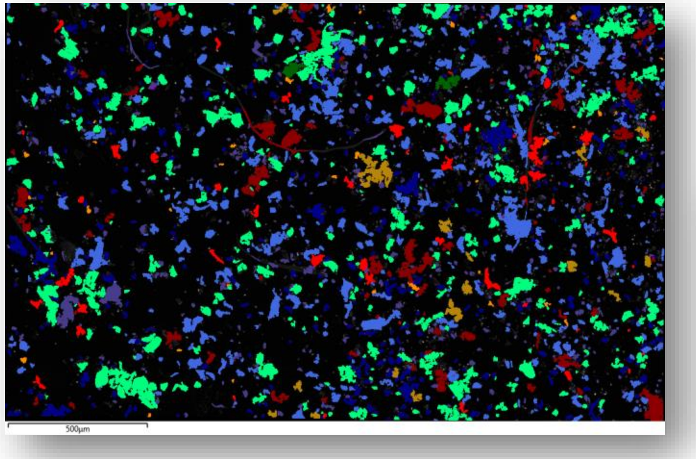
Correlation of LayerProbe result with Oxford Instruments Asylum Research AFM

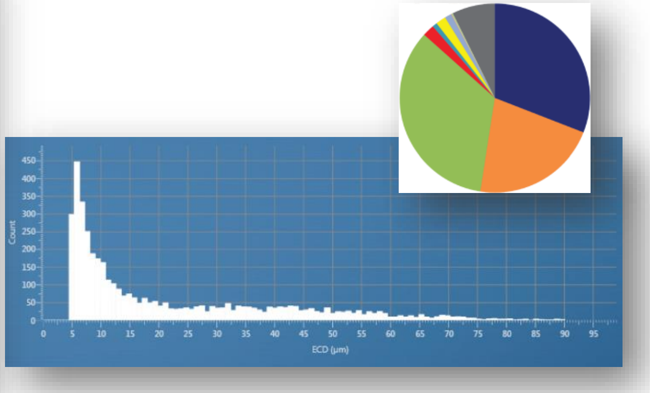


Clear distinction between one and two layers of MoS_2 at 5kV

Automated Particle Analysis
Identification and classification of contaminants







Problem:

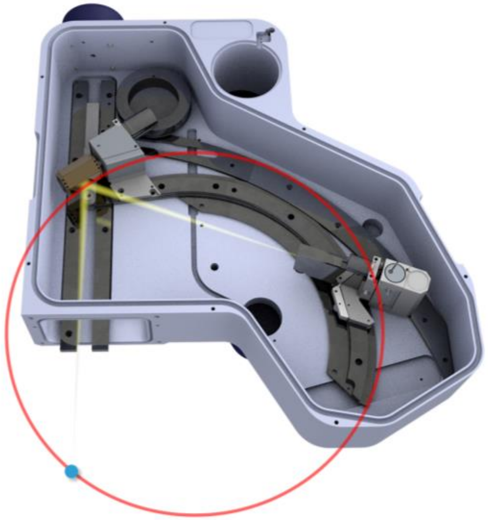
- Sample cleanliness is a quality issue
- Nanometer size particles can result in product failure
  - e.g., particles in a hard disk drive can result in disk failure or a head crash
- Process Control and Particle Analysis
- Wafer analyses

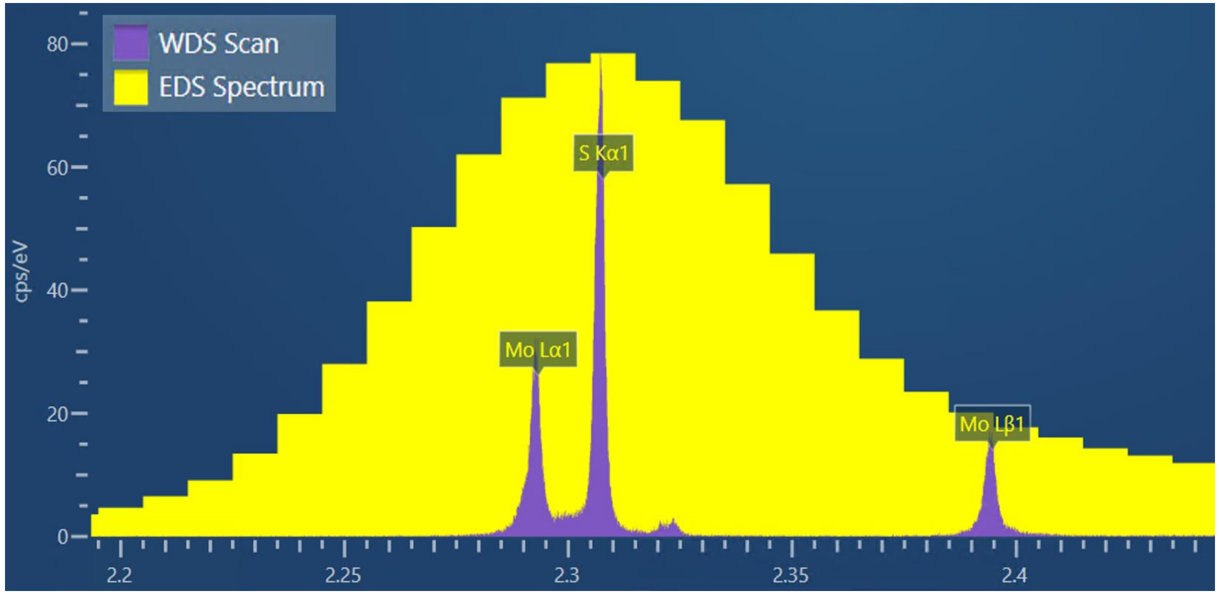
Solution:

- World leading particle analysis solution
- Quickly and accurately identify, analyze and classify contaminants
- Location, morphology & composition of particles

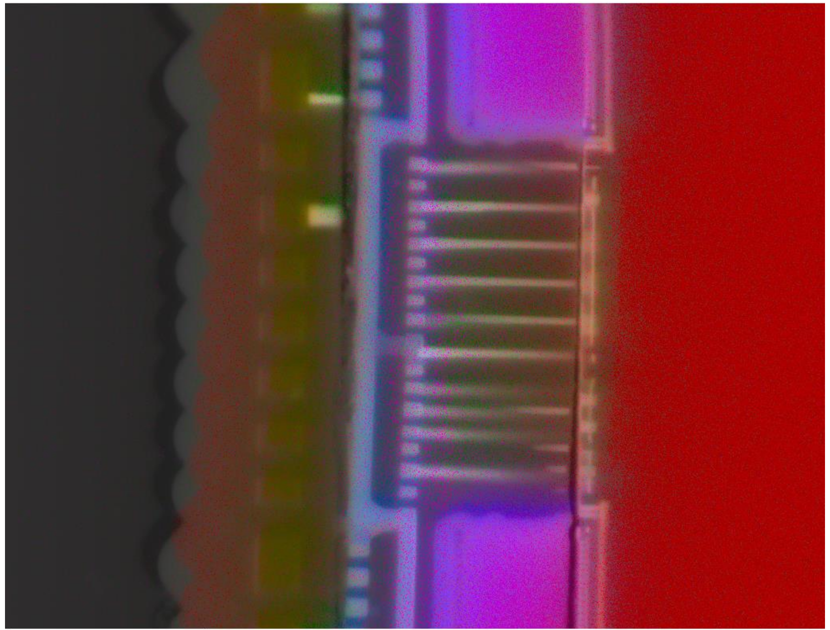
EDS: Some Limitations

- Peak overlaps and minimum detection limit
- …different method: Wavelength Dispersive Spectrometry (WDS)
  - **Spectral resolution** of WDS is ~10x better than EDS
    - WDS provides better separation of peak overlaps
  - **Detection limit** of WDS is ~10x better than EDS
    - WDS can accurately detect and quantitatively measure trace elements (i.e., <1000 ppm)

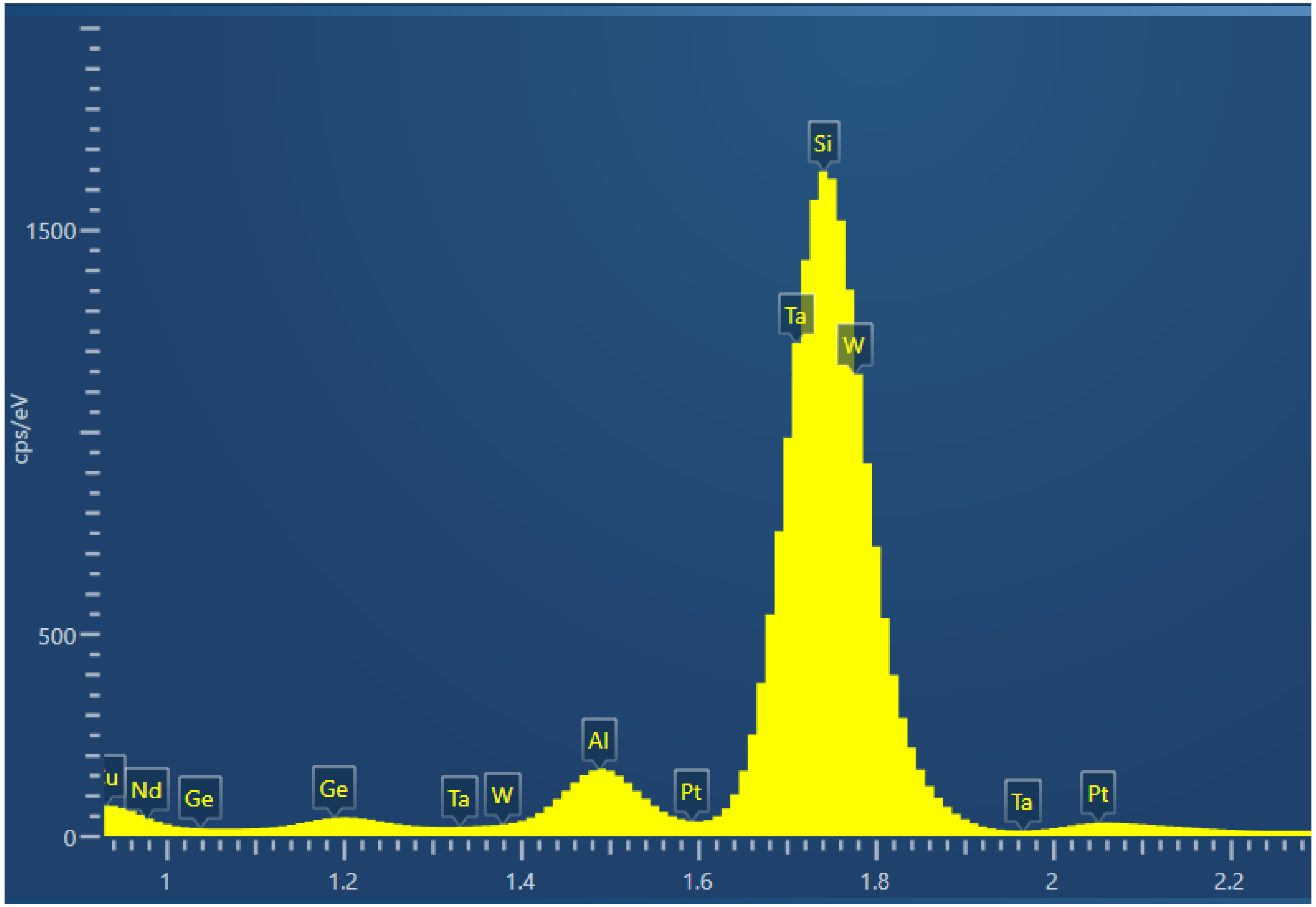




EDS-WDS Maps: Spectral Resolution

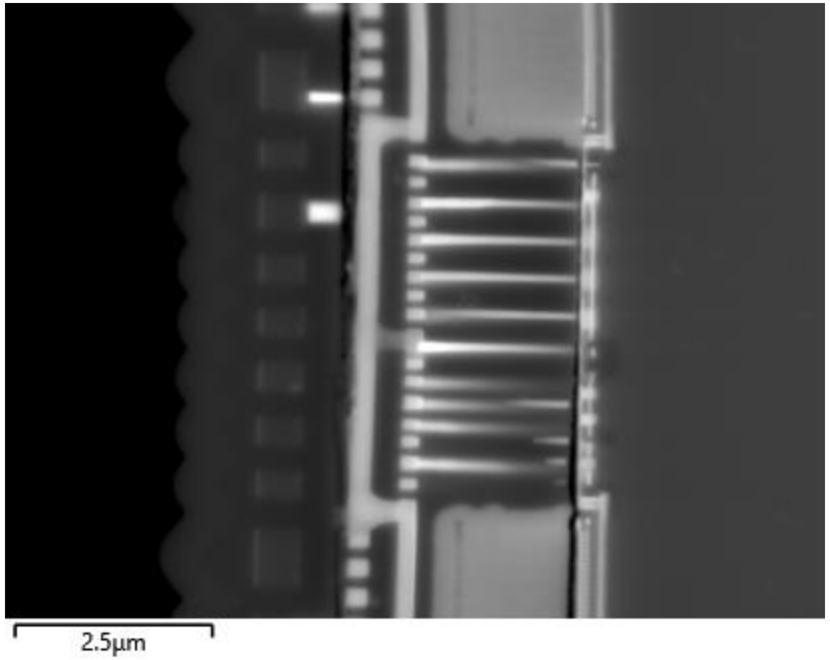


WDS and EDS –Overlay

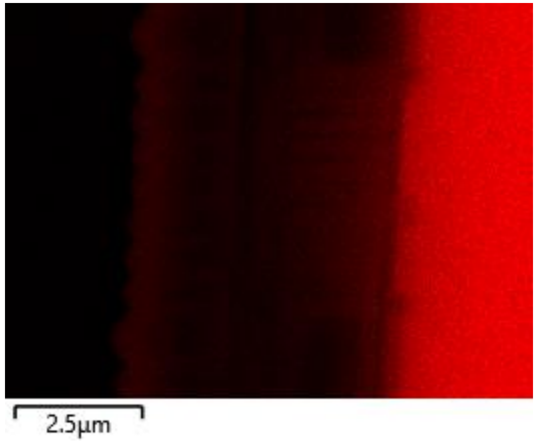


Overlap of Lines Si, Ta and W

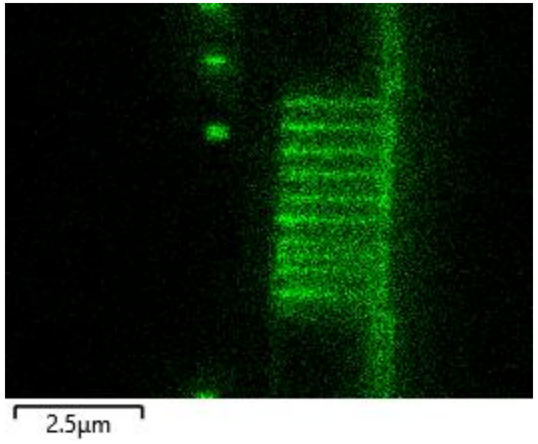
EDS-WDS Maps: Spectral Resolution



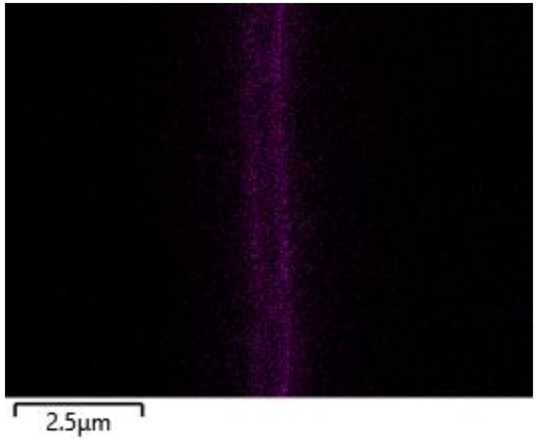
Si WDS Map



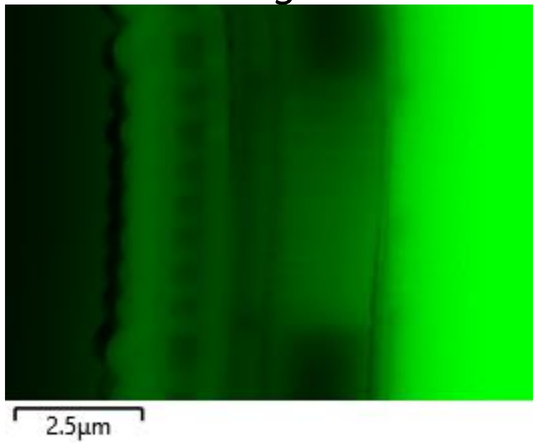
W WDS Map



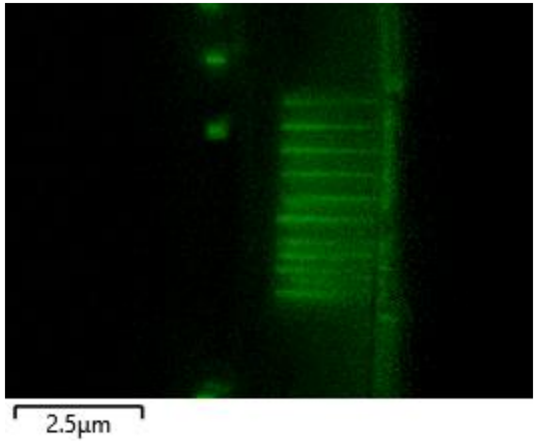
Ta WDS Map



Si EDS Maps– Window integral



W EDS Maps–TruMap



Ta EDS Maps–TruMap

WDS Measurement of Doping in Semiconductors

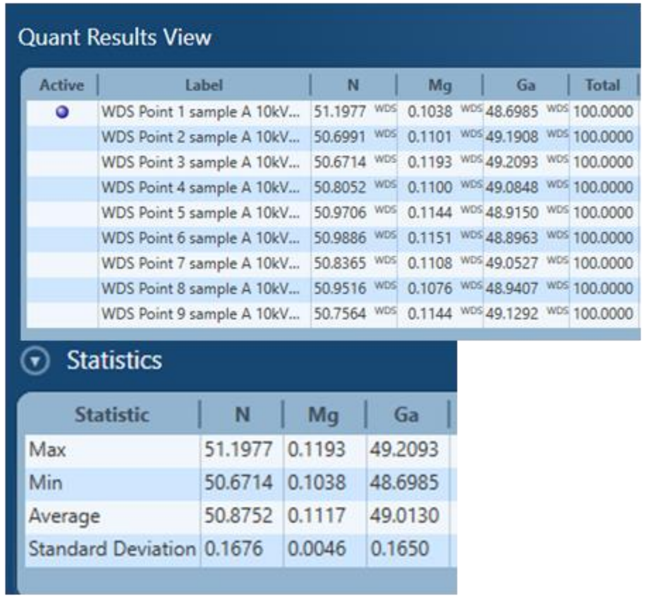
Problem:

