SEM Match Maker
Selecting the right SEM for imaging your samples

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NUANCE
Atomic and Nanoscale Characterization Experimental Center

Northwestern
Exploring Inner Space

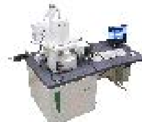
EPIC

SEM Facility Instruments

Hitachi S3400N-II SEM

Hitachi S4800-II cFEG SEM

Hitachi SU8030



LEO Gemini 1525 SEM

FEI Quanta 650 ESEM

SEM

Detailed information about surface morphology, size/shape analysis, local chemistry, crystallography/texture can be obtained with our scanning electron microscopes.

The EPIC Lab houses an impressive array of 5 Scanning Electron Microscopes (SEM's), each with its own specialized set of capabilities. From 1nm resolution imaging, to x-ray microanalysis (EDS), to imaging of pristine materials, to electron beam lithography, the EPIC Lab can help with your sample. An extensive sample preparation facility exists that aids EPIC's users in optimal sample imaging.

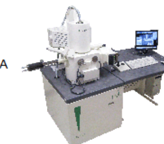
In addition to our state-of-the-art equipment, experienced staff members are on-hand to train and assist users with the microscopes and the sample preparation techniques.

SEM Introductory Video available in the *NUANCE* "Education and Outreach" section.

FEI Quanta 650 ESEM

- Schottky field emission gun for high resolution and excellent beam stability
- Allows for true SE imaging of wet, insulating samples up to 4000Pa chamber pressure
- Standard E-T SE detector, solid-state BSE detector, 2 gaseous SE detectors and a gaseous BSE detector
- Resolution of <2nm @ 30kV in high vacuum, <2nm @ 30kV in ESEM and <3.5nm @ 3kV in Low Vacuum mode
- Automatic, programmable stage with 6" wafer capacity
- Integrated Oxford AZtec EDS and EBSD system
- NPGS pattern generation system for high resolution electron beam lithography (eBL)
- High-speed beam blanking capabilities for lithography
- Integrated beam current measurement system
- Peltier variable temperature stage for humidity cycling experiments
- FEI Quanta 650 ESEM Manual

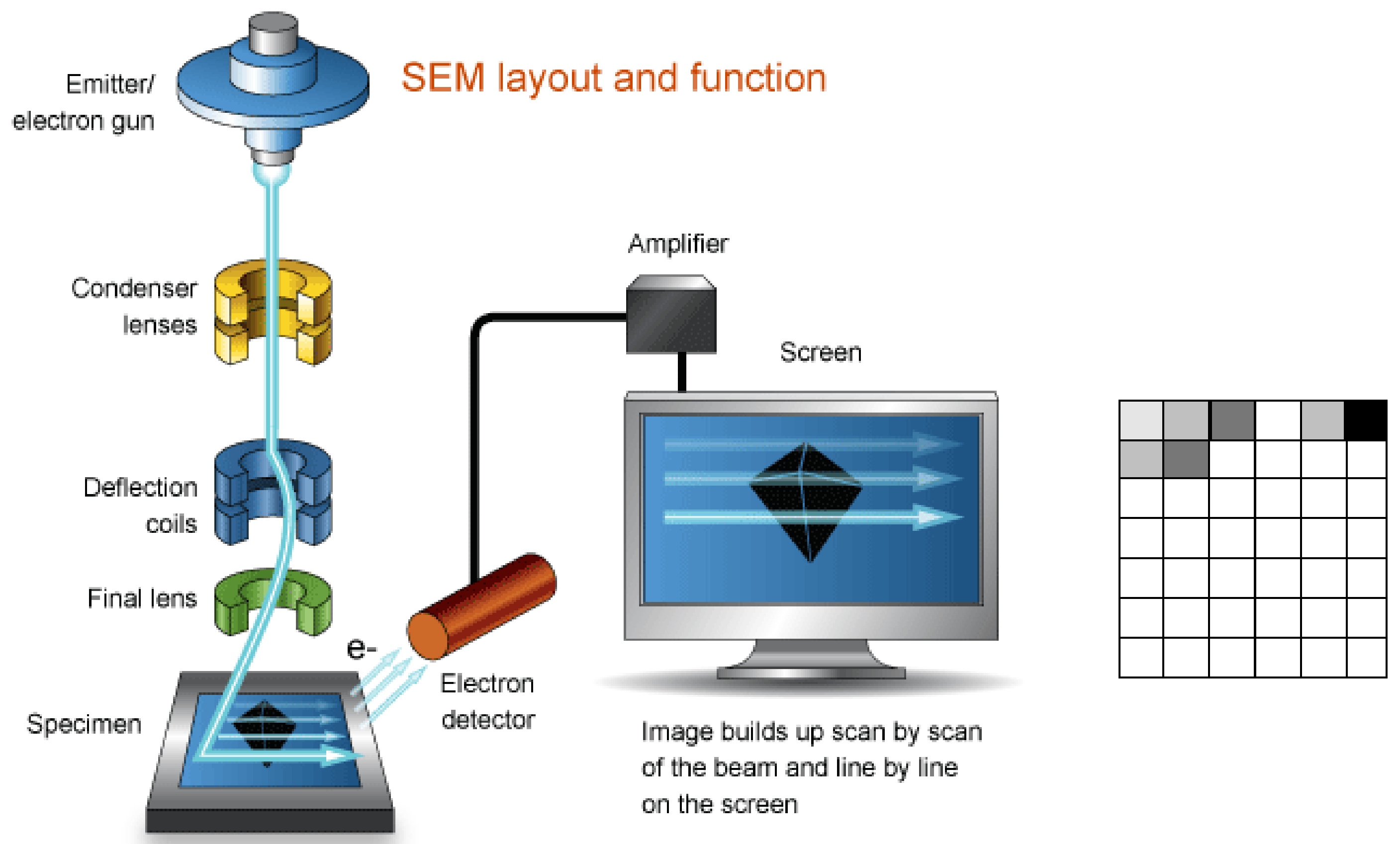
LEO Gemini 1525



- Schottky field emission gun for high resolution and excellent beam stability
- Large specimen chamber capable of imaging up to a 6" wafer
- Spatial resolution of 1.5nm at 20kV and 3.5nm at 1kV
- High probe current with stability better than 0.5% per hour. Variable from 4pA to 10nA
- In-lens and lower SE detectors. Robinson backscattered electron detector for compositional contrast
- Leo Gemini 1525 Manual

Hitachi S4800-II cFEG SEM

- Cold source Field Emission SEM


SEM layout and function
Emitter/
electron gun
Condenser
lenses
Deflection
coils
Final lens
Specimen
e-
Electron
detector
Amplifier
Screen
Image builds up scan by scan
of the beam and line by line
on the screen

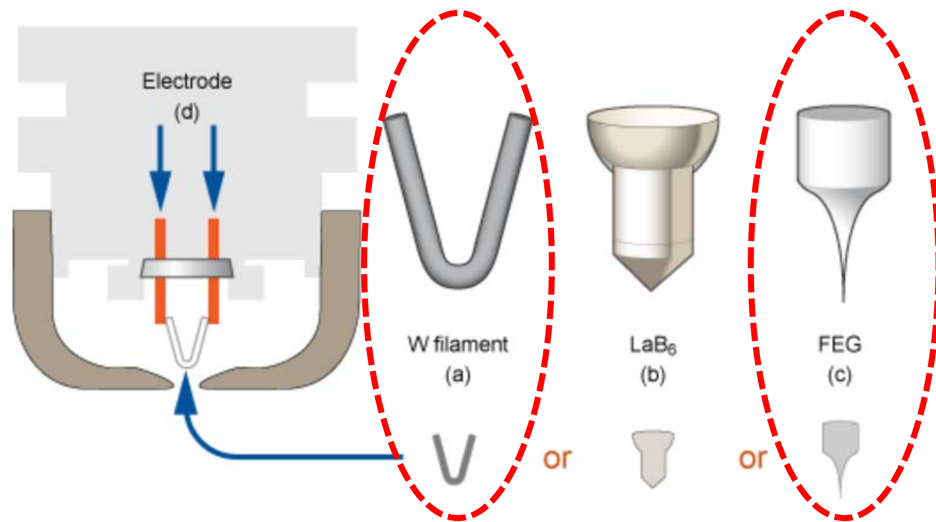


Table 3 A comparison of SEM filament types

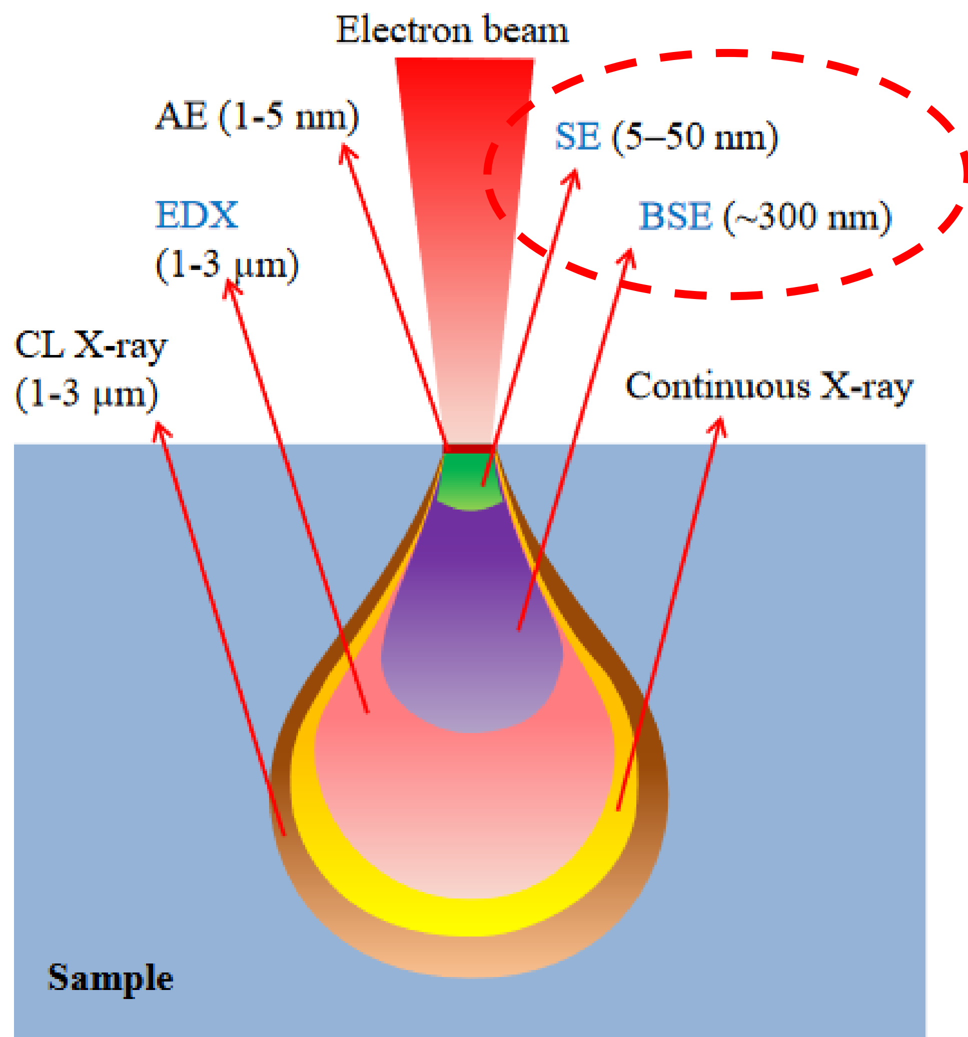
Emission (kind of gun)	Thermionic		Field Emission	Schottky
	W (tungsten hairpin)	LaB6 (single/multi crystal)	FE (tungsten)	Tungsten /zirconium oxide (single crystal)
Diameter of Electron Source (nm)	30,000	10,000	5	20
Brightness (A/cm2.sr)	106	107	109	108
Energy spread (eV)	1-5 (~2)	0.5-3.0 (~1.5)	0.2-0.3	0.3-1.0

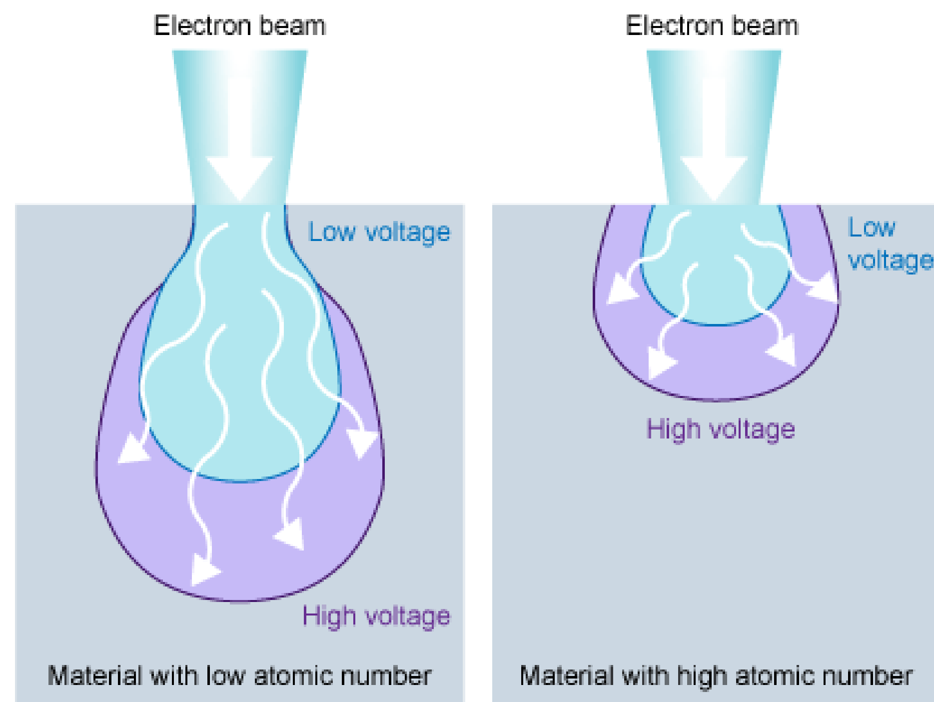
Thermionic (W, LaB_6): Apply thermal energy to filament to coax electrons away from gun towards specimen

- High temperature (large energy spread)
- Large source size

Field emission gun (FEG): Create strong electric field to pull electrons from source

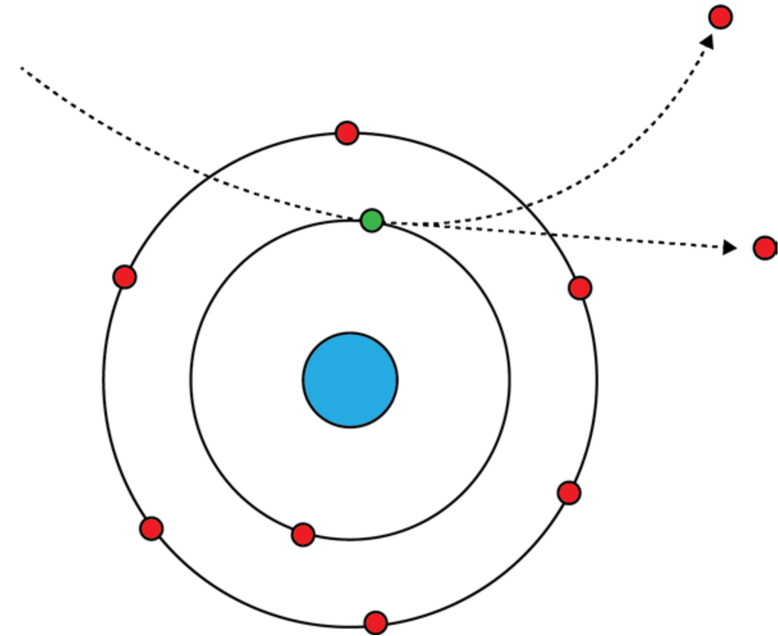
- Cold cathode FEG
  - Low temperature
  - Very small source size
  - Unstable current
- Schottky FEG
  - High temperature
  - Small source size
  - Very stable current

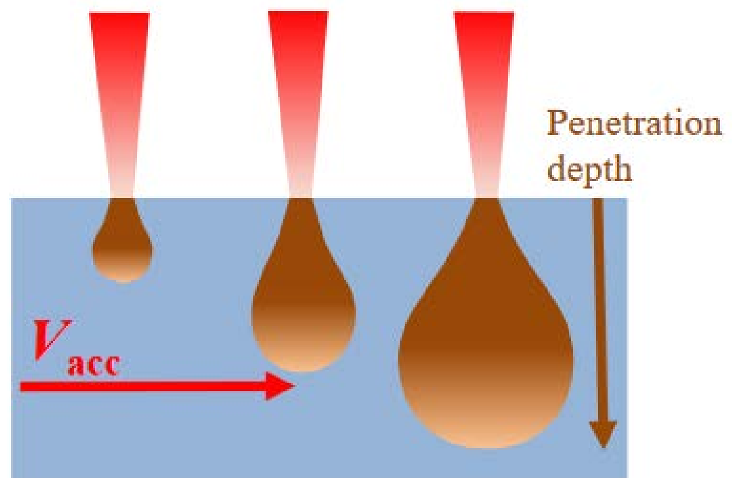

Electron beam
AE (1-5 nm)
SE (5–50 nm)
BSE (~300 nm)
EDX
(1-3 μm)
CL X-ray
(1-3 μm)
Continuous X-ray
Sample

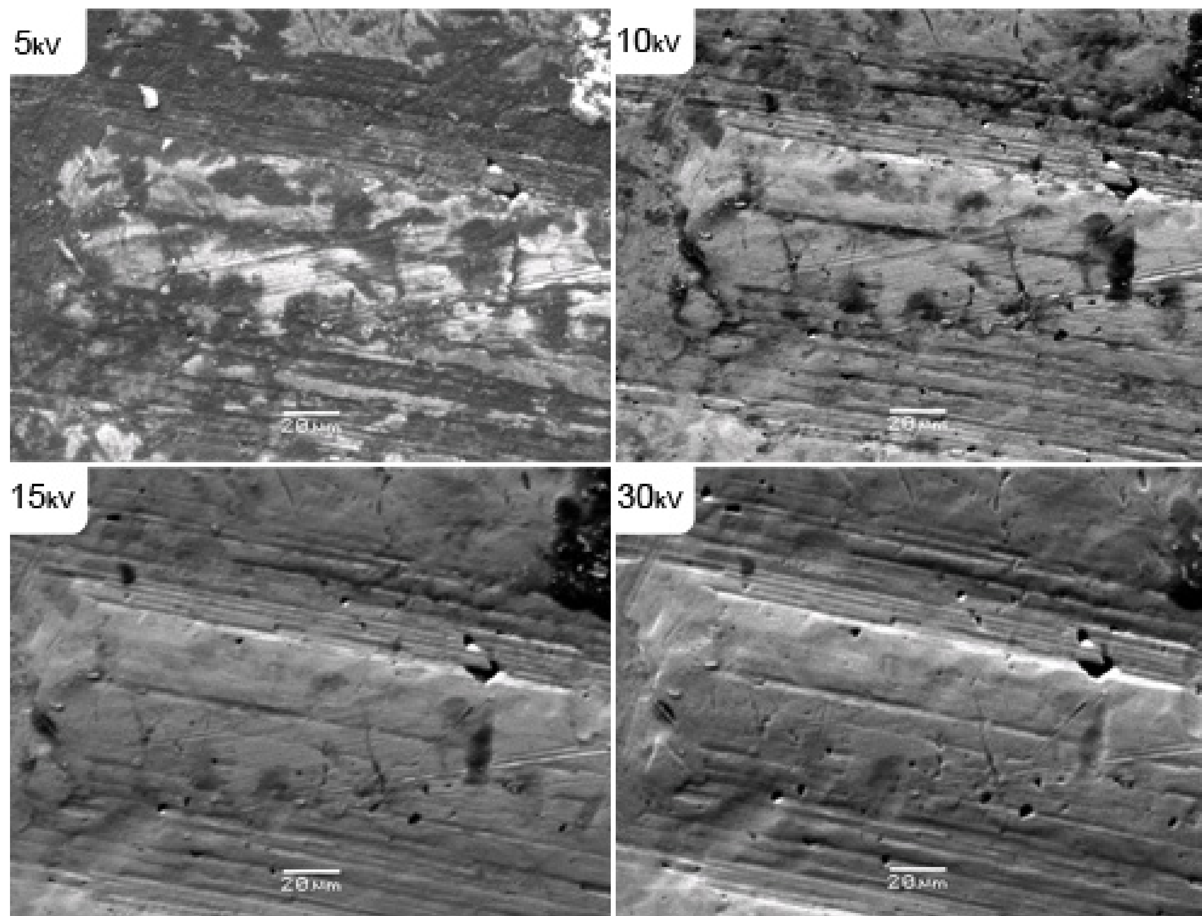

Electron beam
Electron beam
Low voltage
Low voltage
High voltage
High voltage
Material with low atomic number
Material with high atomic number

Secondary Electrons

- Generated from collision between primary beam electrons and loosely bonded outer electrons
- Low energy electrons (~10-50 eV)
- Only SE close to surface escape
  - Topographic information obtained
- Number of SE is greater than number of incoming electrons