- Mg is present at a trace level (below 1.0 wt. %) which would be difficult to accurately measure with EDS

Solution:

- WDS measurement ideal due to superior detection limit

Atomic %



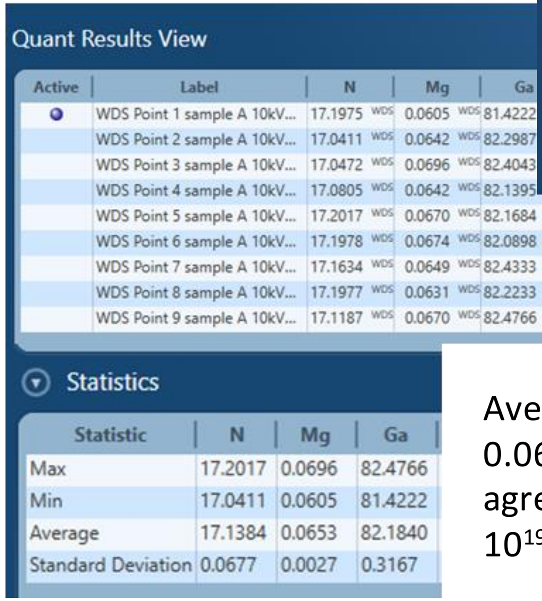
Quant Results View

Active	Label	N	Mg	Ga	Total
●	WDS Point 1 sample A 10kV...	51.1977 WDS	0.1038 WDS	48.6985 WDS	100.0000
	WDS Point 2 sample A 10kV...	50.6991 WDS	0.1101 WDS	49.1908 WDS	100.0000
	WDS Point 3 sample A 10kV...	50.6714 WDS	0.1193 WDS	49.2093 WDS	100.0000
	WDS Point 4 sample A 10kV...	50.8052 WDS	0.1100 WDS	49.0848 WDS	100.0000
	WDS Point 5 sample A 10kV...	50.9706 WDS	0.1144 WDS	48.9150 WDS	100.0000
	WDS Point 6 sample A 10kV...	50.9886 WDS	0.1151 WDS	48.8963 WDS	100.0000
	WDS Point 7 sample A 10kV...	50.8365 WDS	0.1108 WDS	49.0527 WDS	100.0000
	WDS Point 8 sample A 10kV...	50.9516 WDS	0.1076 WDS	48.9407 WDS	100.0000
	WDS Point 9 sample A 10kV...	50.7564 WDS	0.1144 WDS	49.1292 WDS	100.0000

Statistics

Statistic	N	Mg	Ga
Max	51.1977	0.1193	49.2093
Min	50.6714	0.1038	48.6985
Average	50.8752	0.1117	49.0130
Standard Deviation	0.1676	0.0046	0.1650

Weight %

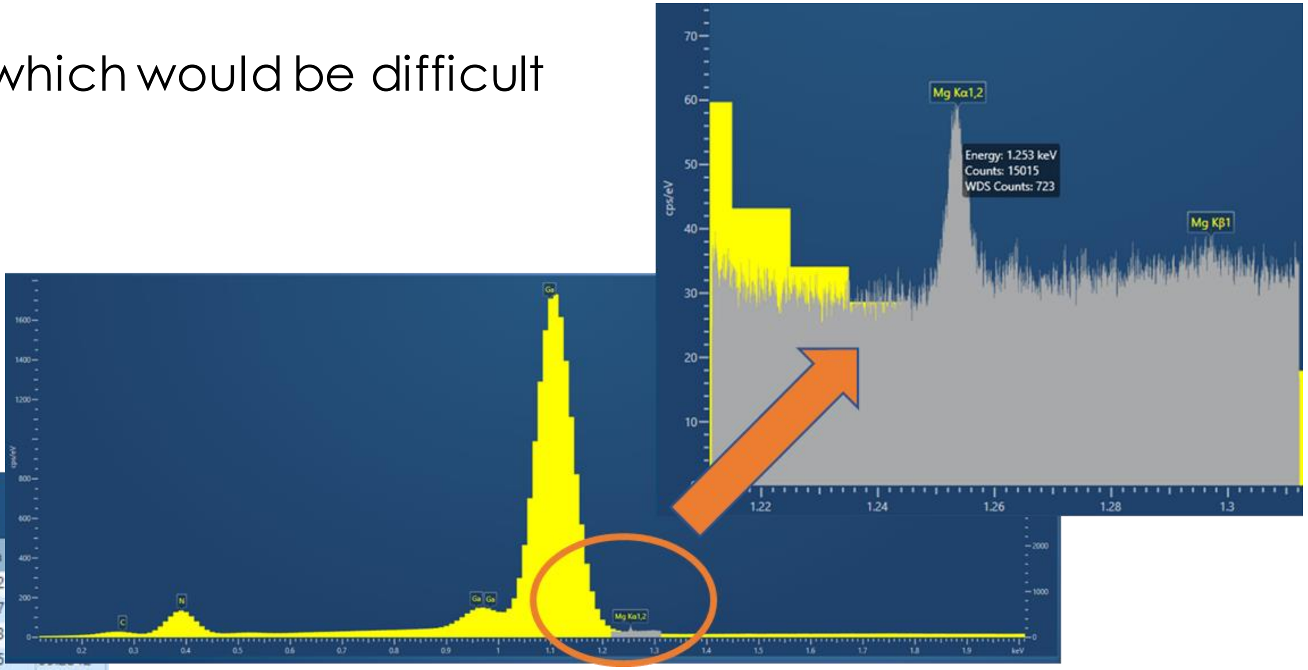


Quant Results View

Active	Label	N	Mg	Ga	
●	WDS Point 1 sample A 10kV...	17.1975 WDS	0.0605 WDS	81.4222	
	WDS Point 2 sample A 10kV...	17.0411 WDS	0.0642 WDS	82.2987	
	WDS Point 3 sample A 10kV...	17.0472 WDS	0.0696 WDS	82.4043	
	WDS Point 4 sample A 10kV...	17.0805 WDS	0.0642 WDS	82.1395	
	WDS Point 5 sample A 10kV...	17.2017 WDS	0.0670 WDS	82.1684 WDS	99.4371
	WDS Point 6 sample A 10kV...	17.1978 WDS	0.0674 WDS	82.0898 WDS	99.3550
	WDS Point 7 sample A 10kV...	17.1634 WDS	0.0649 WDS	82.4333 WDS	99.6616
	WDS Point 8 sample A 10kV...	17.1977 WDS	0.0631 WDS	82.2233 WDS	99.4841
	WDS Point 9 sample A 10kV...	17.1187 WDS	0.0670 WDS	82.4766 WDS	99.6623

Statistics

Statistic	N	Mg	Ga
Max	17.2017	0.0696	82.4766
Min	17.0411	0.0605	81.4222
Average	17.1384	0.0653	82.1840
Standard Deviation	0.0677	0.0027	0.3167



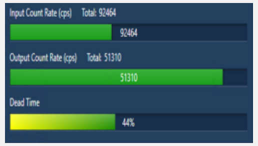
WDS scan of the Mg Kα peak using the TAP crystal in the Wave spectrometer

Average [Mg] measured in the sample corresponds to a mass percentage of 0.0653%, equivalent to a concentration of 9.8 × 10^{19} cm^{-3} which shows good agreement with the SIMS values of 9.5 × 10^{19} cm^{-3} and EPMA values of 9.4 (±0.2) × 10^{19} cm^{-3}

Making Imaging Elementary

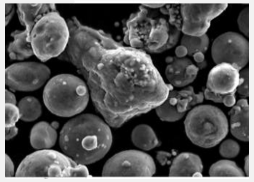
BEX- UNITY Detector

Traditional Imaging & Analysis

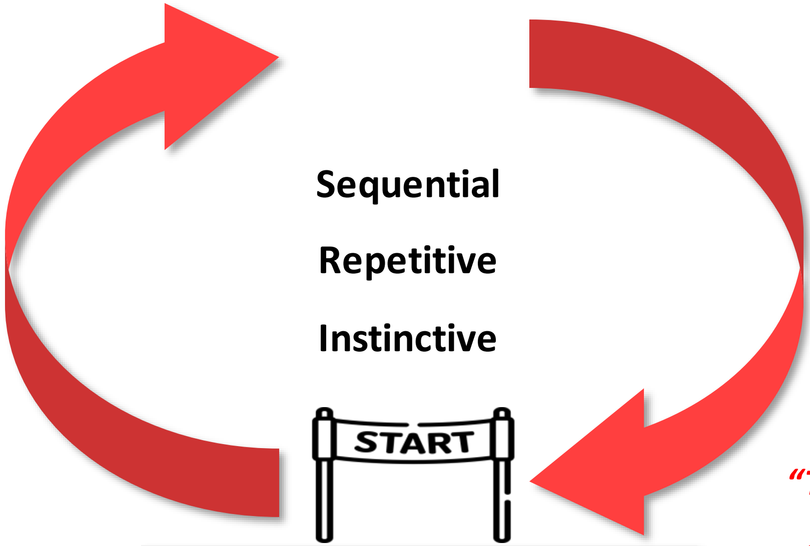

Input Count Rate (cps) Total: 92464
92464
Output Count Rate (cps) Total: 51310
51310
Dead Time
44%

Optimize SEM for EDS analysis

Acquire electron image



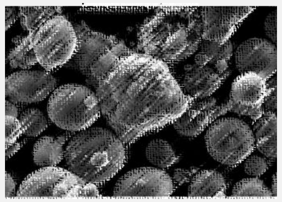
Acquire EDS data



Sequential

Repetitive

Instinctive



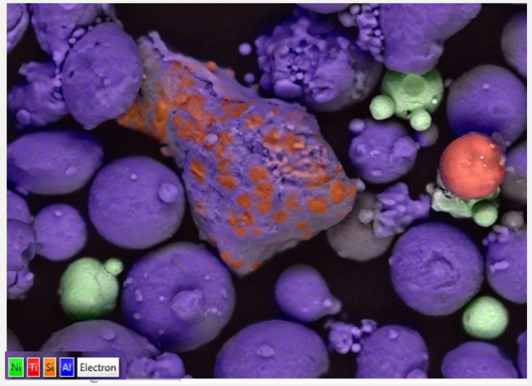
Instinctive Search for AoI

Confirm elements and AoI

Al Ti Ni

☹ ☺

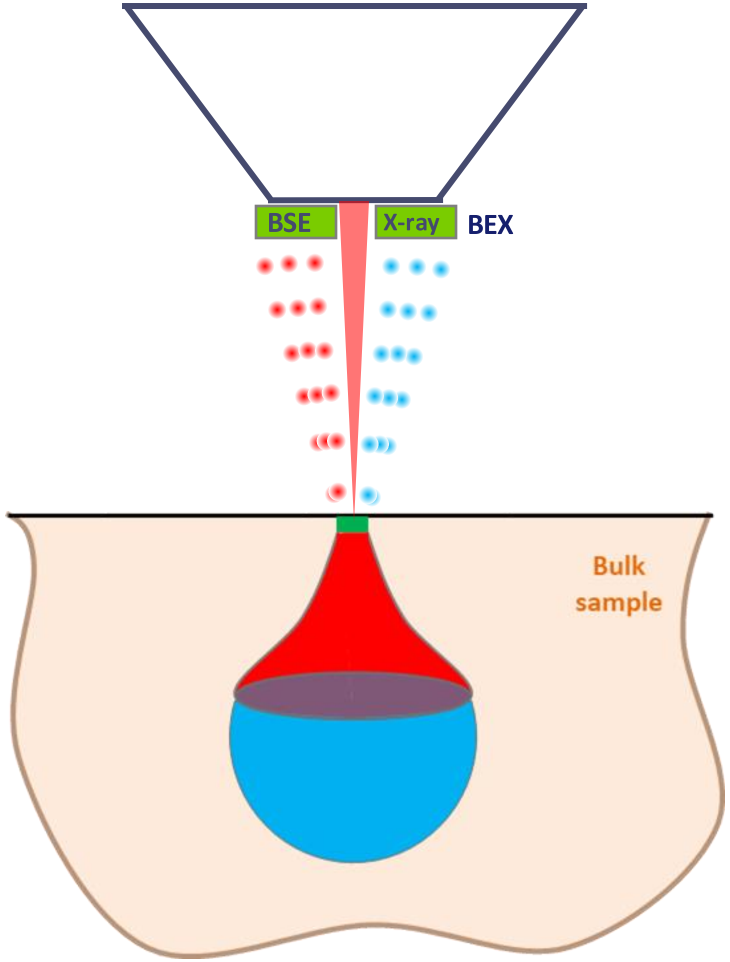
"The loop of suffering"

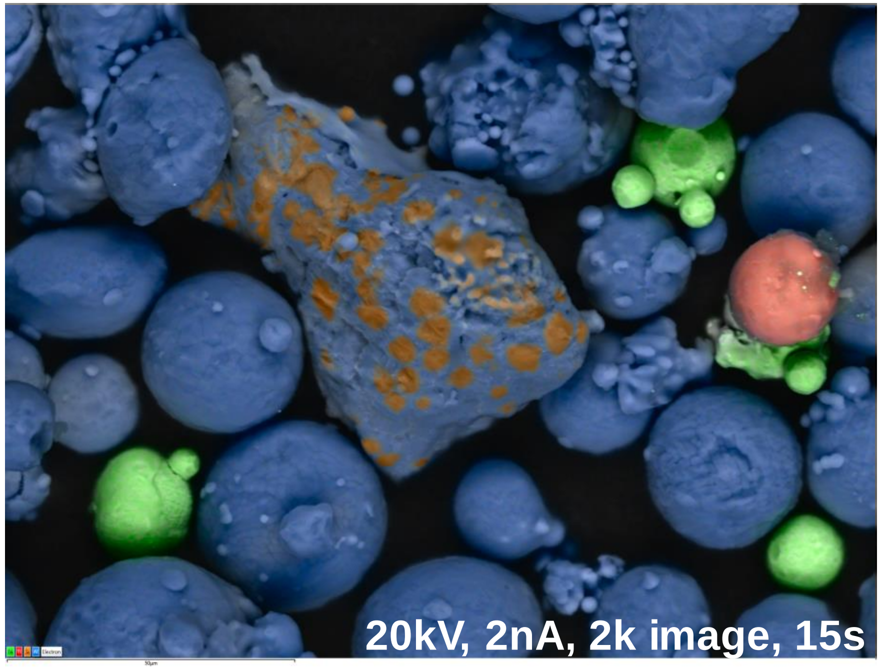


Acquire high quality EDS/EBSD/WDS results

BEX Imaging

Backscattered Electron and X-ray Detectors





SE Image
Greyscale
Topography contrast

BSE Image
Greyscale
Z-number contrast
Topography contrast

BEX Image
Colored by Element
Atomic contrast
Topography contrast

- Combines BSE and x-ray signals
- Characteristic x-rays for composition
- Acquired simultaneously
- **Works at the speed and conditions of a traditional imaging detector!**