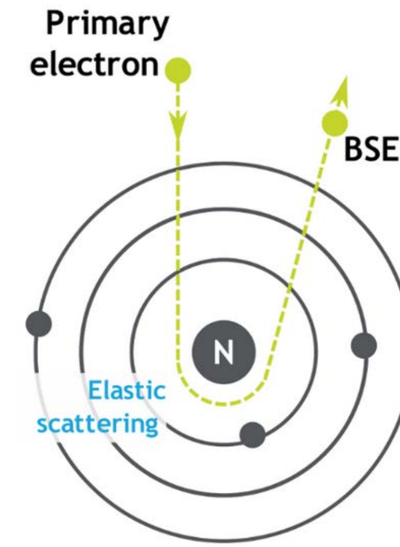


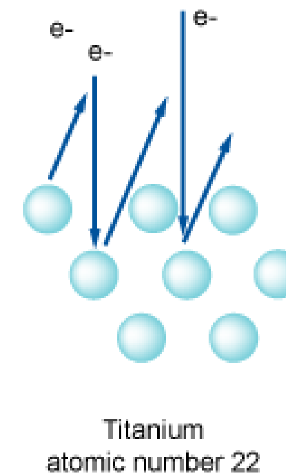

Penetration
depth
V_{acc}

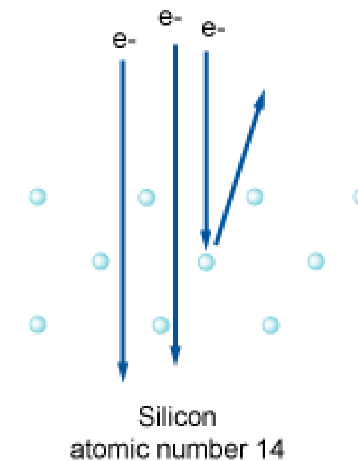

5kV
10kV
15kV
30kV
20μm
20μm
20μm
20μm

Backscattered Electrons

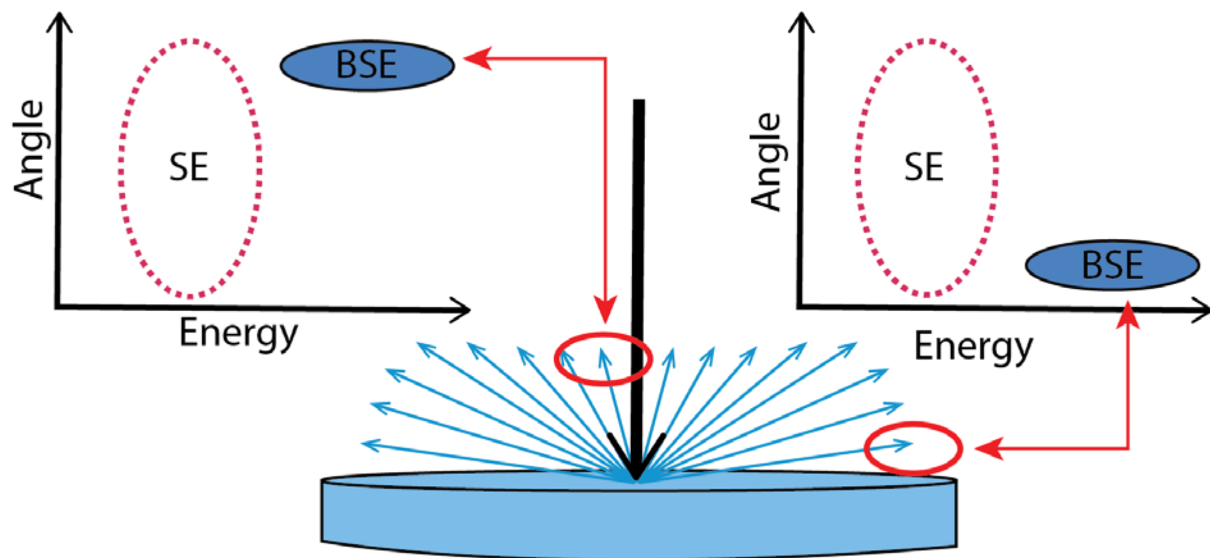
- BSE
  - Electron (from incoming beam) is deflected by electrostatic field of positive nucleus
  - Elastically scattered electrons
    - Don't lose energy
  - High-energy electrons (>50 eV)
    - Able to escape from deeper in sample
    - Information less restricted to surface detail – lower spatial resolution



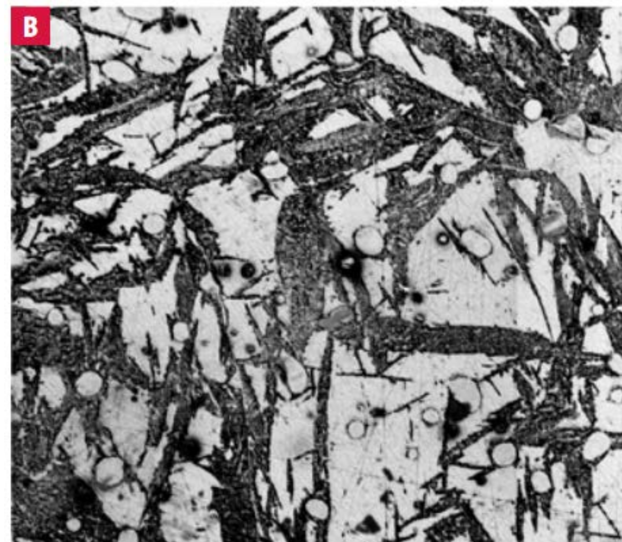




HA BSE vs. LA BSE



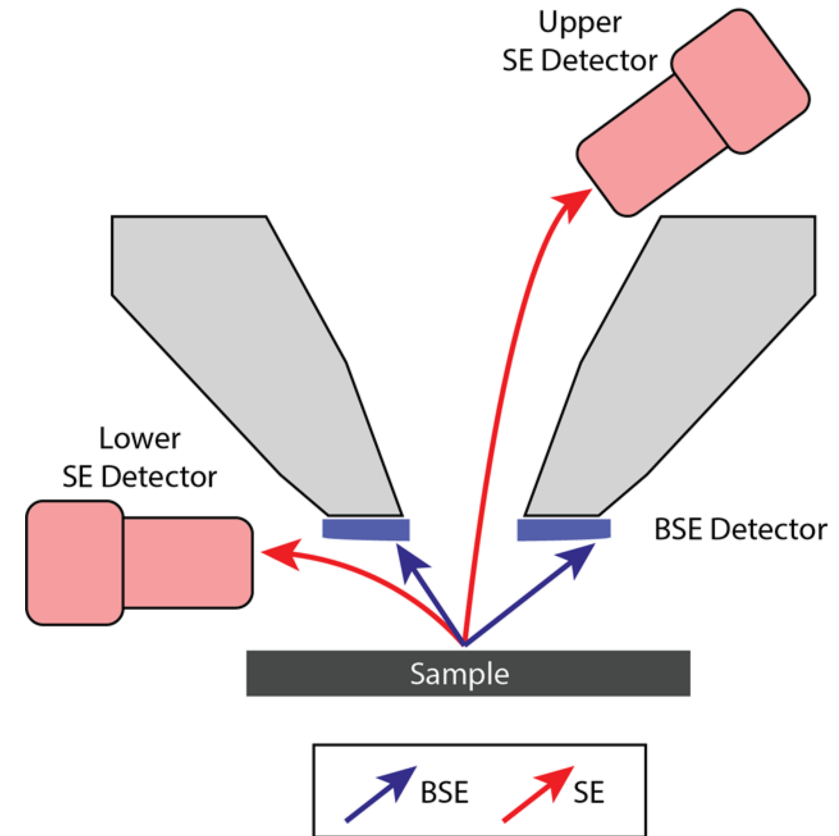
- Angles of elastic scattering range from 0-180 degrees



Steel sample (B) high angle BSE showing z contrast and (C) low angle BSE showing topography (photos from FEI)

Electron Detectors

- SE
  - Upper SE – SE or BSE by energy filter
  - Lower SE (E-T) – SE1-4 + BSE
- BSE
  - Solid state (multi-segment)
- STEM
  - BF/DF (multi-segment)



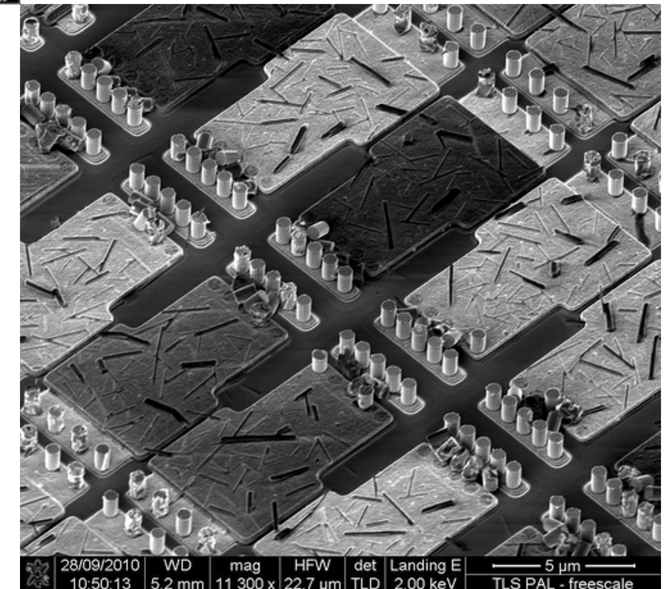
Hitachi S-3400N

- W hairpin filament
- E-T lower SE detector
- Variable pressure
- 5 segment BSE detector (3D reconstruction)
- Oxford SDD EDS
- Oxford Wave WDS
- Applications – general SEM, quantitative EDS/WDS, low vac imaging, medium resolution (>50 nm features)

Charging

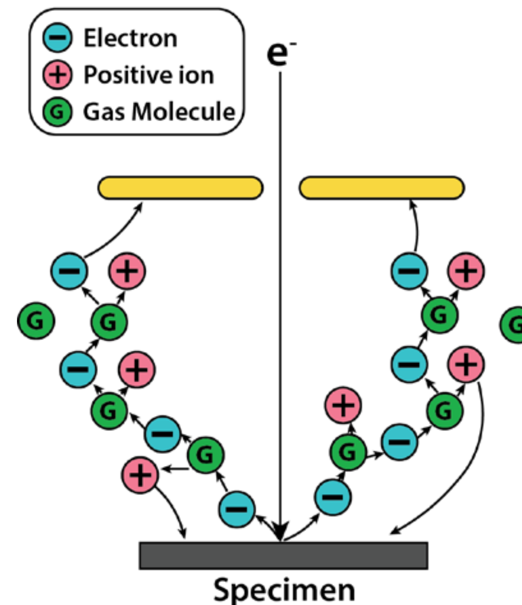
- Image artifacts
  - Streaking
  - Voltage contrast

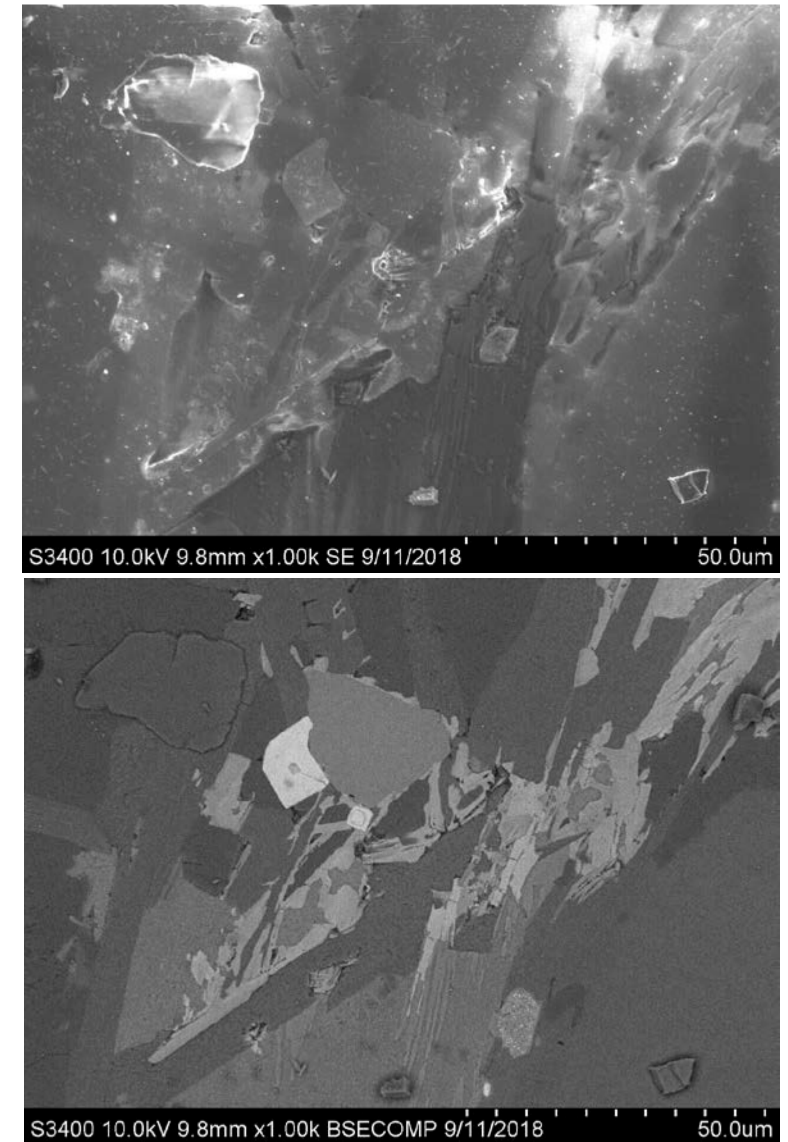
- Solutions
  - Coating (Au, Au/Pd, Pt, Ir, Os)
  - Variable pressure SEM



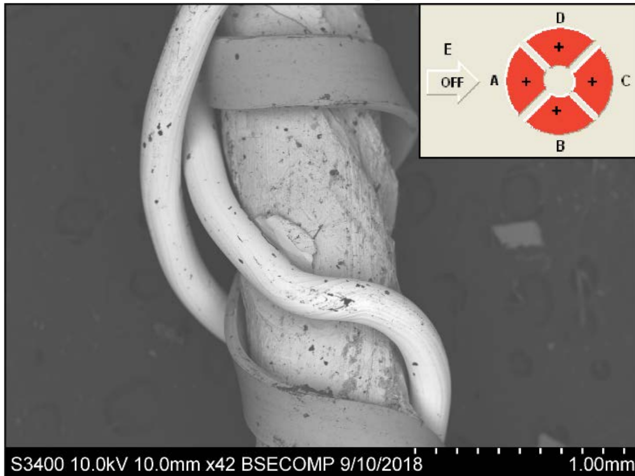
Variable Pressure (VP-SEM)

- Introduce gas into chamber
  - 270 Pa (~2 Torr)
- ESED
- Viewing polymers, biological samples, and museum samples that cannot be changed in any way, and geological materials
- Beam broadening = noise