Introducing Unity

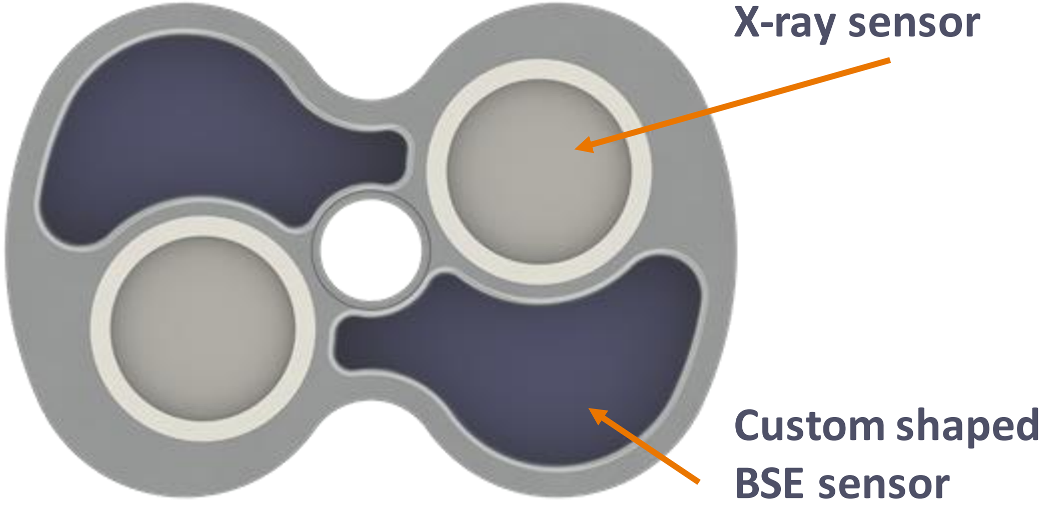
Unity BEX Detector

- Retractable below pole piece BSE and x-ray detector for simultaneous acquisition
- Optimized head thickness and central hole for daily use and flexible imaging

Unity Sensor Head

Optimized to collect maximum signal without obscuring EDS

- BSE sensors
  - Peltier cooled sensors for enhanced sensitivity
  - Two segments for topography modes
- X-ray sensors
  - Large solid angle for SDDs for high speed



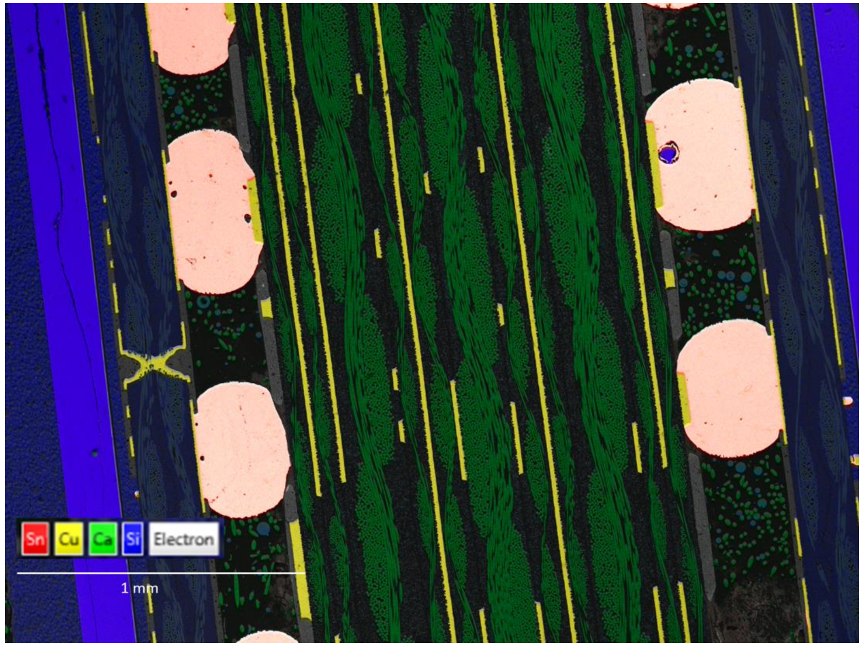
Unity BEX Imaging Advantages of BEX

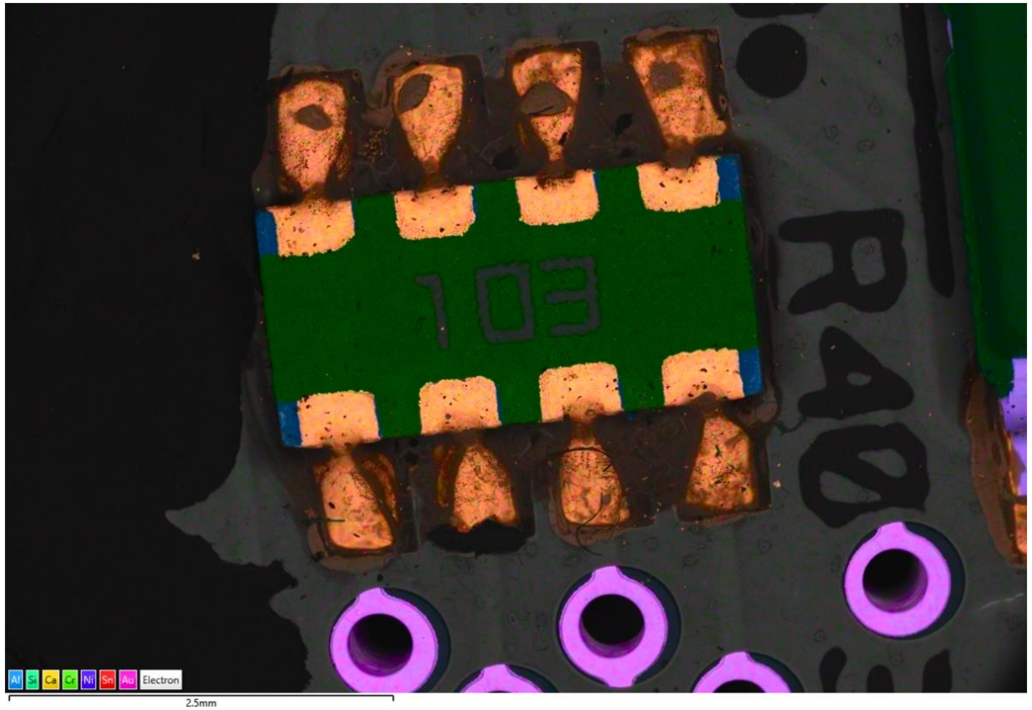
Optimized for normal imaging conditions **<20kV, 1nA**

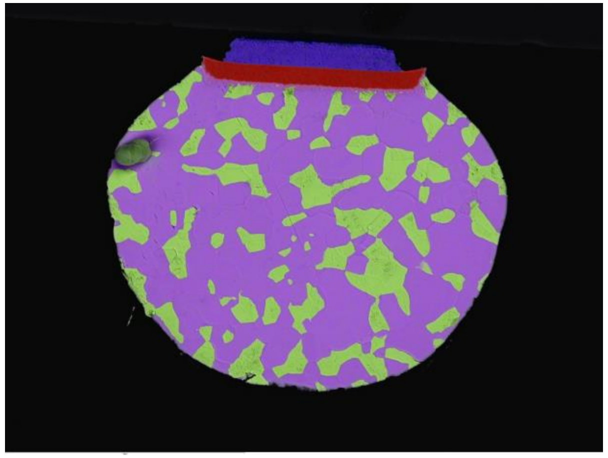
Flexible Working Distance >**6mm**

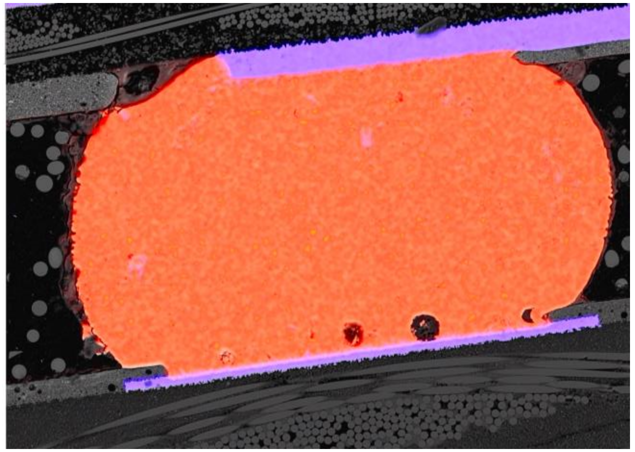
Large Field of View (compatible with Wide Field modes)

No shadowing unlike SEs and conventional EDS





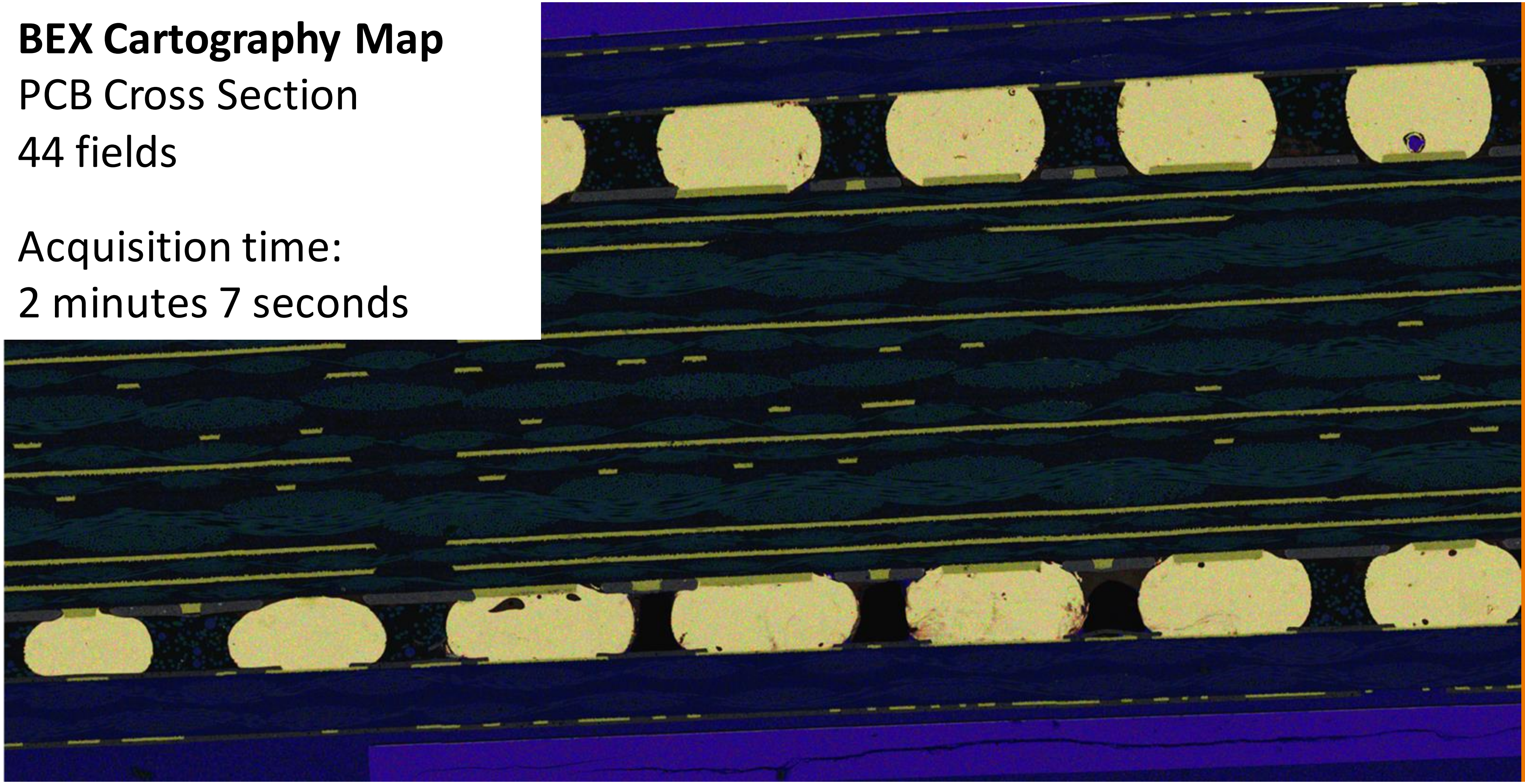




Unity BEX Imaging Rapid PCB-level Inspection

BEX Cartography Map
PCB Cross Section
44 fields

Acquisition time:
2 minutes 7 seconds



Navigation with Chemistry

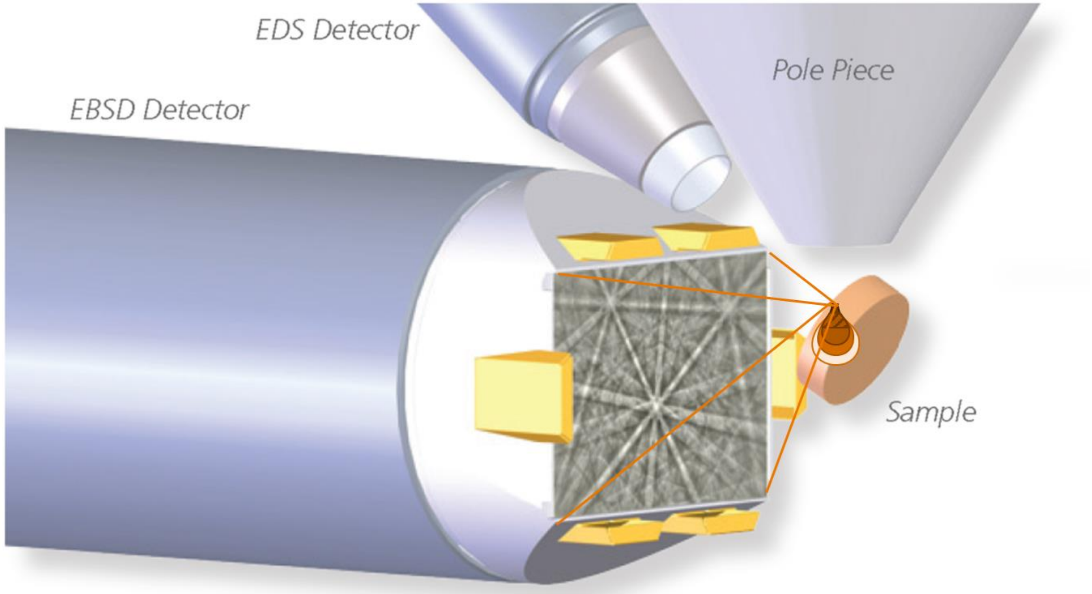
Fast High-Definition Imaging

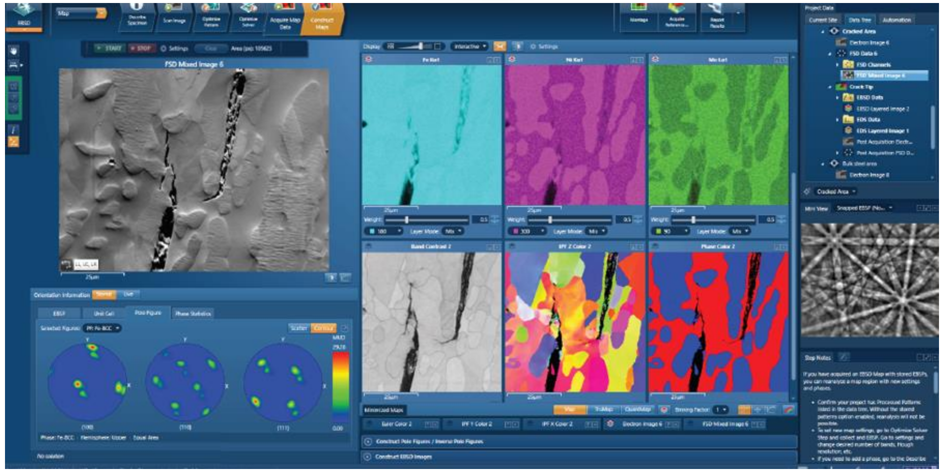
Whole Sample Cartography

Microstructure Characterization

With Electron Backscattered Diffraction (EBSD)