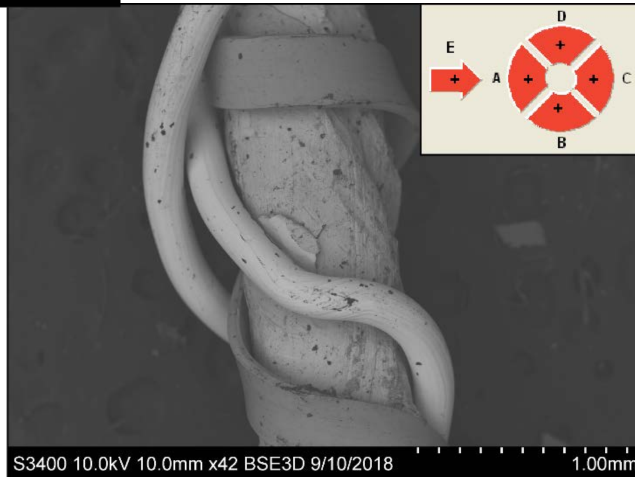


5-Segment BSE Detector

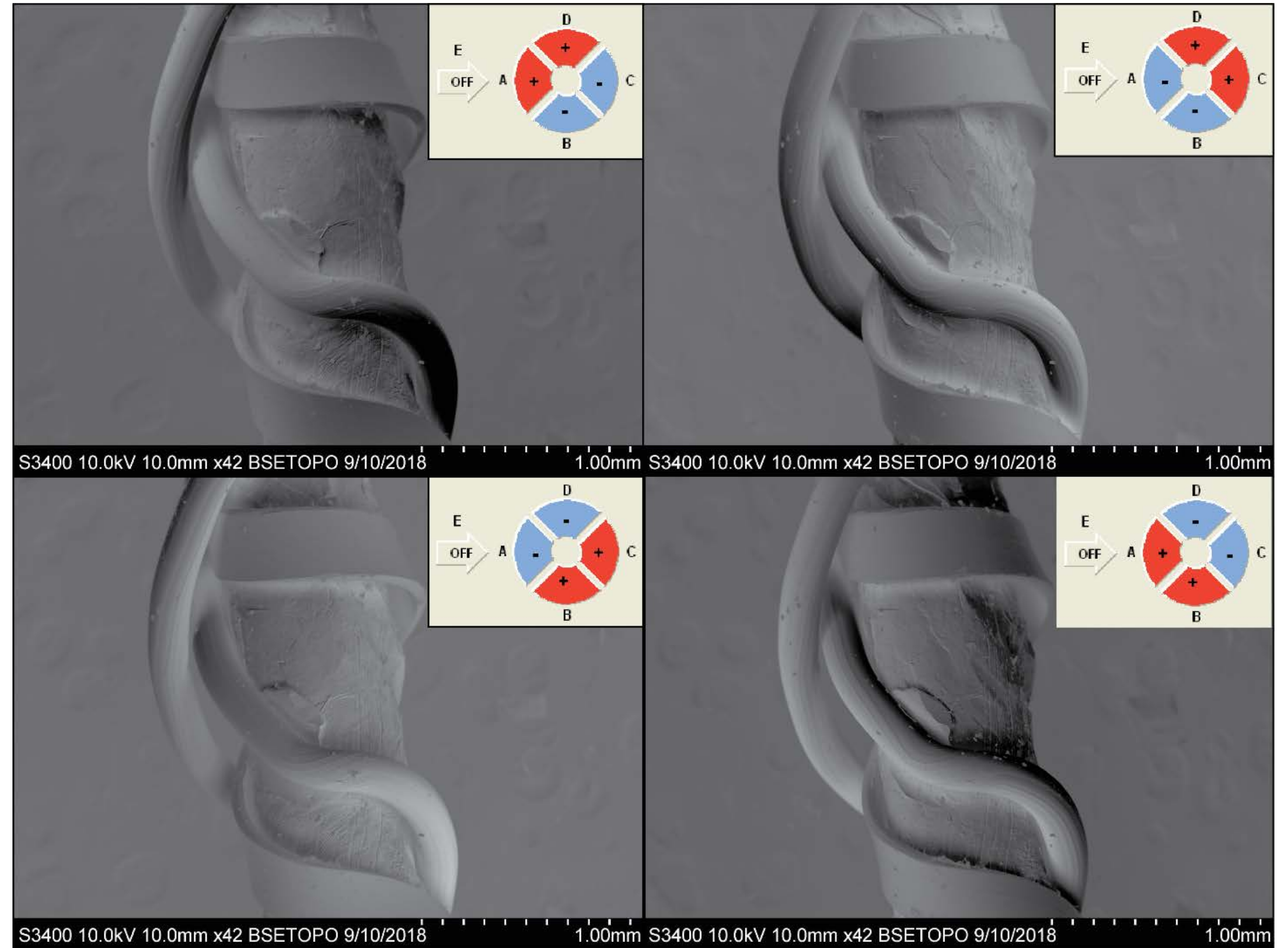


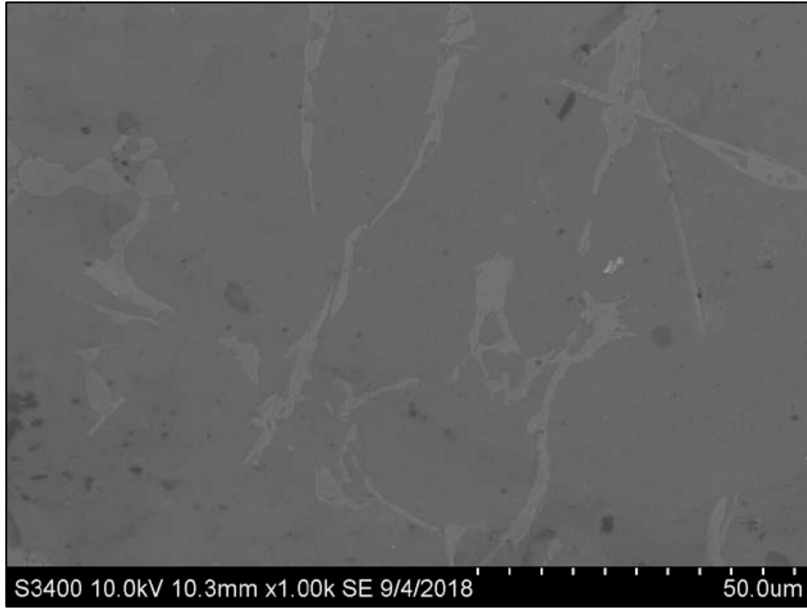
Comp Mode

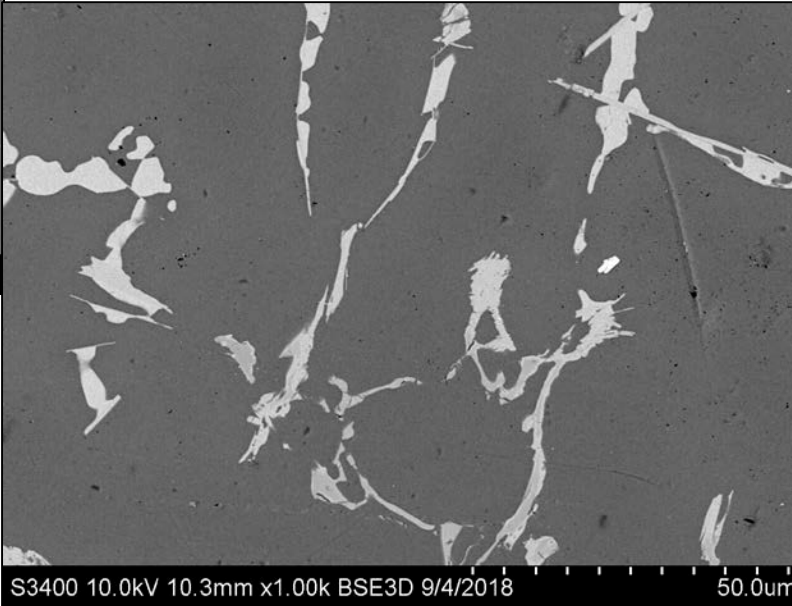
3D Mode

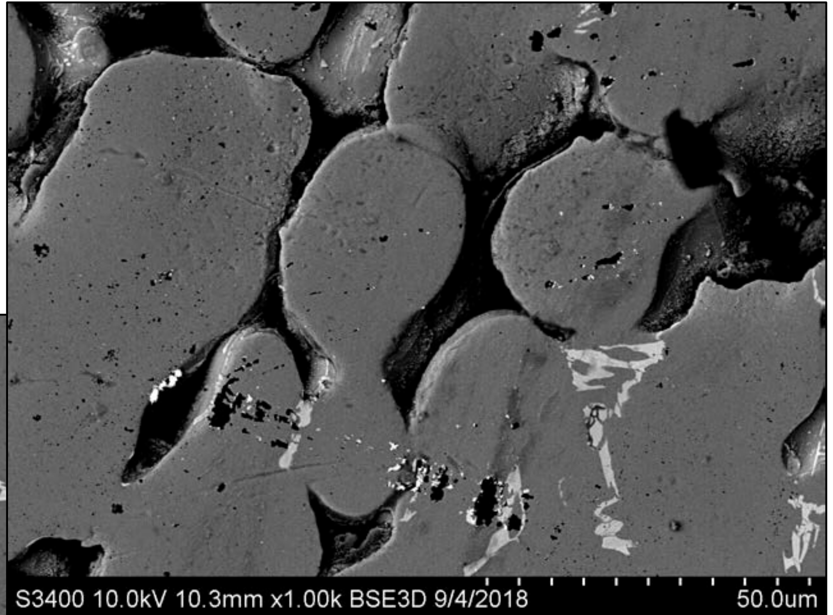


Topo Mode

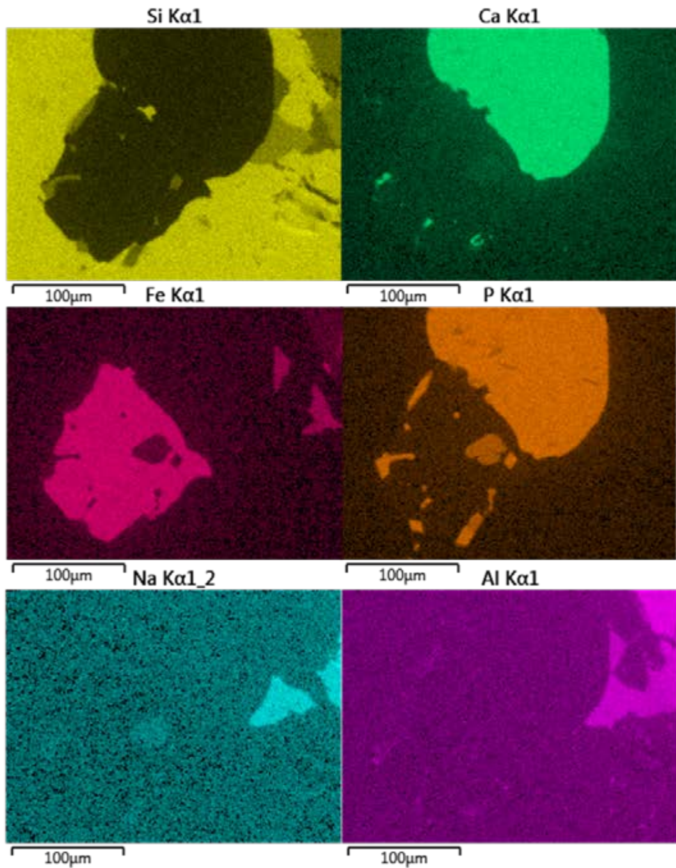


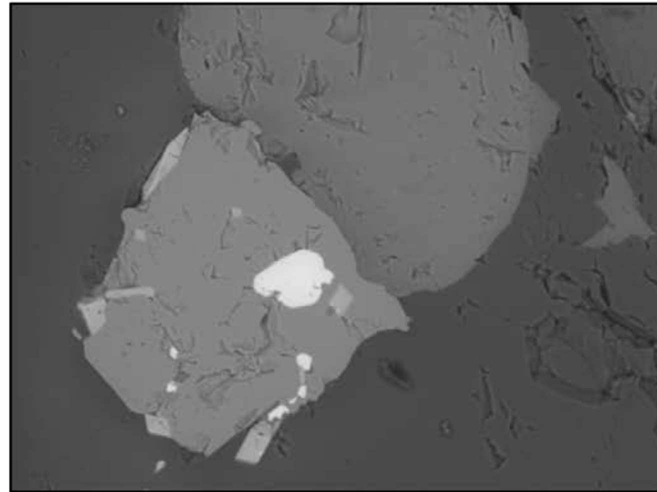

S3400 10.0kV 10.3mm x1.00k SE 9/4/2018
50.0um


S3400 10.0kV 10.3mm x1.00k BSE3D 9/4/2018
50.0um

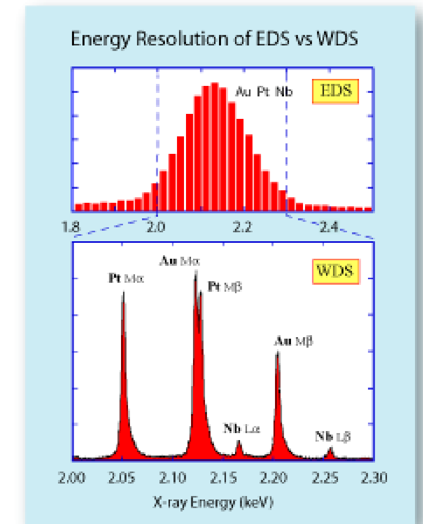

S3400 10.0kV 10.3mm x1.00k BSE3D 9/4/2018
50.0um

Microanalysis





- Wavelength-dispersive X-ray spectrometry (WDS)
  - Number of X-rays of a specific wavelength diffracted by a crystal.
  - One wavelength at a time



- Energy-dispersive X-ray spectrometry (EDS)
  - Number and energy of the X-rays emitted from a specimen
  - Chemical composition information

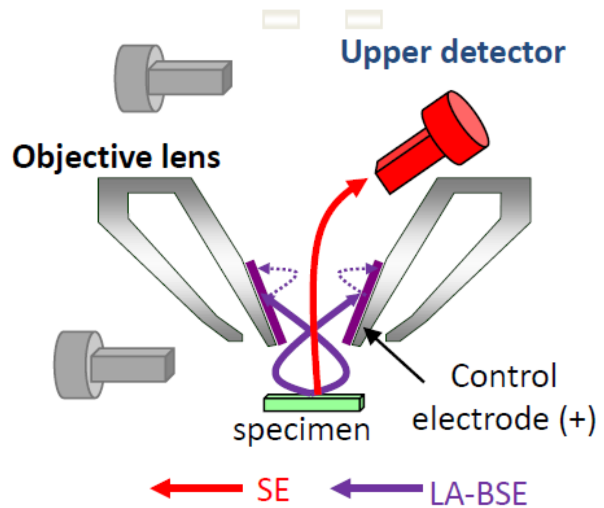
Hitachi S4800-II

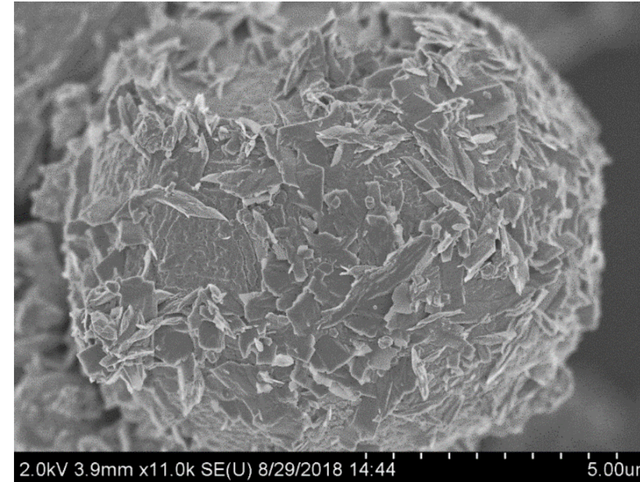
- cFEG
- Oxford EDS (Si(Li))
- E-T lower SE detector
- Upper SE/BSE
- Stage biasing for beam deceleration
- Leica Cryo Stage
- Applications: low kV imaging, cryo-SEM, general purpose high res imaging (~5-10 nm features)

Hitachi SU8030

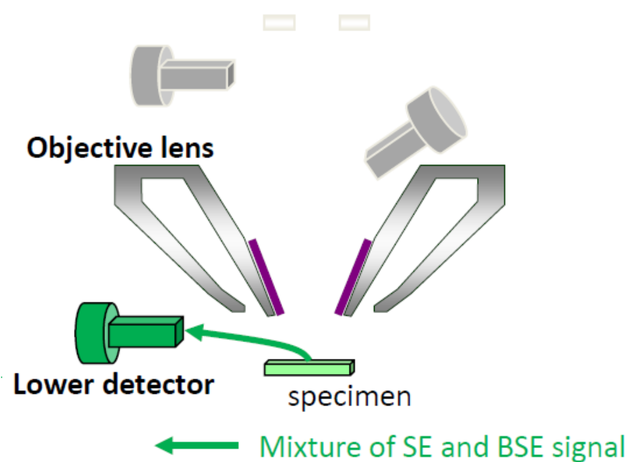
- cFEG
- Oxford SDD
- E-T lower SE detector
- Upper SE/BSE, Top detector
- Stage biasing for beam deceleration
- STEM detector
- Applications: low kV imaging, ultra-high res imaging (sub 5nm features), low kV EDS

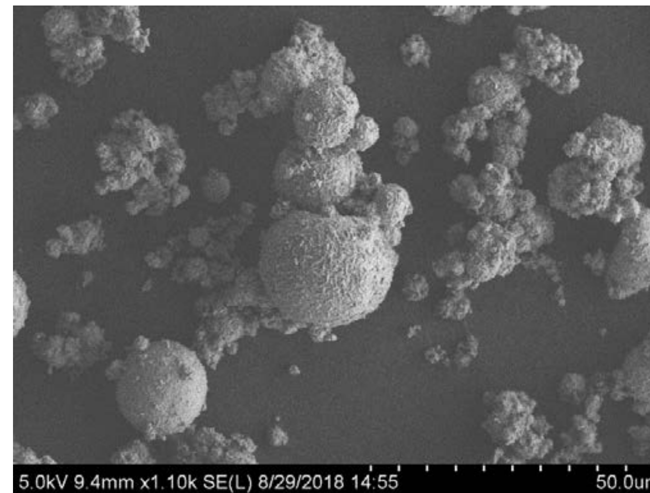
In-Lens Detector and Lower Detector



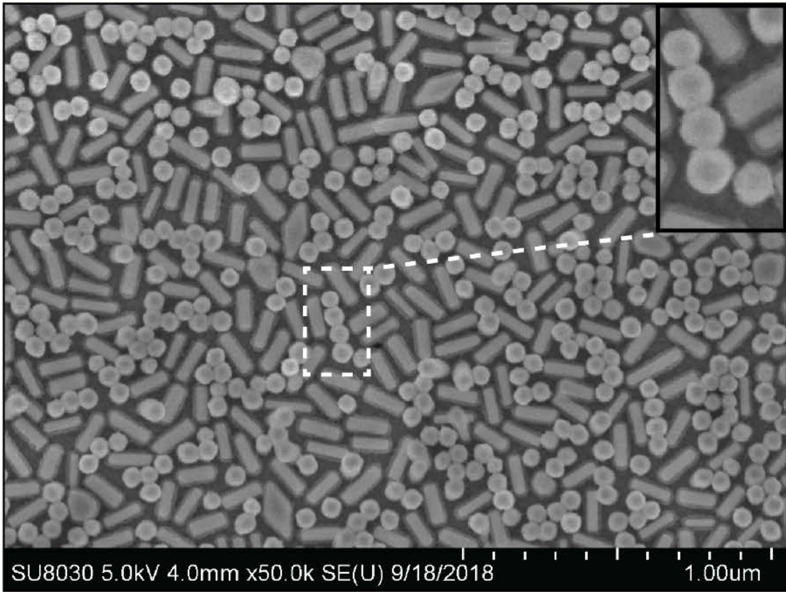


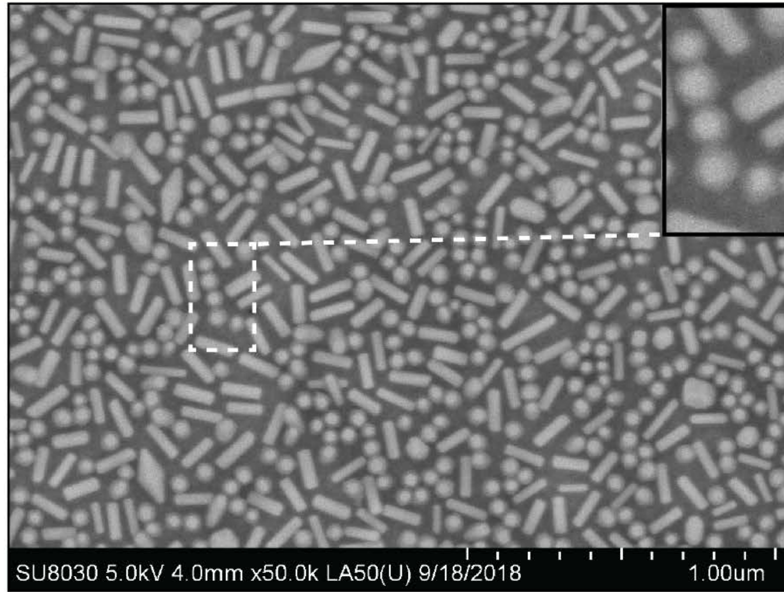
- SE detected with Upper through objective lens
- BSE not detected
- Rich topo info
- High resolution imaging

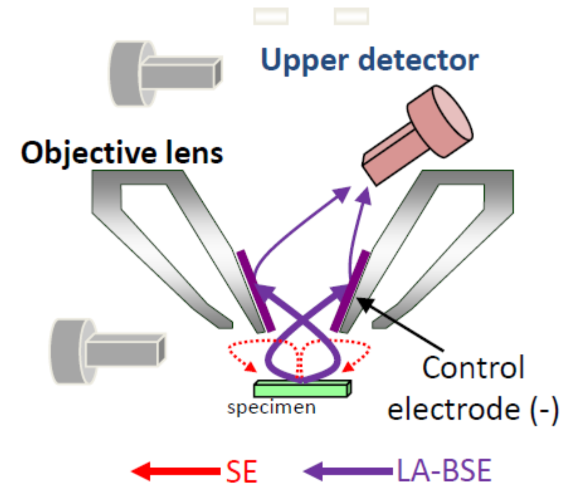


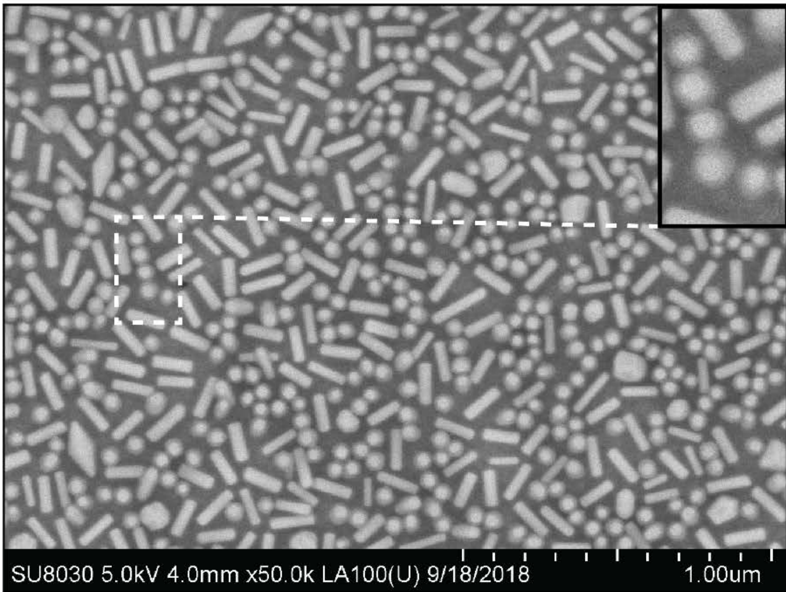


- Low angle BSE and SE are detected with lower detector
- Rich topo information
- Less sensitive to specimen charging artifacts


SU8030 5.0kV 4.0mm x50.0k SE(U) 9/18/2018
1.00um


SU8030 5.0kV 4.0mm x50.0k LA50(U) 9/18/2018
1.00um


Upper detector
Objective lens
Control electrode (-)
specimen
SE
LA-BSE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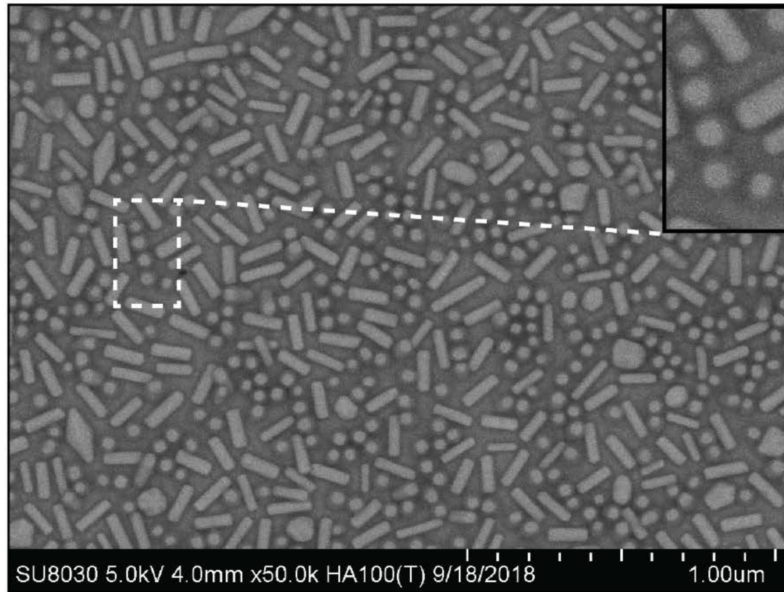
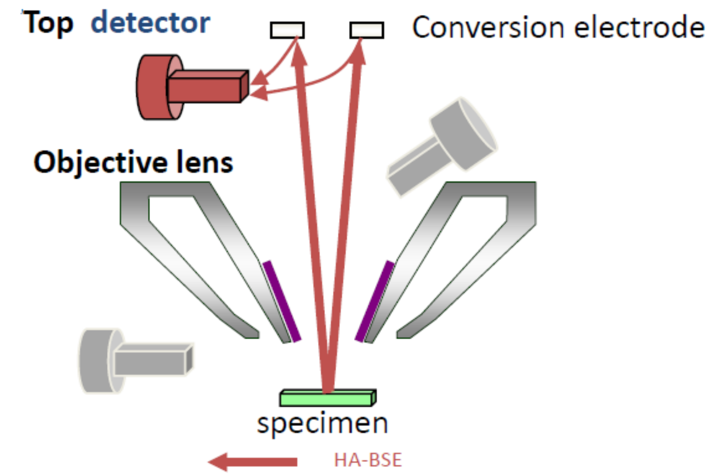
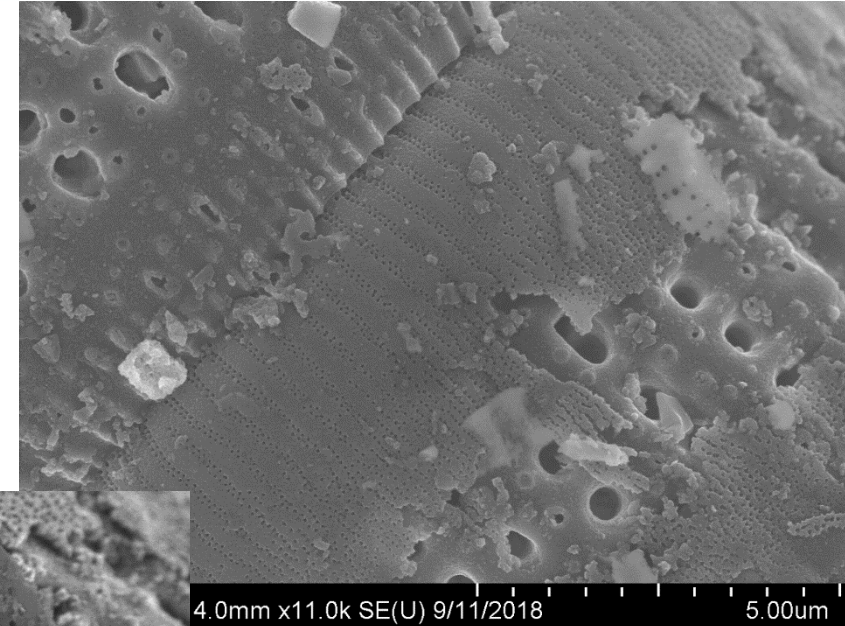
1.00um