Electron BackScatter Diffraction (EBSD)





EBSD provides

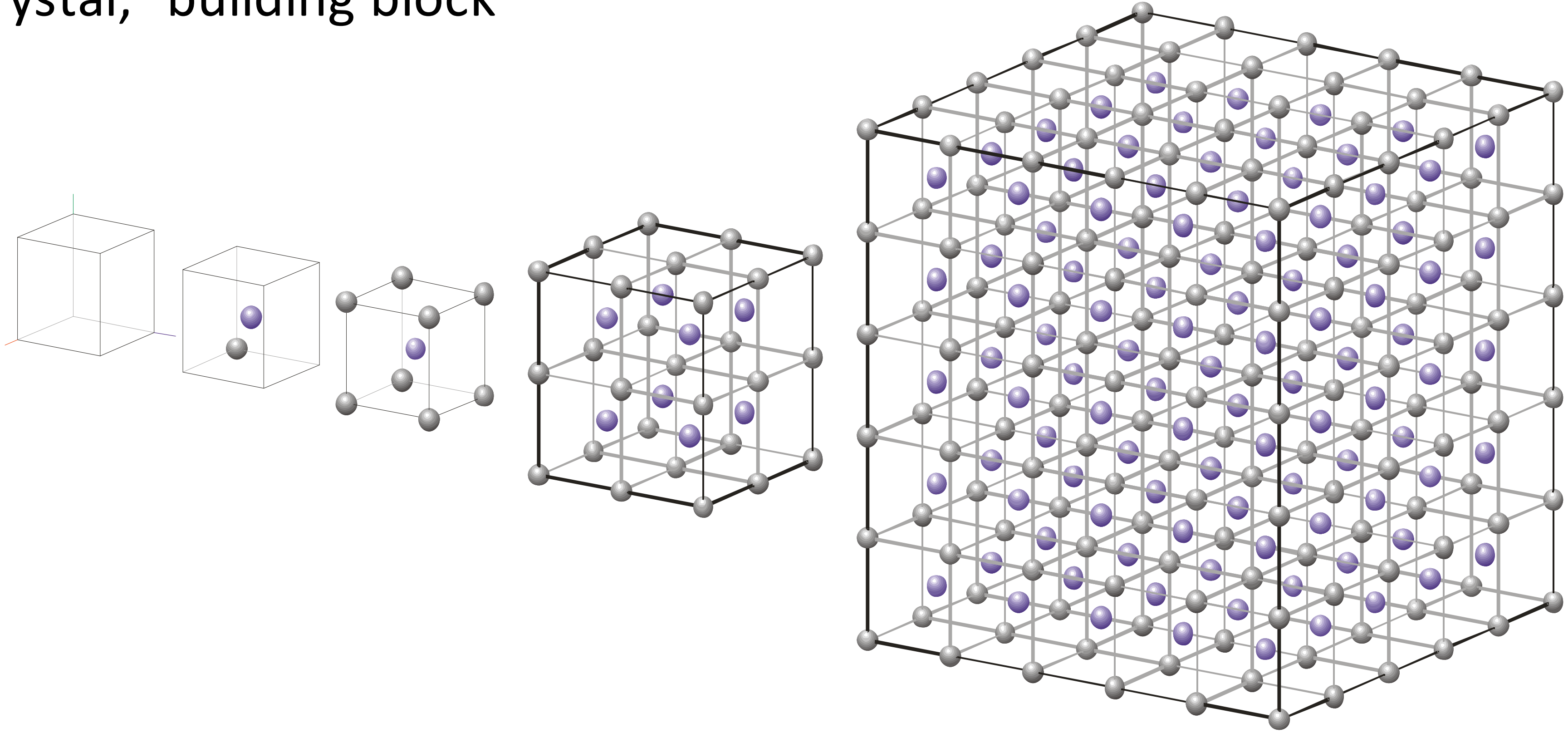
- EBSD is a SEM based technique, used to characterize crystalline materials with sub-micron resolution
- Crystal orientations and all associated measurements (texture, grain size, strain, boundary characterization etc.)
- Discrimination between phases
- Identification of unknown phases when integrated with EDS

SYMMETRY S3

- Custom design CMOS sensor and fiber optics giving high sensitivity
- Fast mapping possible on real-life samples
- Detector elevation control
- No compromise – one detector for all applications

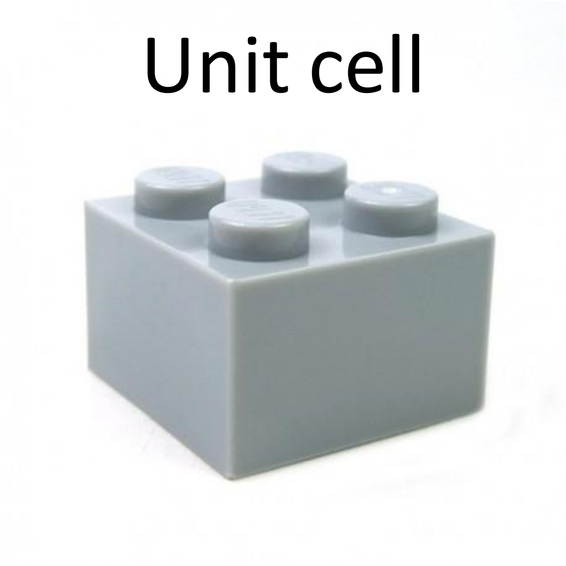
Crystallography Basics

- Crystal: Posesses a regular arrangement of atoms
- Unit cell: Essentially, smallest unique unit of the crystal; ”building block”

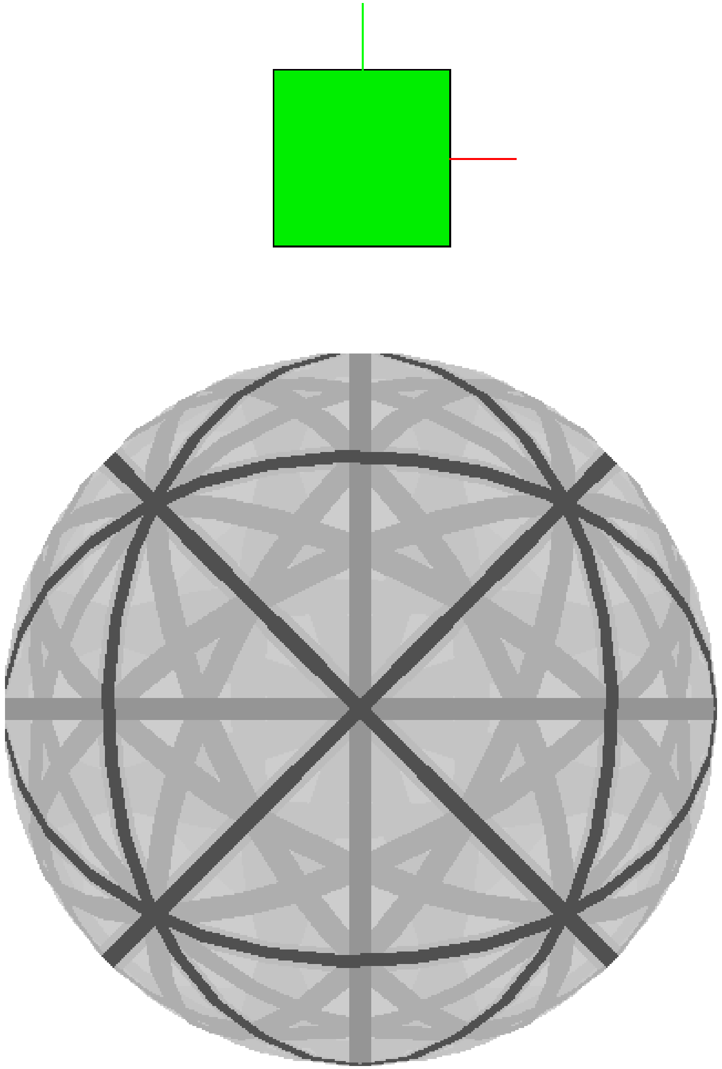


Crystallography Basics

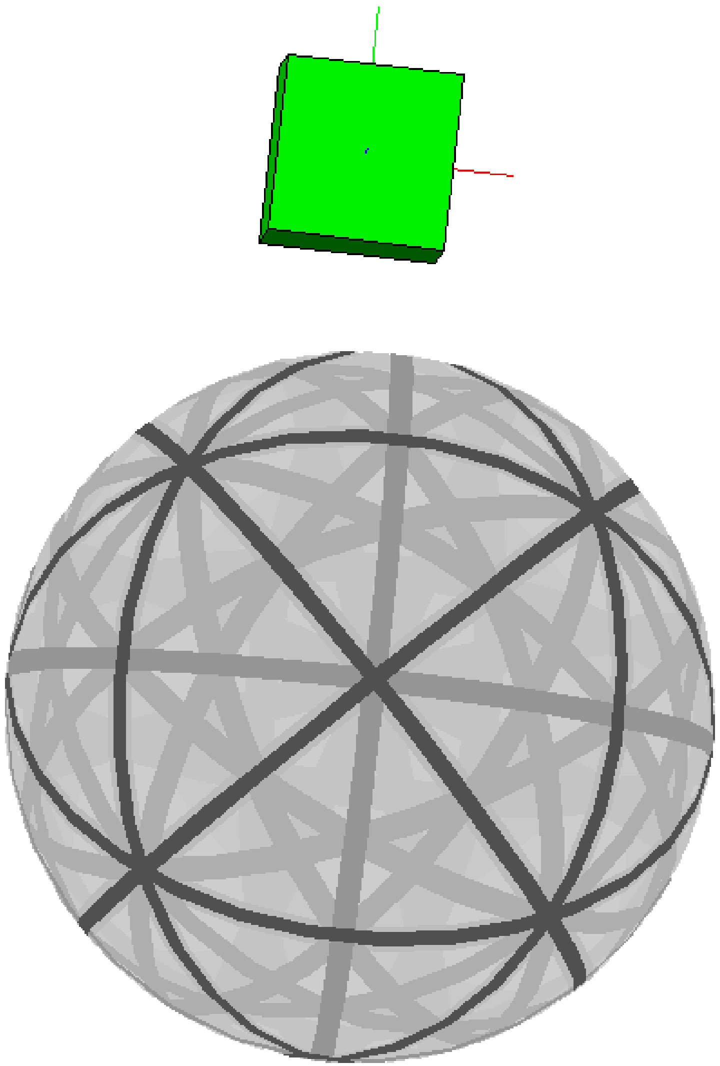
Unit cell



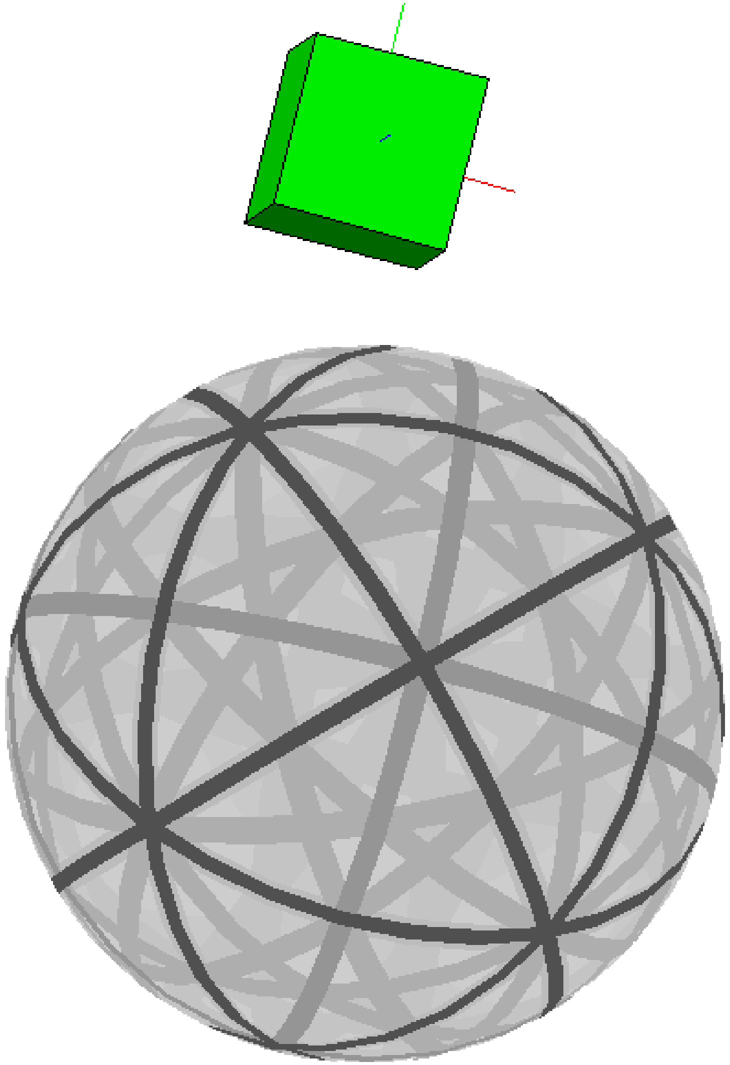
Pattern Change with Orientation



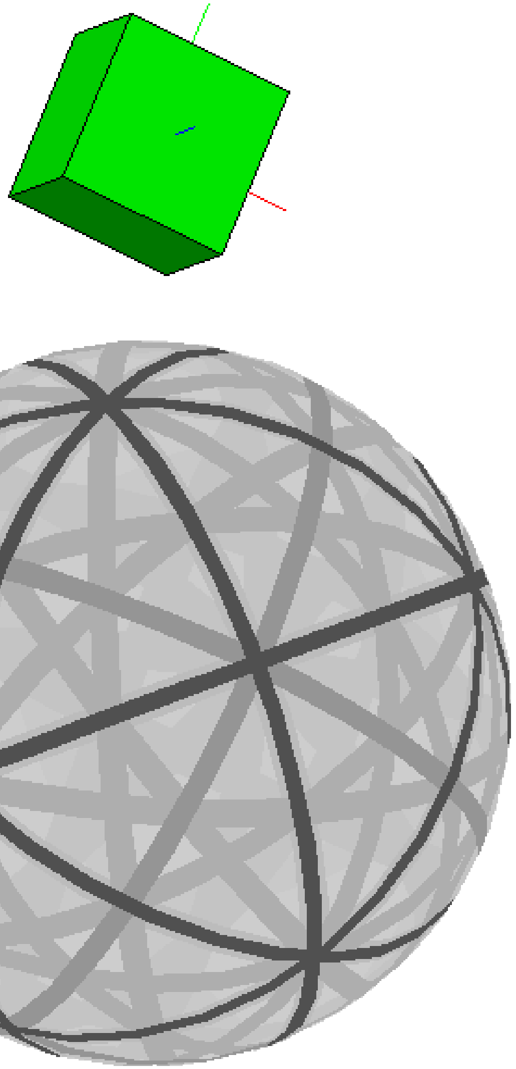
Pattern Change with Orientation



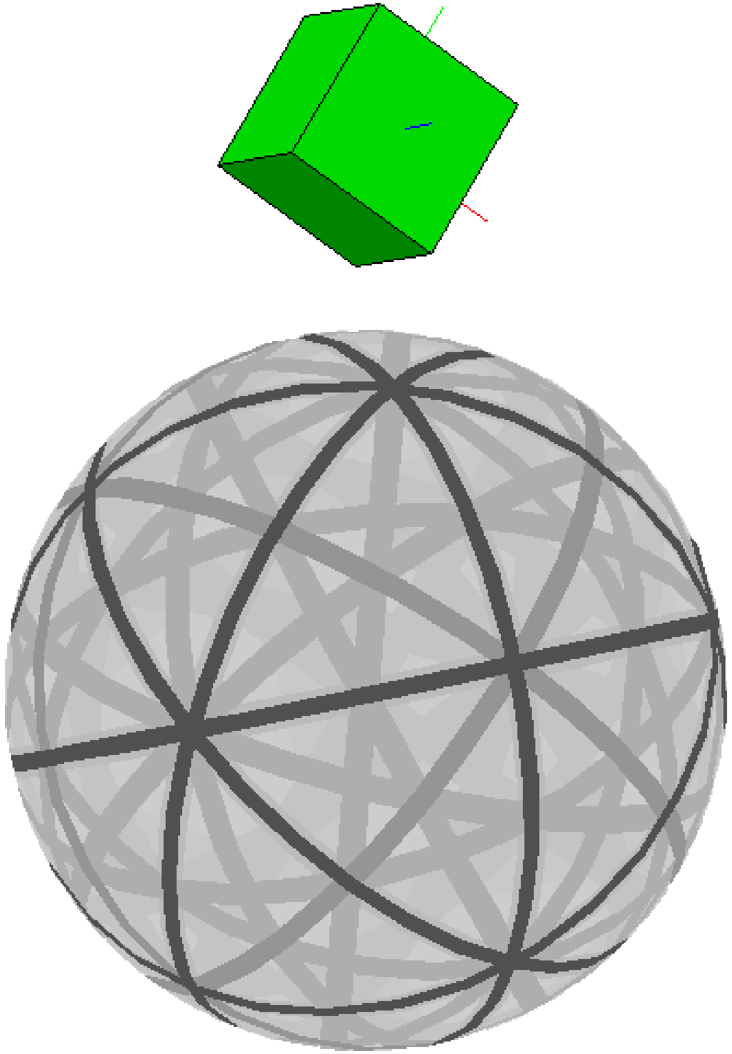
Pattern Change with Orientation



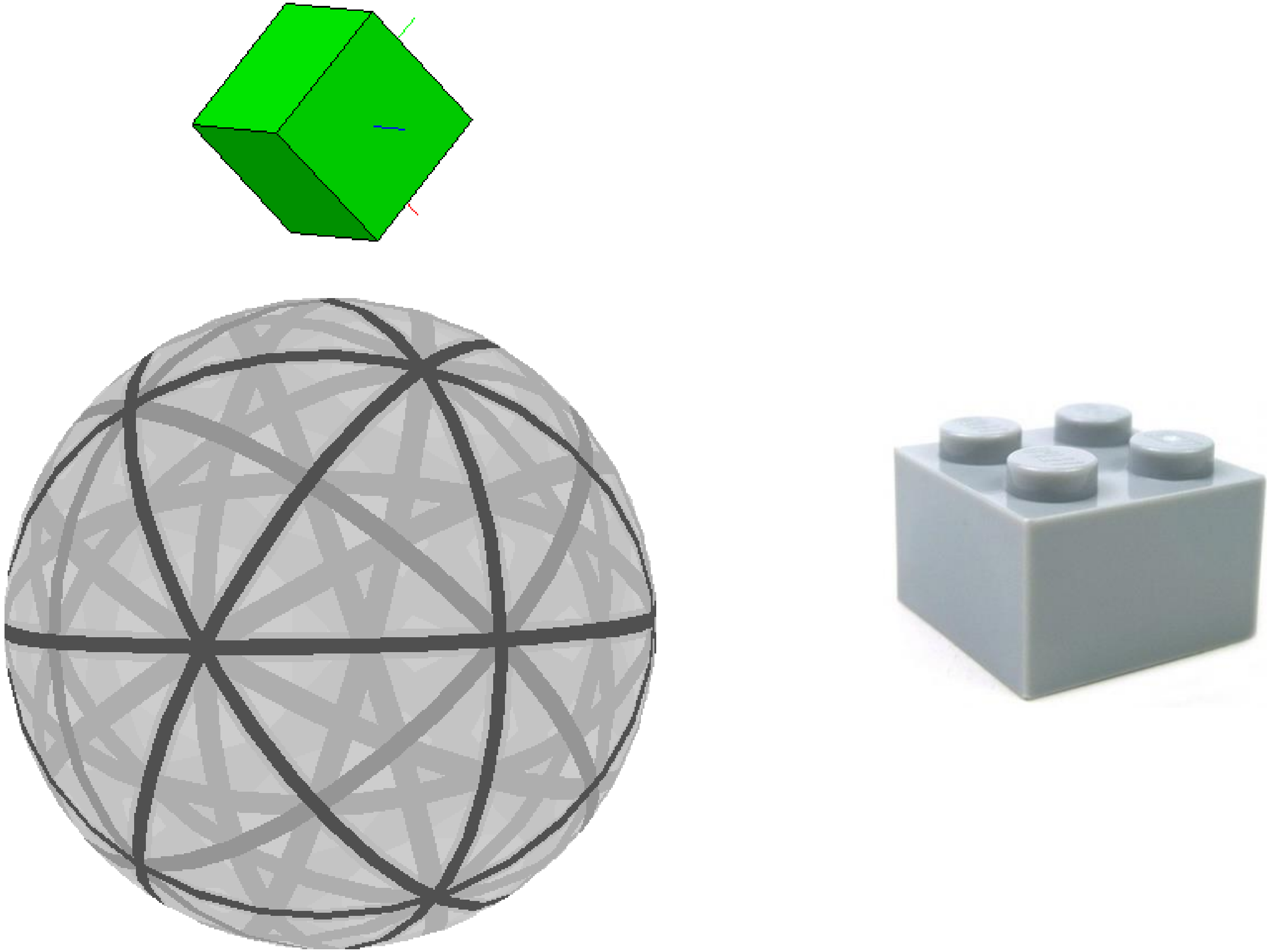
Pattern Change with Orientation



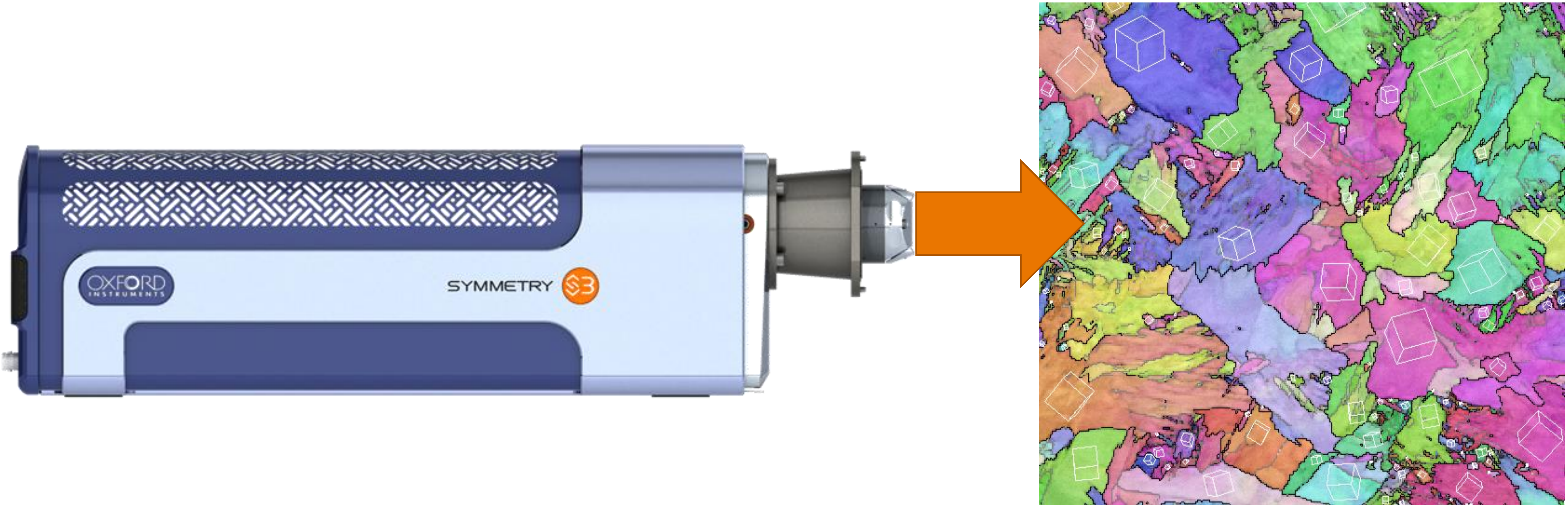
Pattern Change with Orientation



Pattern Change with Orientation

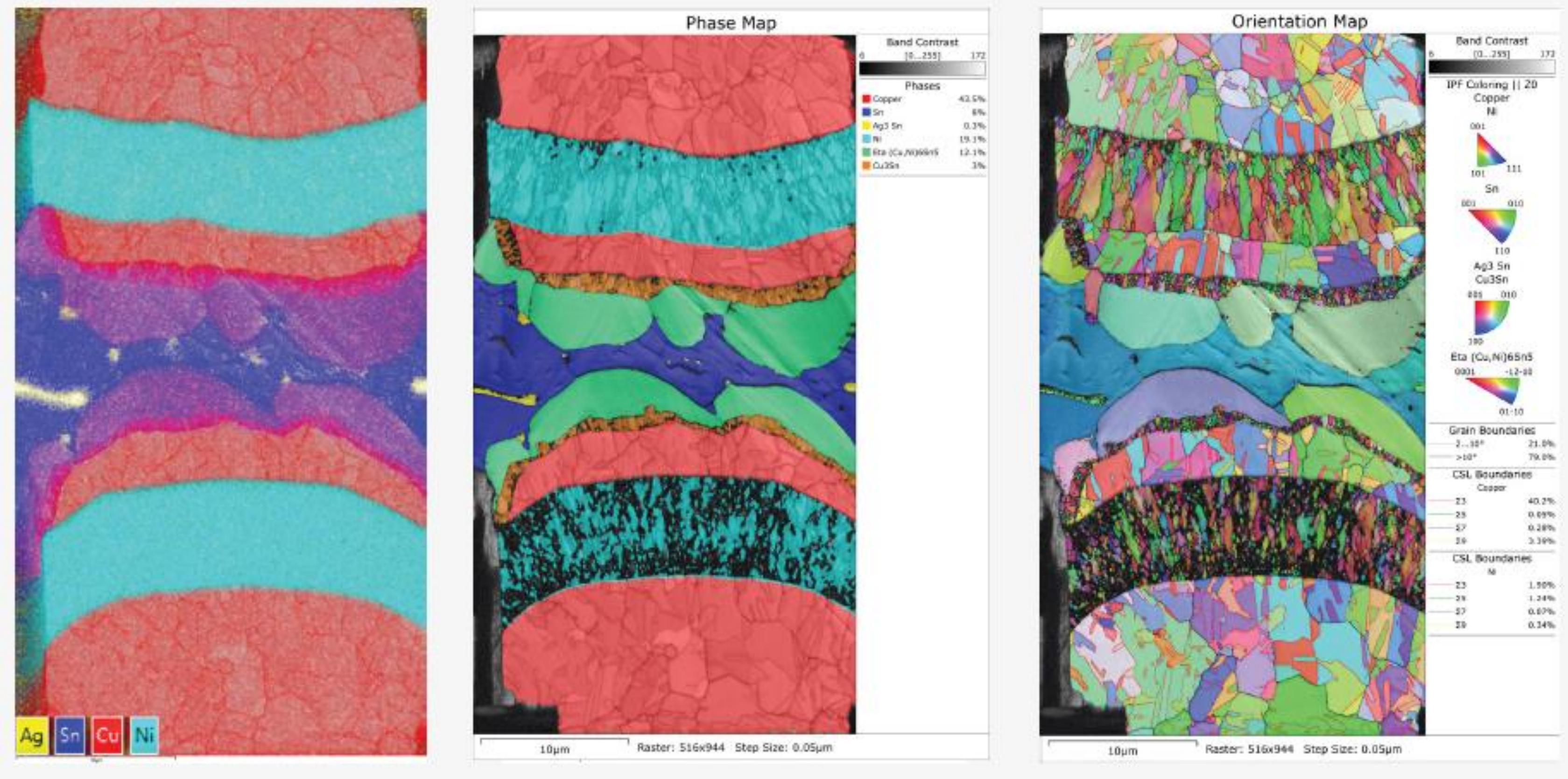


Electron BackScatter Diffraction (EBSD)



Microstructure characterization: Solder MicroBumps

Problem:
- Interconnects are critical for devices to function
- Formation of intermetallics causes stress which leads to failure
- Understanding where and why failure occurs

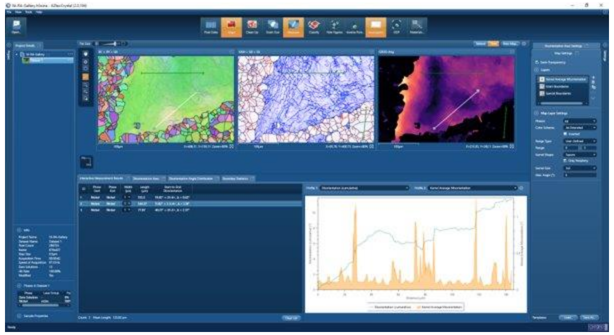


Micro-bump in flip-chip packaging

Solution:

Rapid microstructural analysis with Symmetry S3, the fastest, most sensitive CMOS EBSD camera

And AZtecCrystal, modern & intuitive data processing software



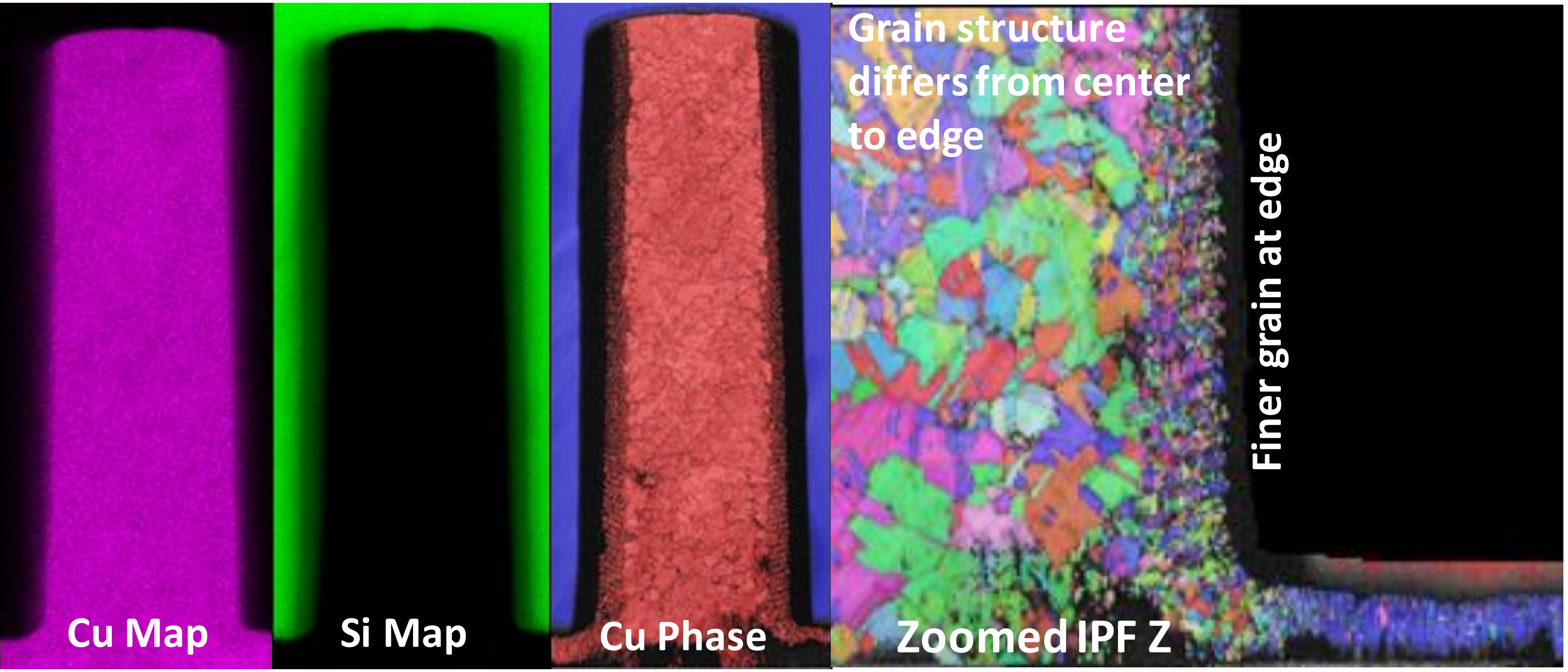
Microstructure characterization: Through Silicon Vias

Problem:

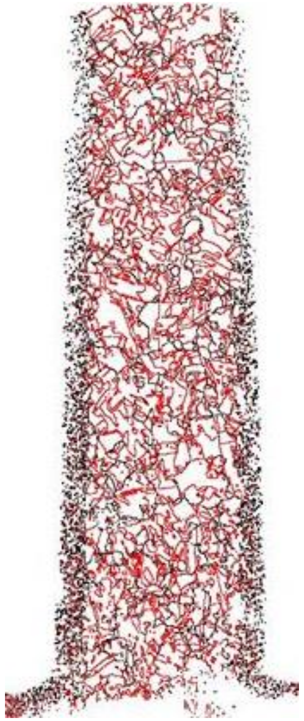
- Performance and lifetime depend on several factors including grain size, grain orientation, grain boundary types, etc.
- During operation, the TSVs will be subject to heat – which may impact the microstructure
- Preferred crystal orientation may prolong the life-span of the devices