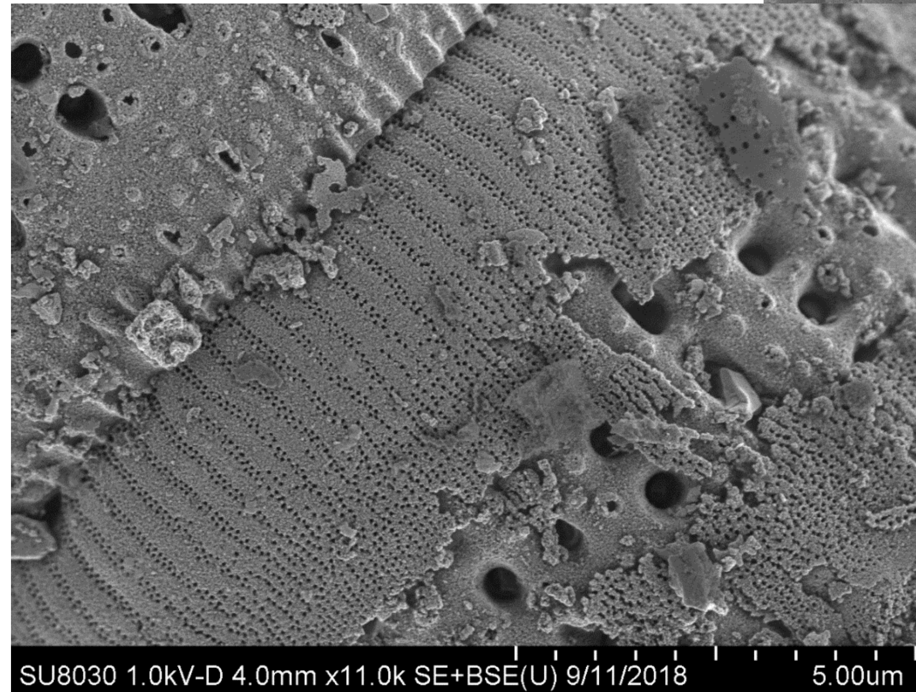

SU8030 5.0kV 4.0mm x50.0k HA100(T) 9/18/2018
1.00um


Top detector
Conversion electrode
Objective lens
specimen
HA-BSE

Beam Deceleration

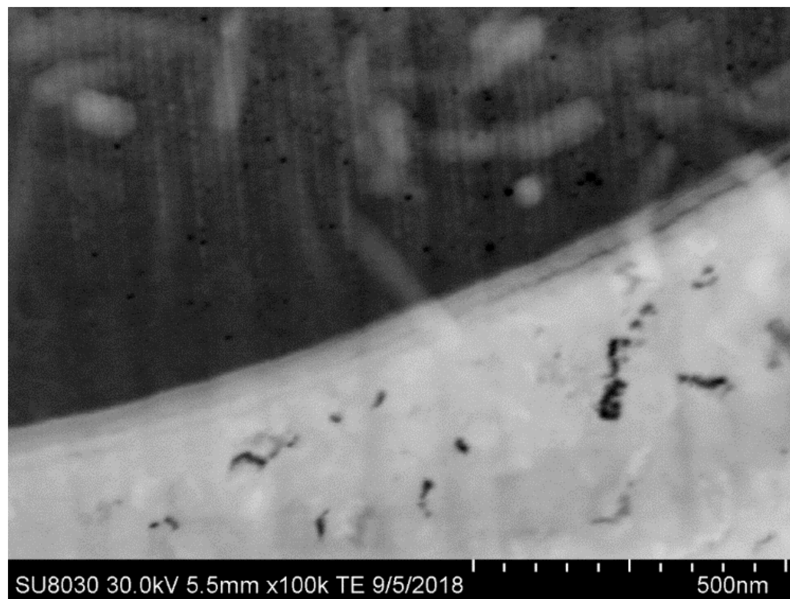
- Negative voltage bias on sample stage
- Energy of electrons in beam decreased before impact with specimen surface
- High resolution images at low energies
- Advantageous for reducing charging effects on non-conductive samples and beam-sensitive samples (biological samples)



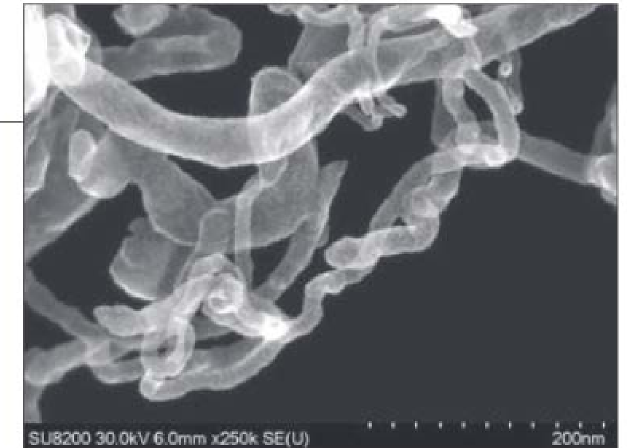


STEM – SU8030 Only

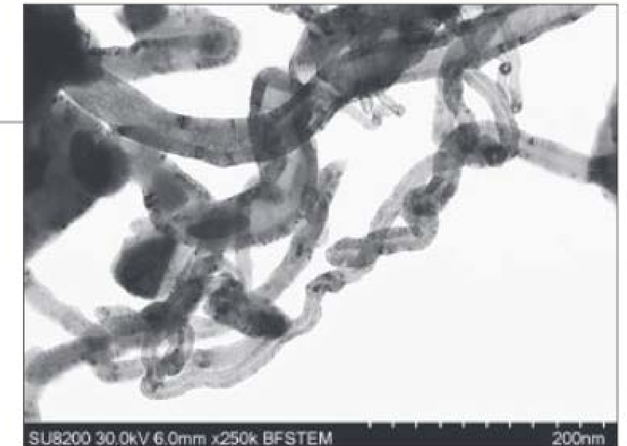
- Lower voltage (30kV) STEM allows higher contrast on low atomic number material



SE image
Surface information



BF-STEM image
Internal information



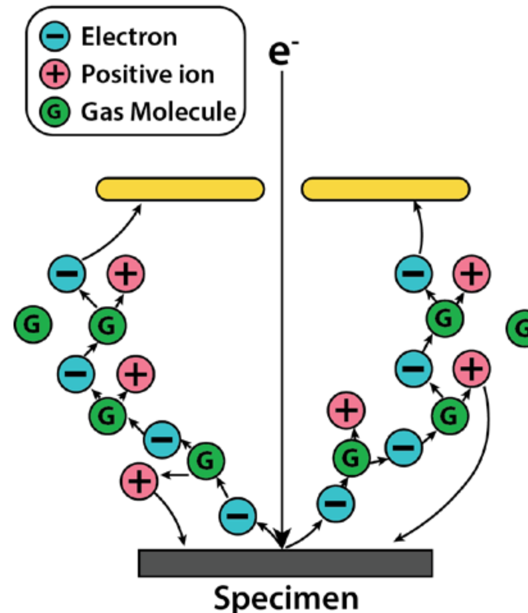
Sample : Carbon nanotubes Acceleration Voltage : 30 kV Magnification : 250,000×

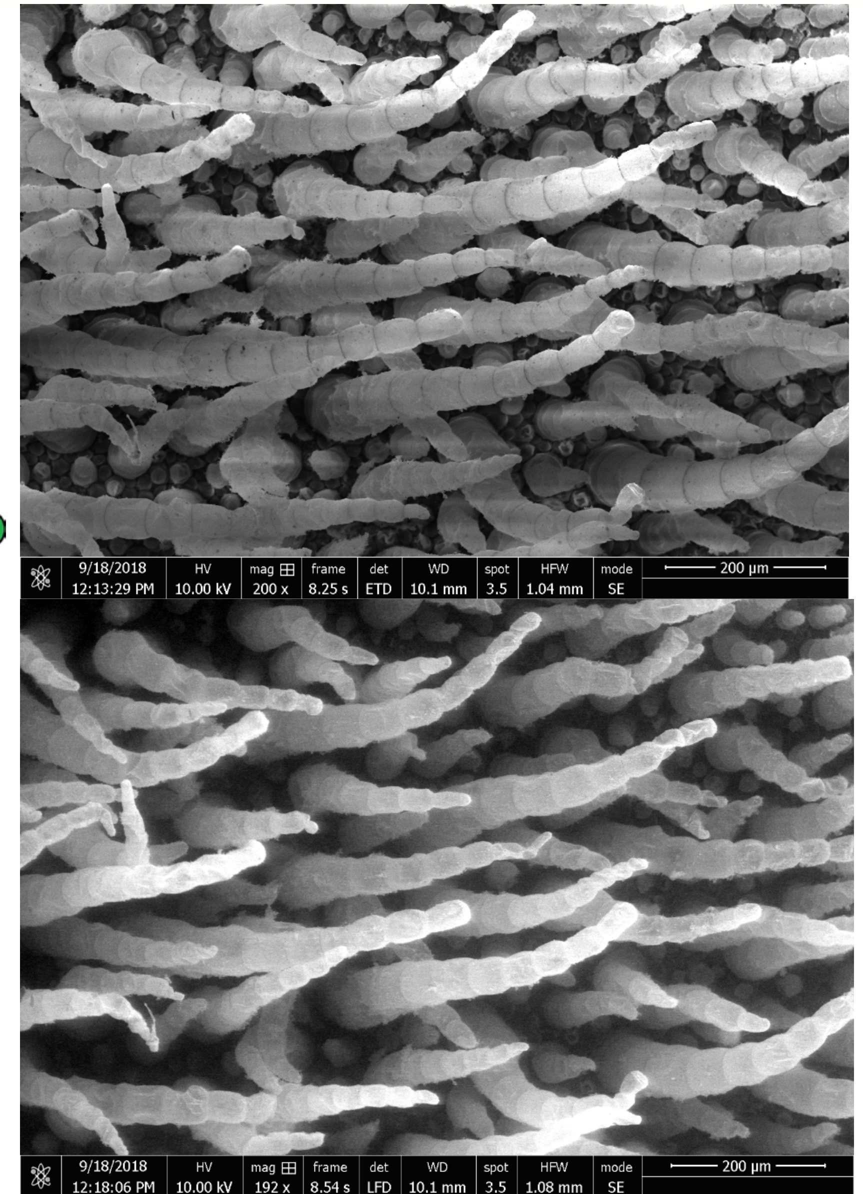
FEI Quanta 650 FEG

- Schottky FEG
- Low vac imaging
- ESEM (w/ Peltier stage)
- E-T SE, SS Directional BSE, gaseous SE
- NPGS lithography system
- Oxford EDS (Si(Li)) and EBSD
- Applications: EBL, EBSD, ESEM/VP-SEM, general purpose medium/high-res imaging (10 nm features)