Solution:

- Use EBSD to understand the Cu deposition process and how aging affects the microstructure



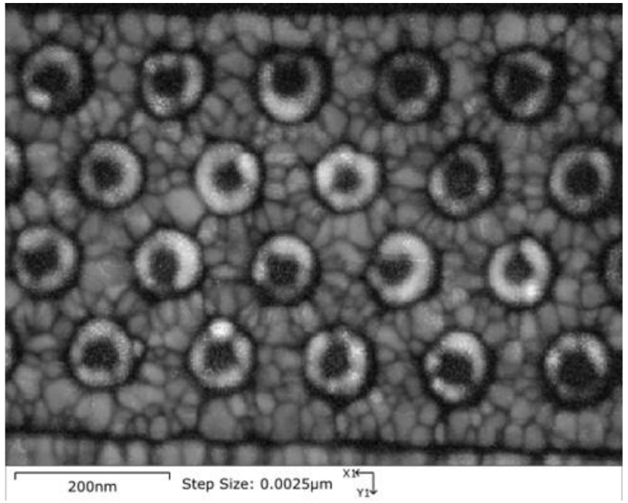
Preferred texture



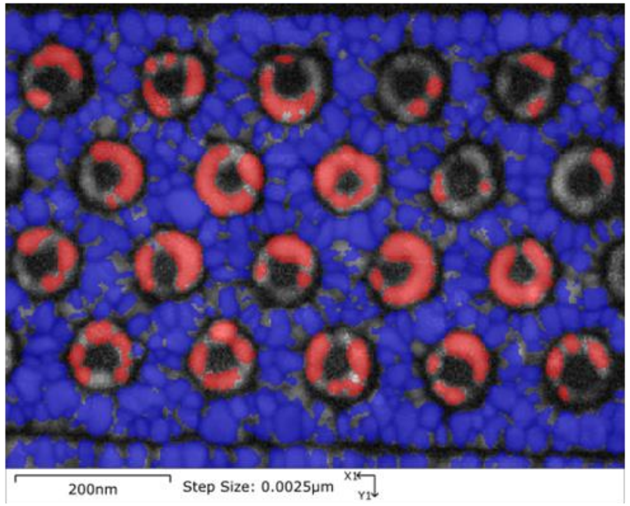
Grain boundaries >10° orientation difference

Twin boundaries shown in red – dominance of twin boundaries in the copper

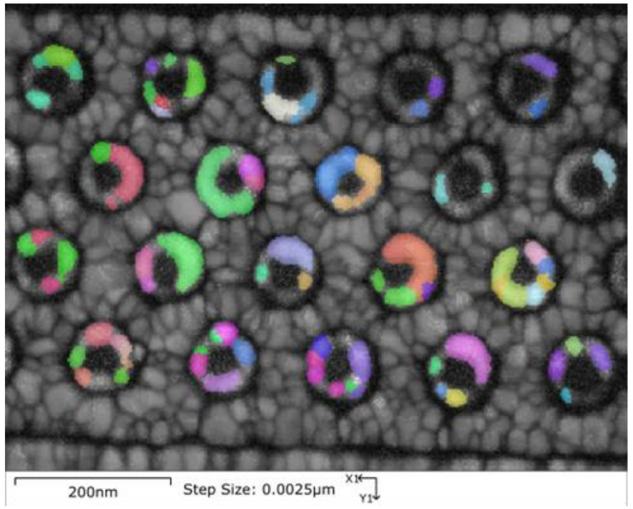
Microstructure characterization: Flash Memory



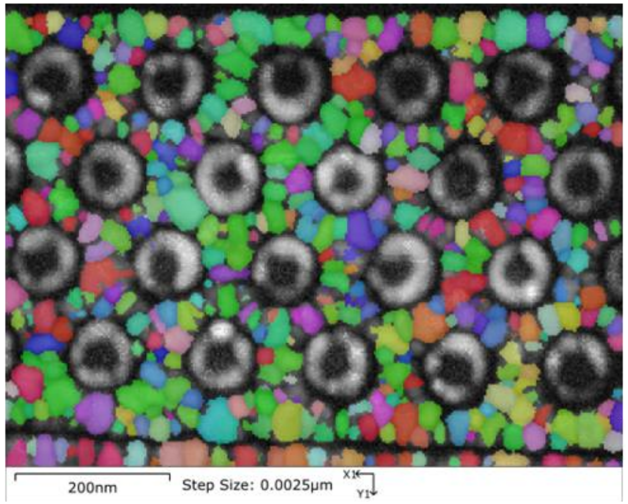
(a) Band Contrast Map



(b) Phase Map
(Red : Si Phase, Blue : W Phase)



(c) IPF Map Z (Si Phase)



(d) IPF Map Z (W Phase)

Problem:

The spatial resolution of the technique on a conventional bulk sample is on the order of 50-100 nm

Solution:

The TKD method(which performs EBSD on an electron transparent sample) using a Symmetry CMOS detector can provide enhanced sub-10 nm spatial resolution.

Acknowledgment: Hiroyuki Ito and Yasushi Kuroda, Hitachi Japan

Non-SEM Characterization

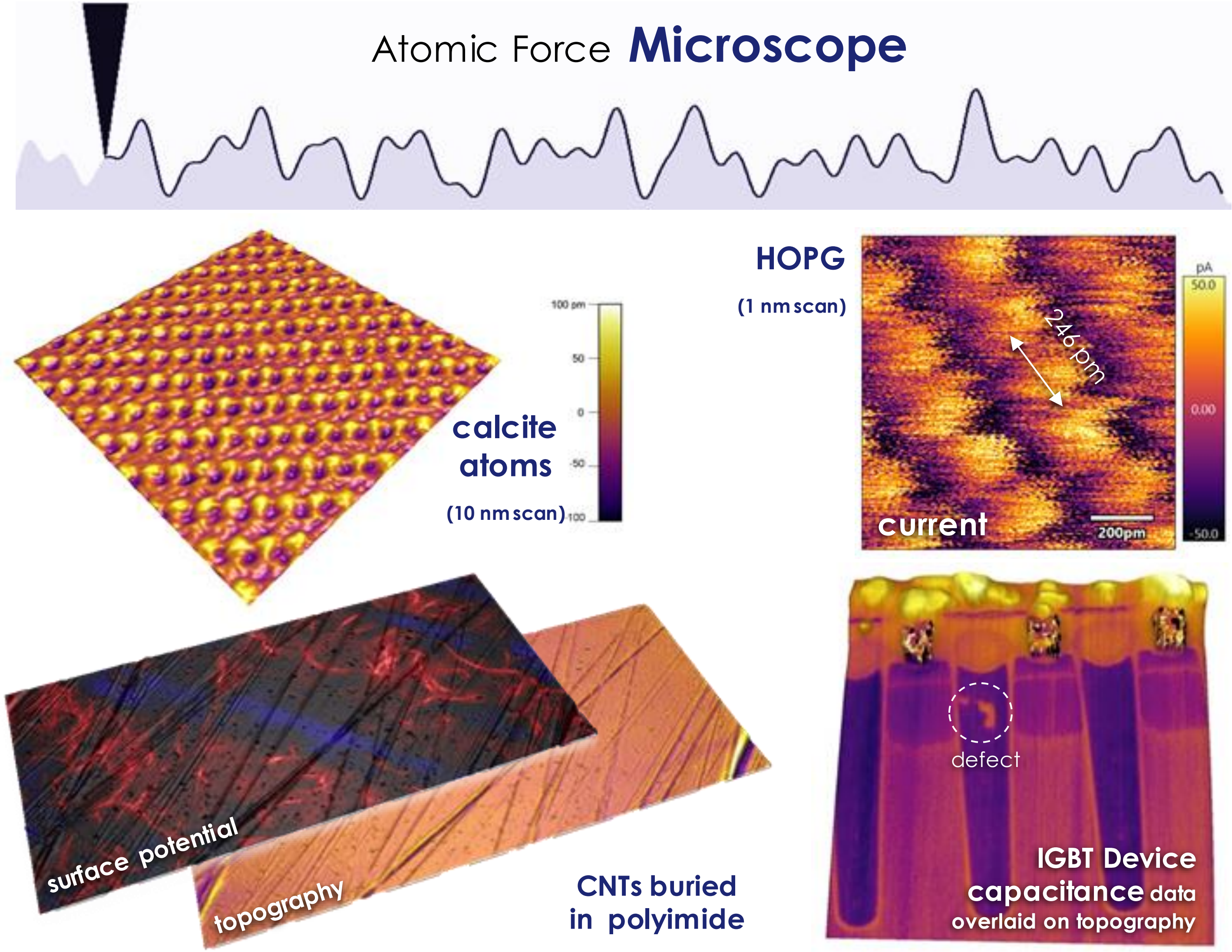
Atomic Force Microscope (AFM) (Asylum Research)

A

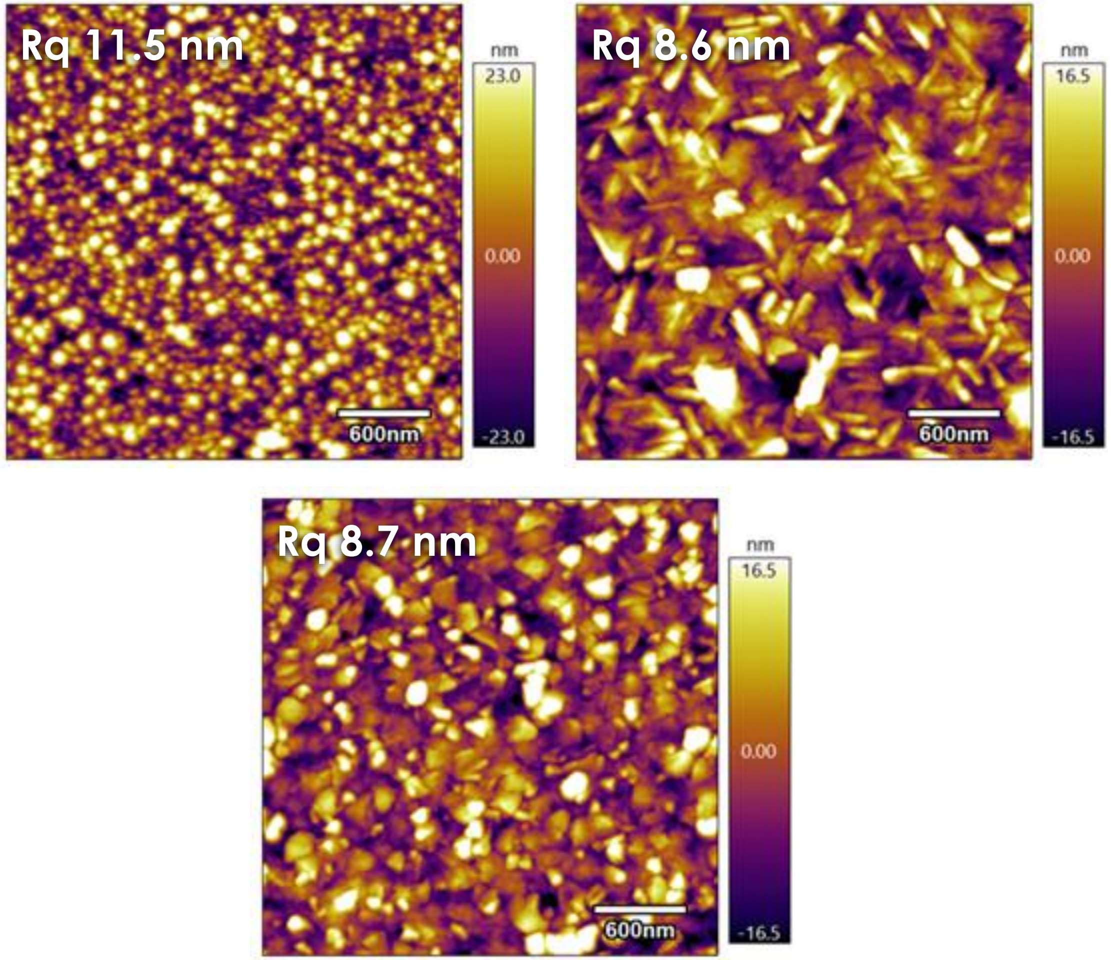
-

AFM **rties**

-
- roperties



Morphology: Growth Conditions



Ga_2O_3 on sapphire

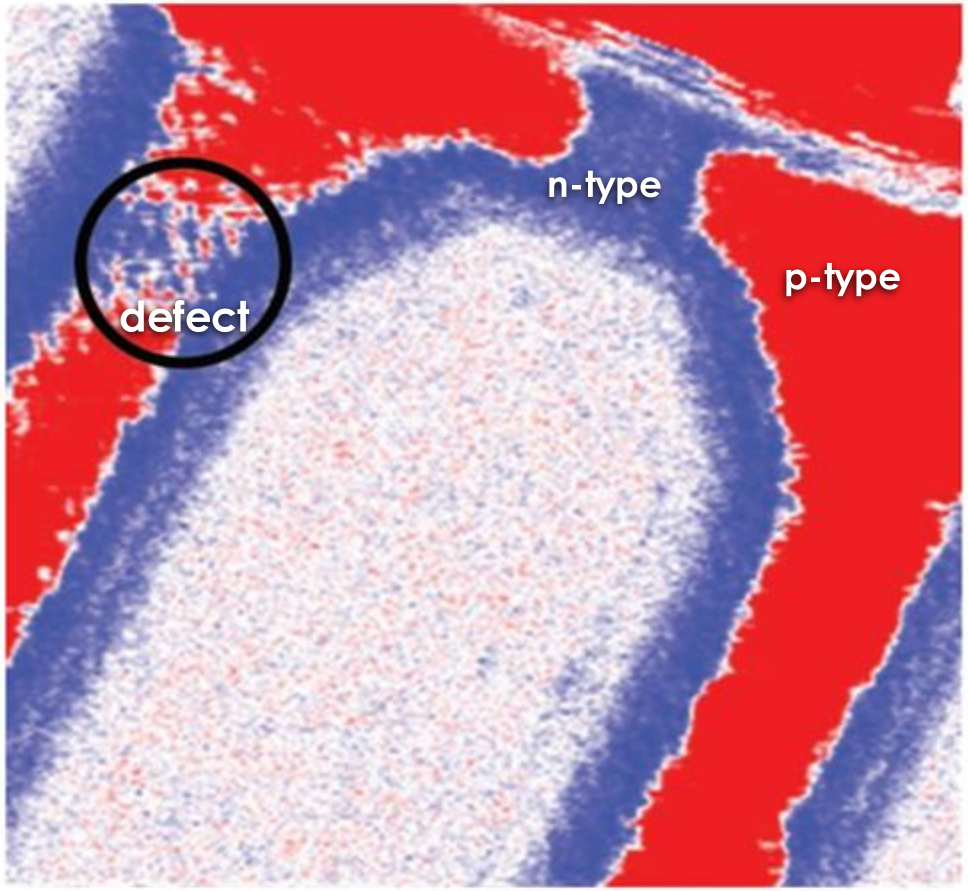
Engineering Problem:

- Growth conditions during PVD or CVD also determine final film quality
- Change in **growth conditions** (temperature, pressure, rate) results in different morphologies
- Challenging to achieve layer growth uniformity
- **Goal**: Monitor the effect of growth conditions on film **morphology**

AFM Solution:

- Ga_2O_3 on sapphire: morphology from island vs. layer-by-layer growth
- InGaTeAs on GaAs: higher rate of deposition produces stress that results in fissures
- Ga_2O_3 on sapphire: temperature and oxygen flow results in different grain sizes and crystal quality

SRAM: Variation in Dopant Type



Map of Dopant Type
shows possible failure on an **SRAM** device

Engineering Problem:

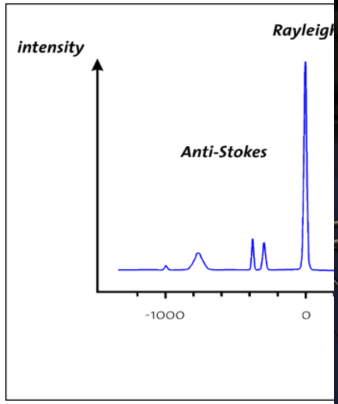
- Semiconductor devices consist of segregated regions of **p-type** and **n-type** silicon
- Failure occurs when there is an unexpected variation in dopant type
- **Goal:** Map out **dopant type variation** to determine failure in devices

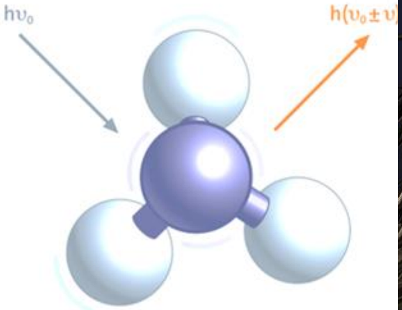
AFM Solution:

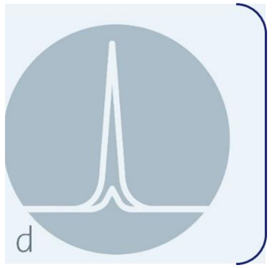
- dC/dV phase image gives **charge carrier type**
- Strong contrast indicates doped regions (red is p-type, blue is n-type)
- Image shows a **leak** between two devices, which may indicate failure

Raman Spectroscopy (WITec)

- Inelastic scat
- Energy shift
  the molecule







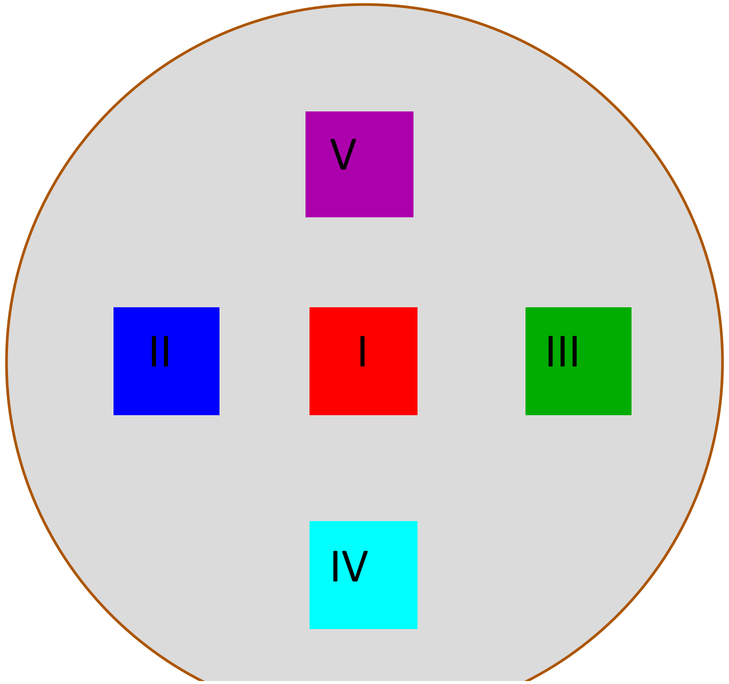
ound

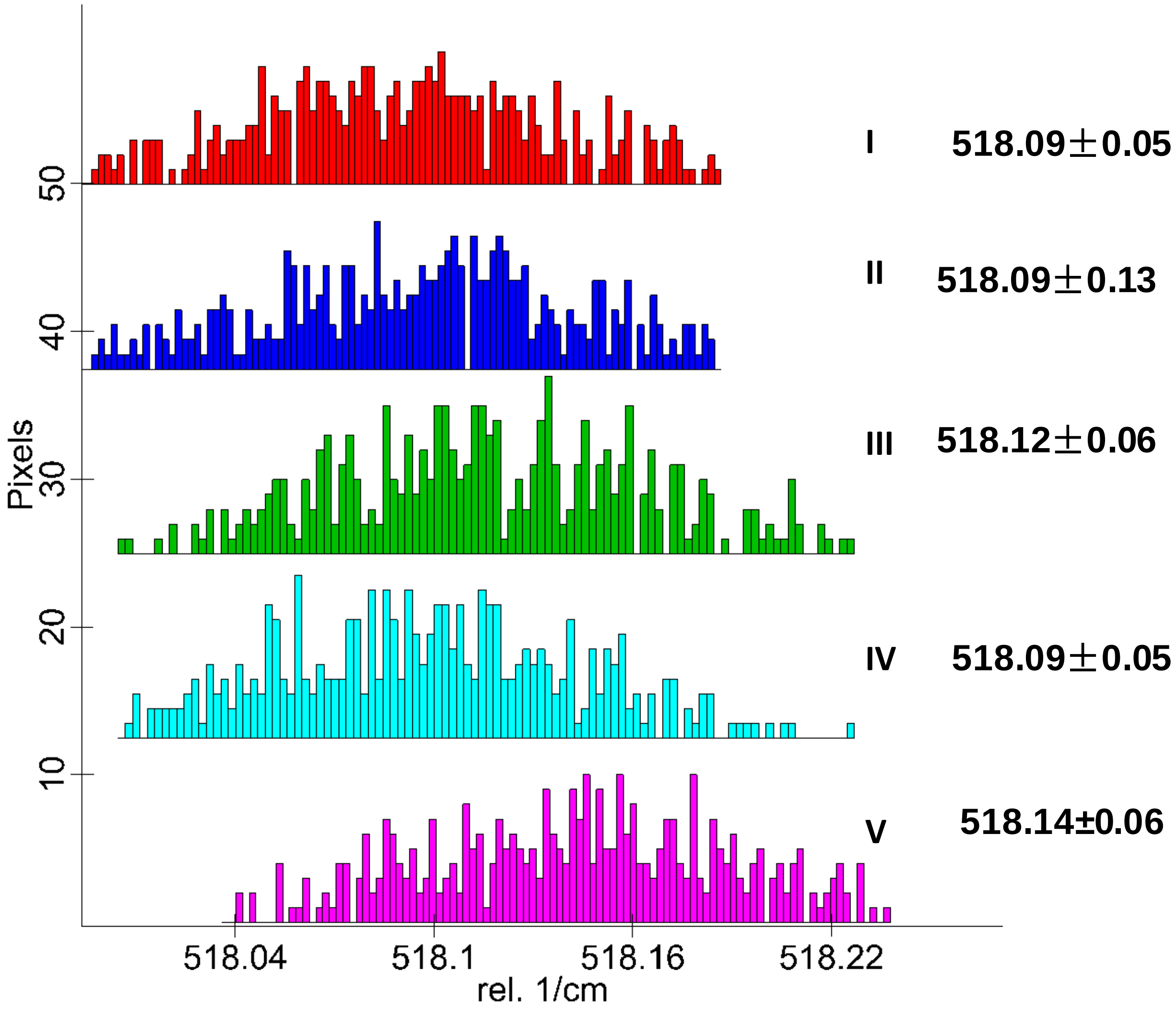
n states

ientation

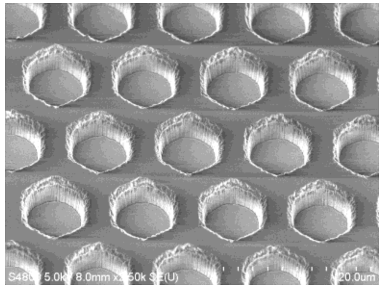
Strain in Si Wafers

- Shifts in the position of the of Si Raman peak are indicative of strain within the lattice

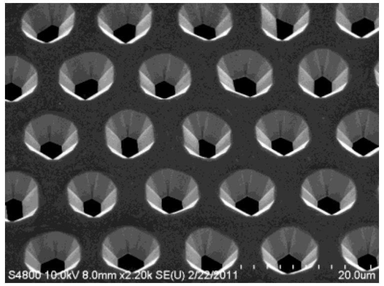




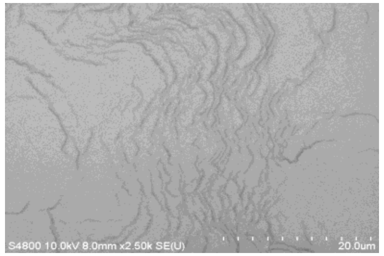
GaN 3D Stress Mapping



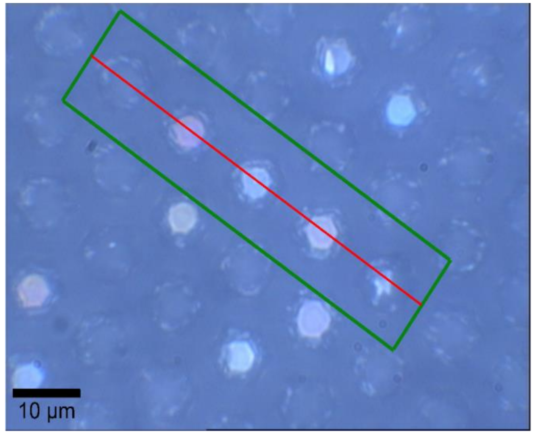
Etched sapphire substrate



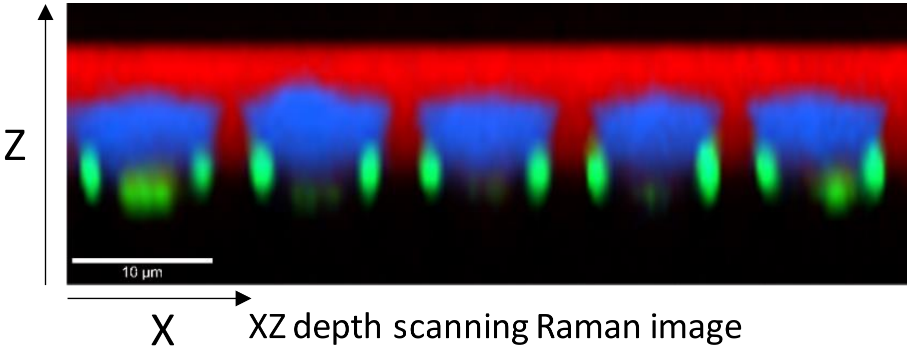
After growth of 3µm GaN



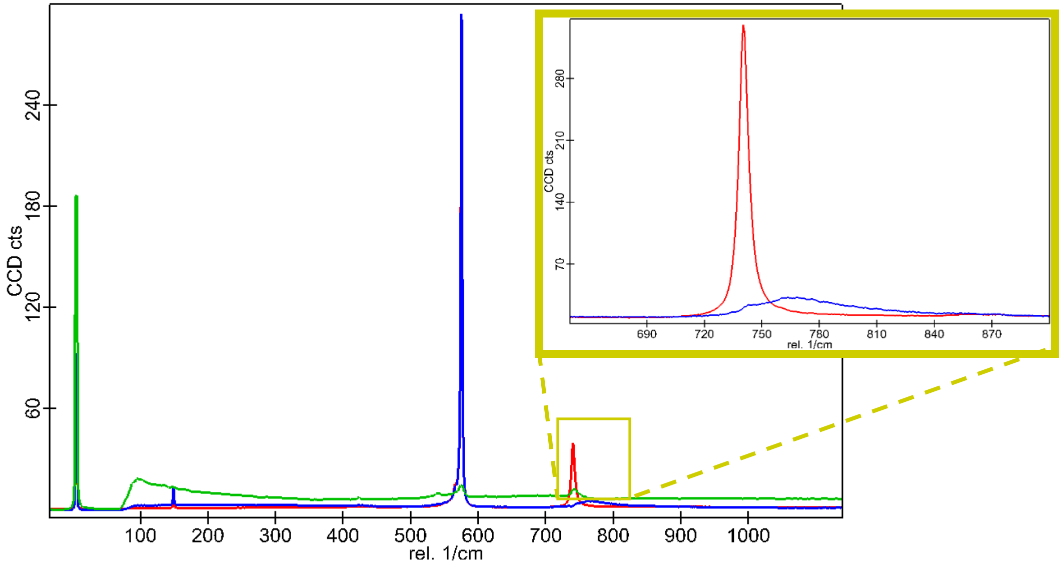
After growth of an additional 17µm thick GaN layer



GaN layer white light image



XZ depth scanning Raman image

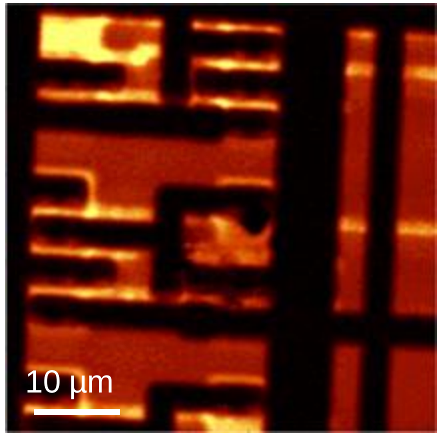


GaN Raman spectra at different locations

Sample & EM Images courtesy of Eberhard Richter; Ferdinand-Braun-Institut
Leibniz-Institut für Höchstfrequenztechnik; Berlin

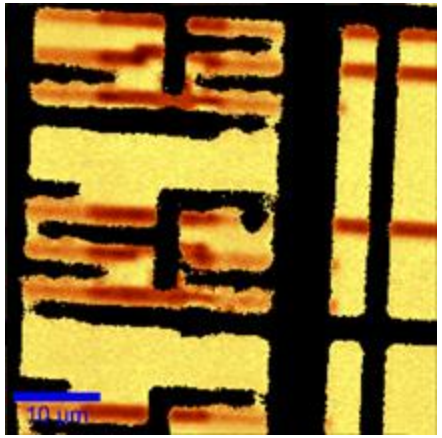
Si IC Device Contamination Analysis

Integral intensity
Si Raman line @520/cm



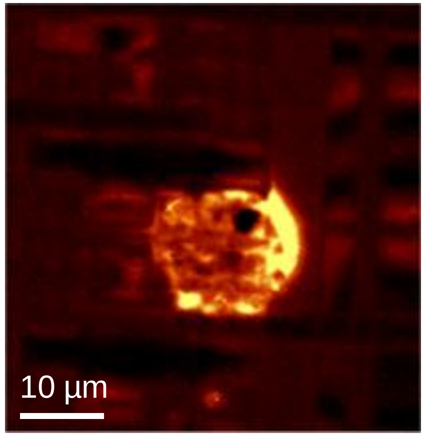
silicon

shift of peak Si line
515 - 525 /cm



stress

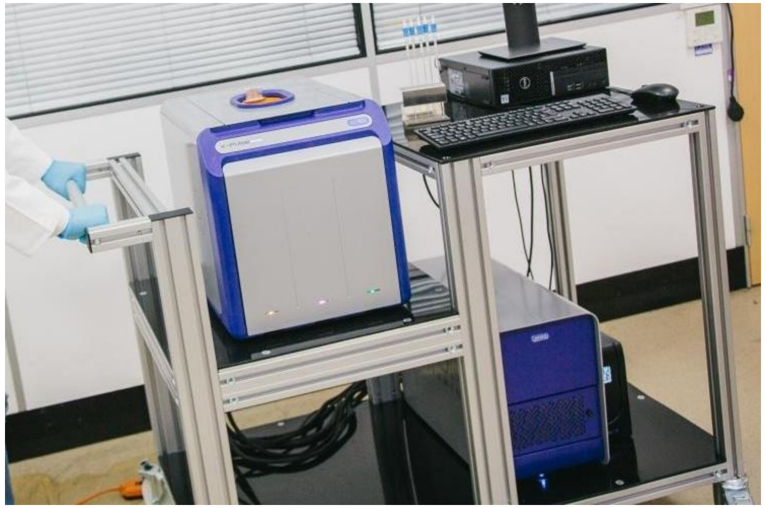
Integral intensity
carbon @ 1355-1575 /cm

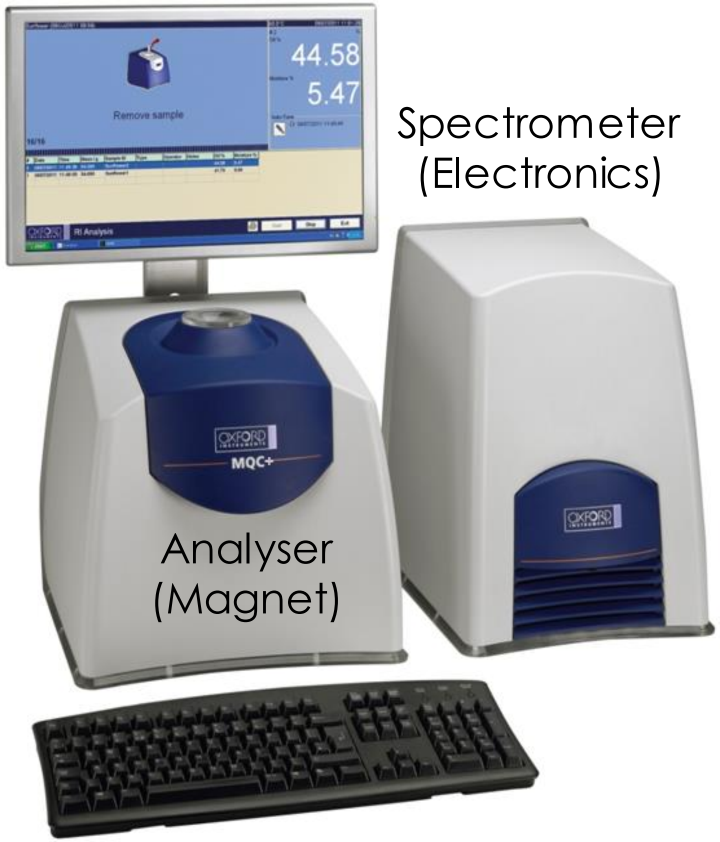


contamination

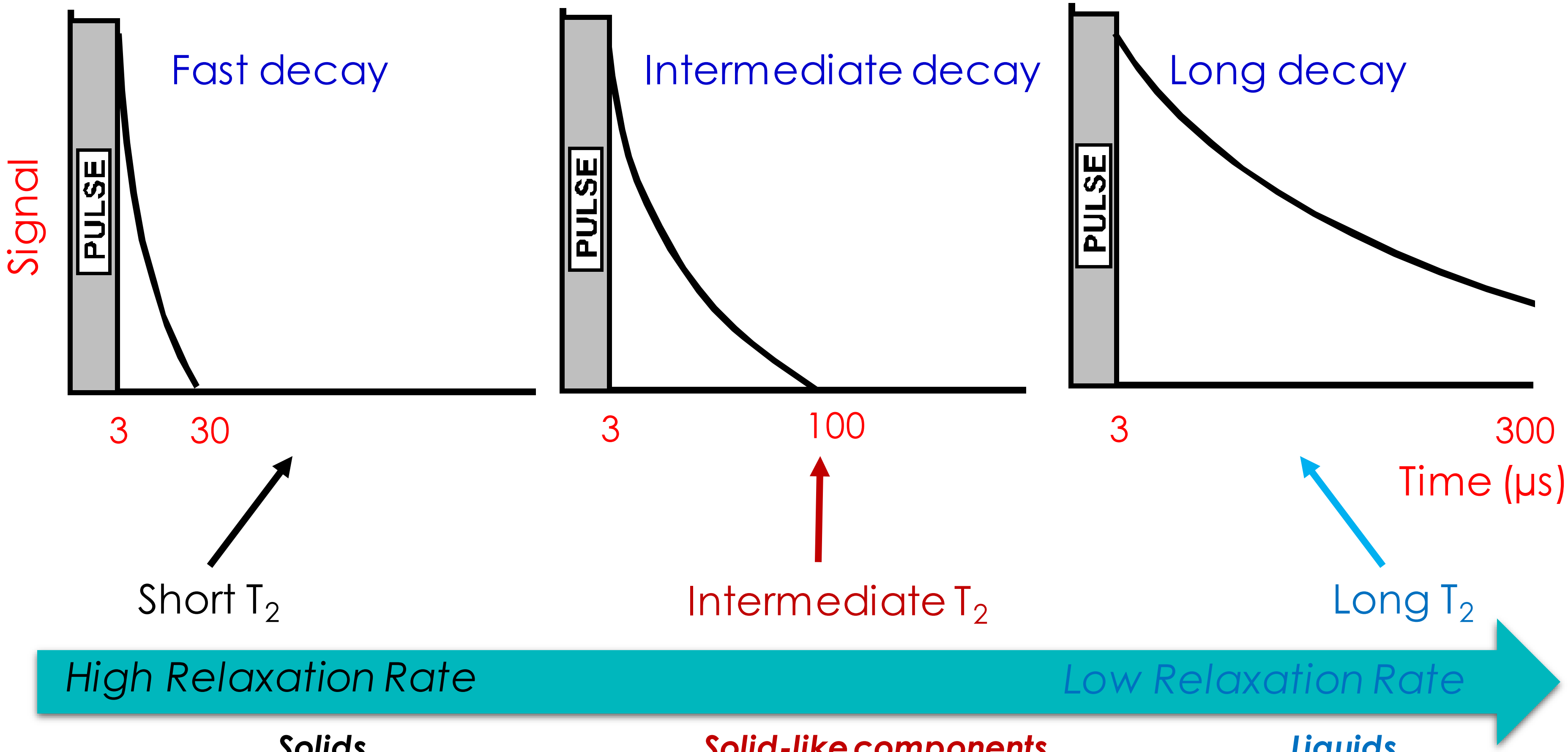
Nuclear Magnetic Resonance Spectroscopy (NMR)

- The sample is exposed to an external magnetic field (Strong magnetic fields are necessary for NMR spectroscopy)
- The intramolecular magnetic field around an atom in a molecule changes the resonance frequency, thus giving access to details of the electronic structure of a molecule and its individual functional groups.
- Applications in Polymers
  - Crosslinking
  - Plasticizer content
  - Density
  - Oil and rubber content
  - Crystallinity… and many more!





Time Domain NMR Signal



Short T_2

Intermediate T_2

Long T_2

High Relaxation Rate

Low Relaxation Rate

Solids

- *crystallite components of polymers,*
- *polymer/biopolymer matrices of samples,*
- *fibre in food products*

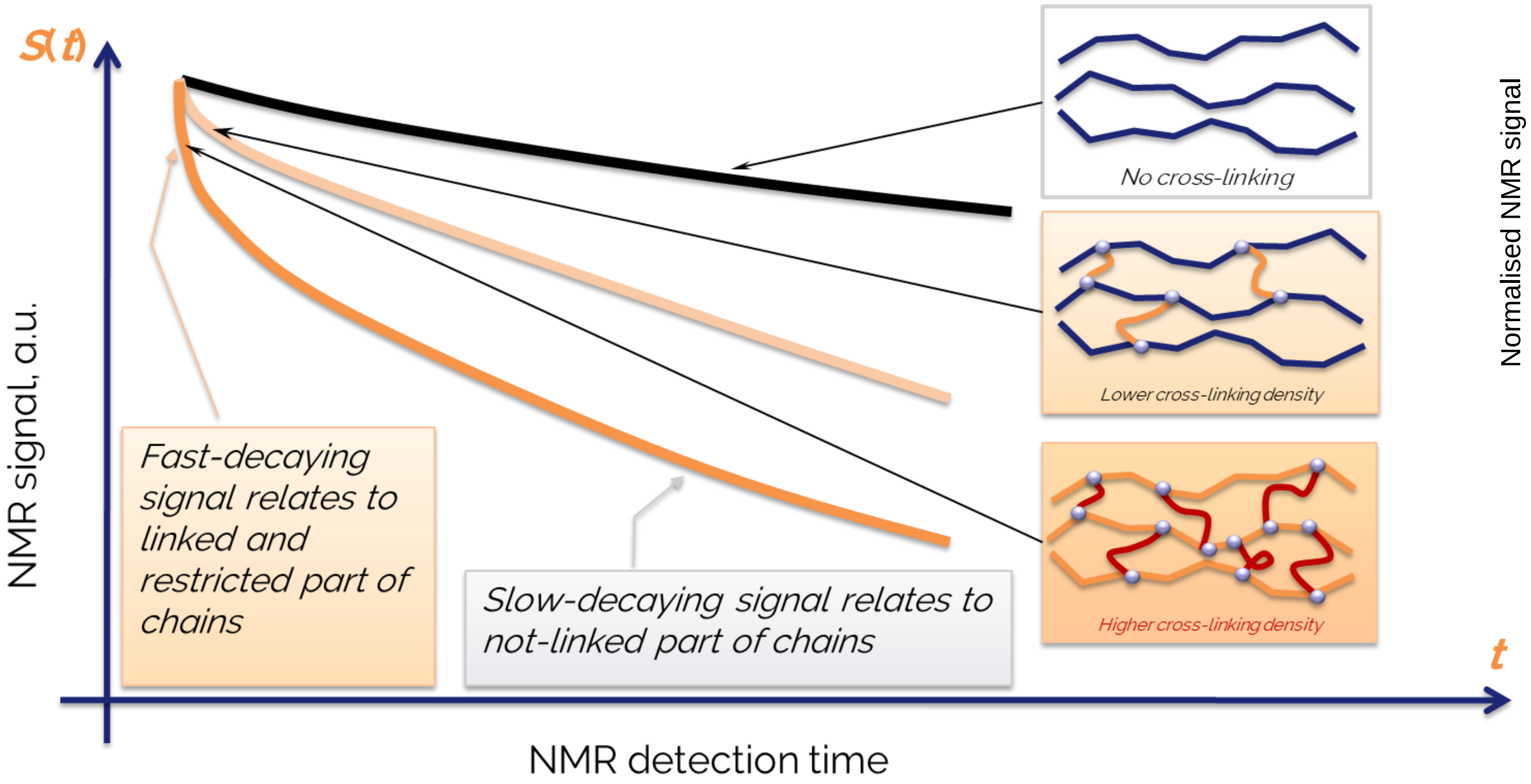
Solid-like components

- *amorphous polymer components,*
- *bound water,*
- *residual moisture*

Liquids

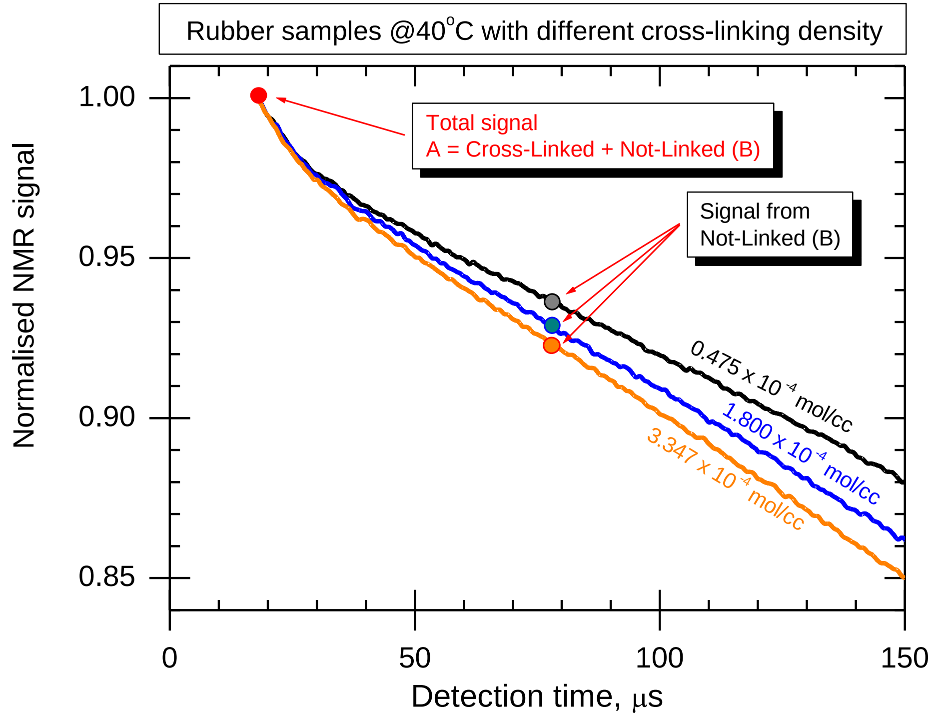
- *oils / fats,*
- *spin-finish lubricants,*
- …

Cross-linking Density in Polymers/Elastomers:



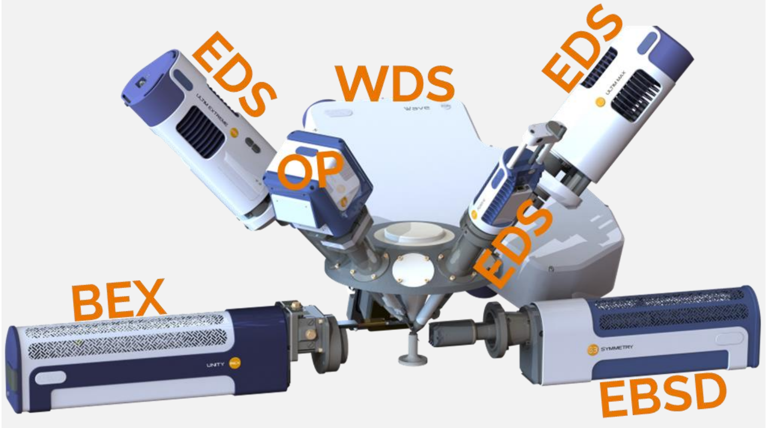
Fast-decaying signal relates to linked and restricted part of chains

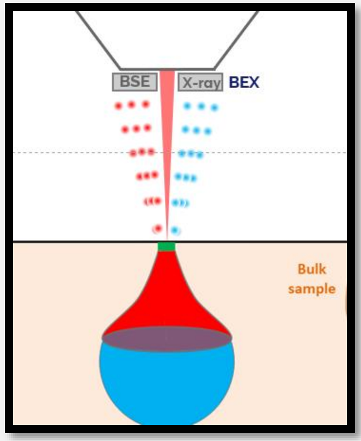
Slow-decaying signal relates to not-linked part of chains

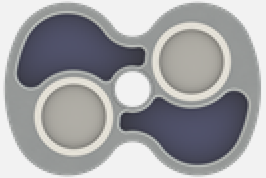


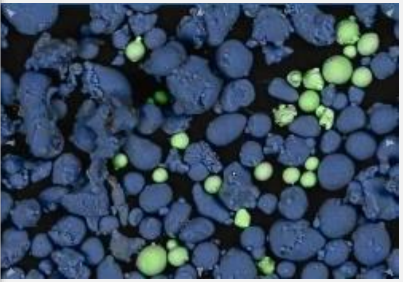
Summary:

SEM Characterization

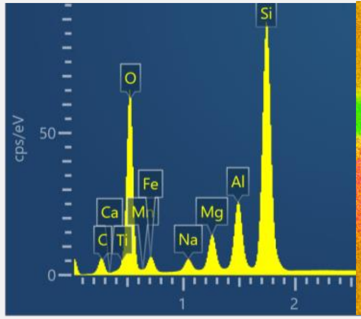


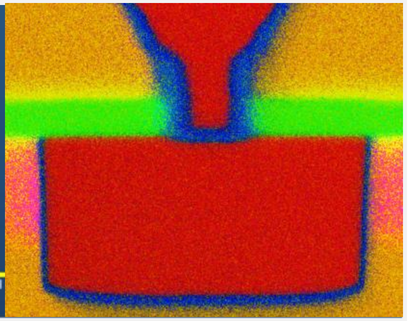


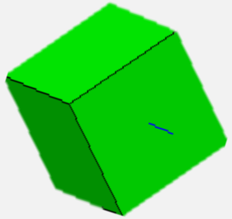


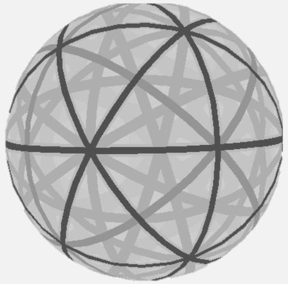


Chemistry and Crystal Orientation

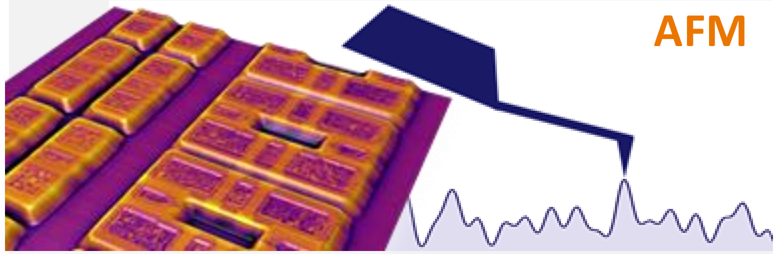




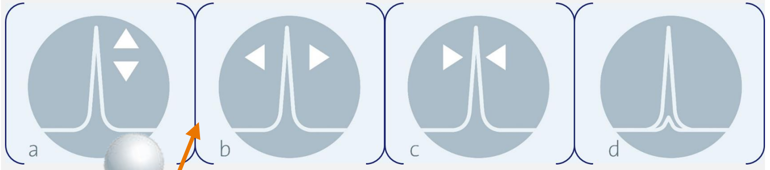


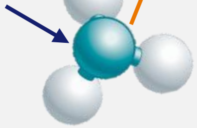


Non-SEM Characterization

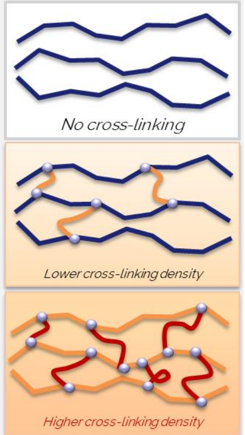


Raman





NMR



Questions and Answers

Thank you for your attention!

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