Low-Vac Mode

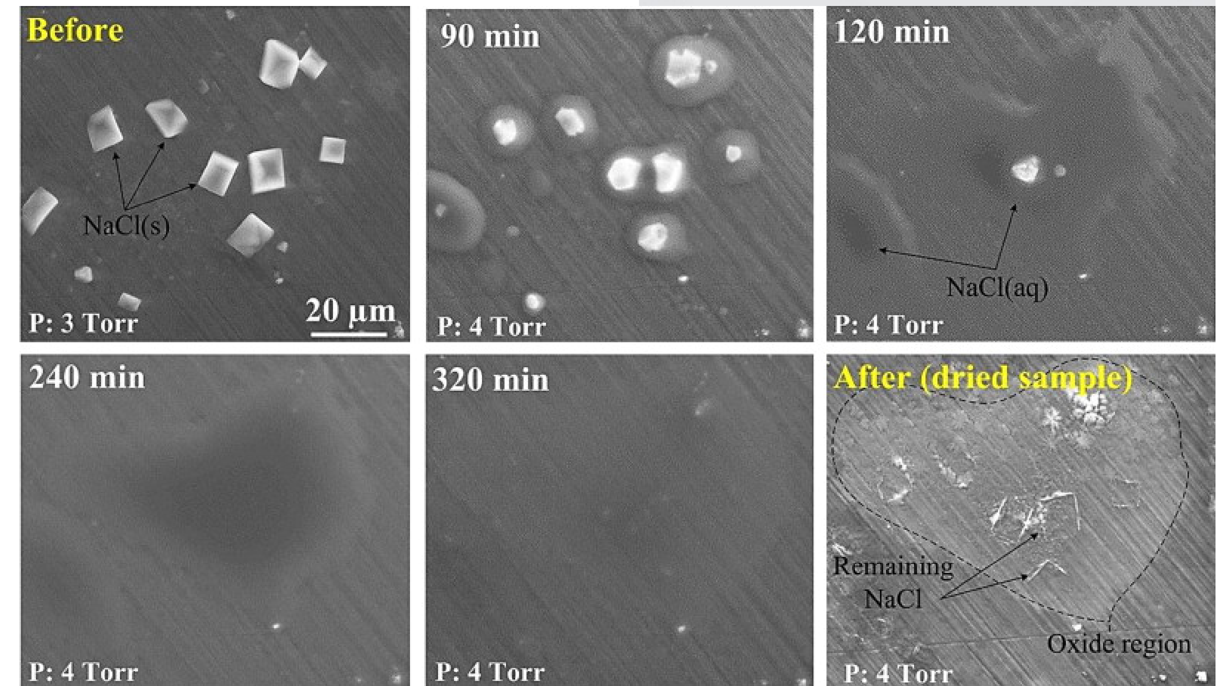
- LFD (Large-Field Detector)
  - Low-vac mode
  - 0-1 Torr
- GSED (Gaseous Secondary Electron Detector)
  - PLA – Higher chamber pressures
  - ESEM mode





ESEM (Peltier stage)

- GSED
- Wet samples
  - Sample cooling (1 °C)
  - Water vapor (4.7 Torr)
  - Humidity cycling experiments
- Dissolving and re-precipitating salt
- Condensing water on hydrophobic surfaces



Esmaily et al., 2015

BSE Imaging – Directional BSE

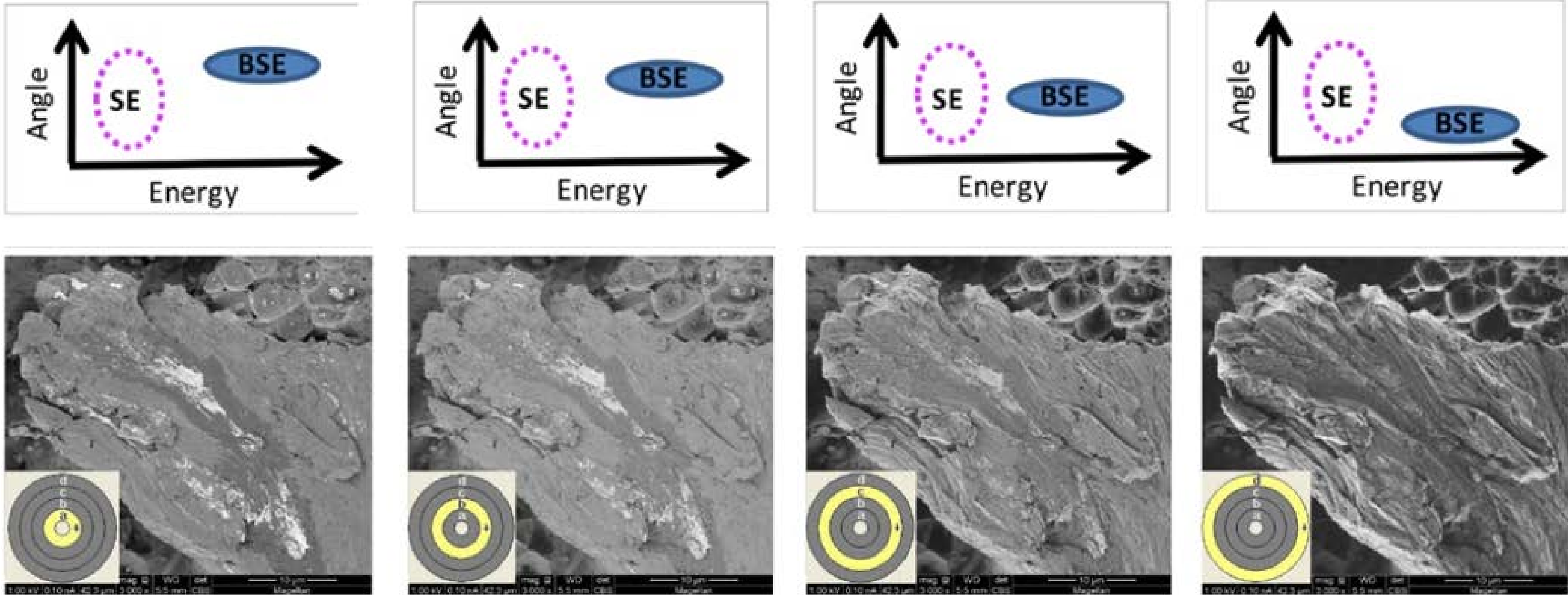


Figure 9: Simultaneously acquired BSE images of a fractured aluminum sample with inclusions acquired at 1 keV.

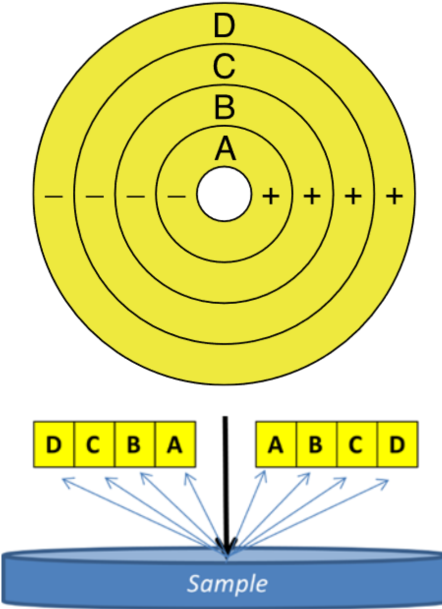
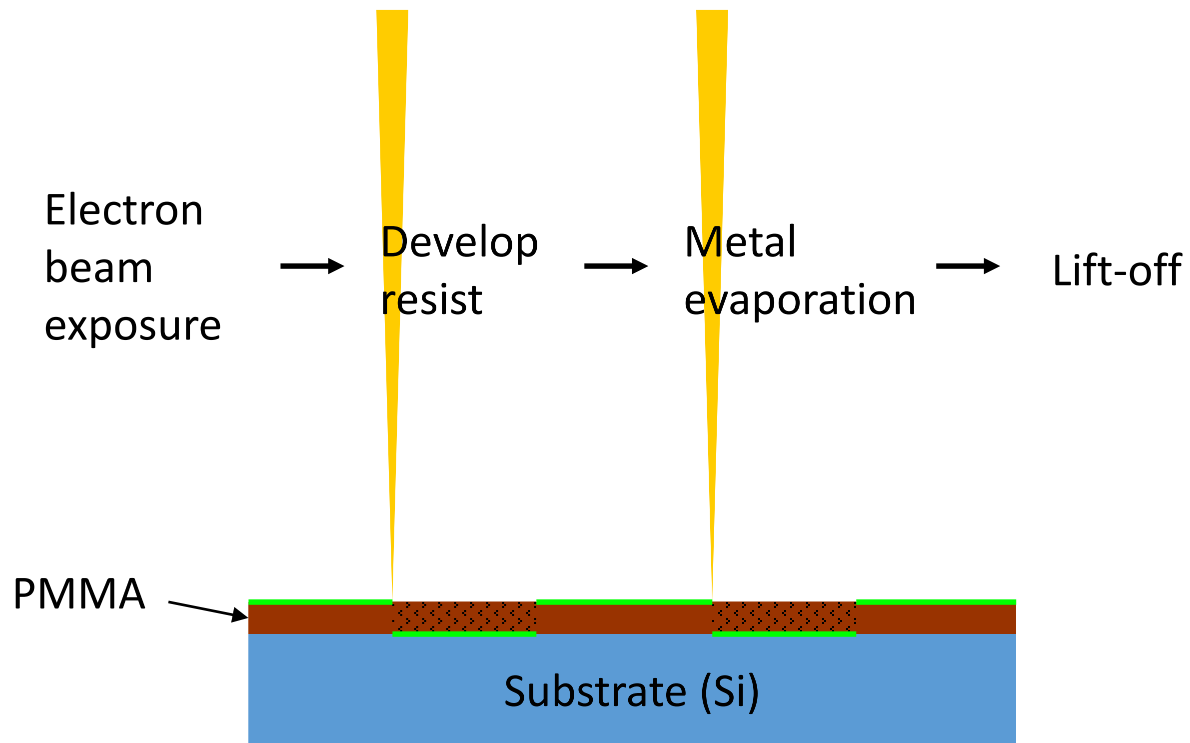
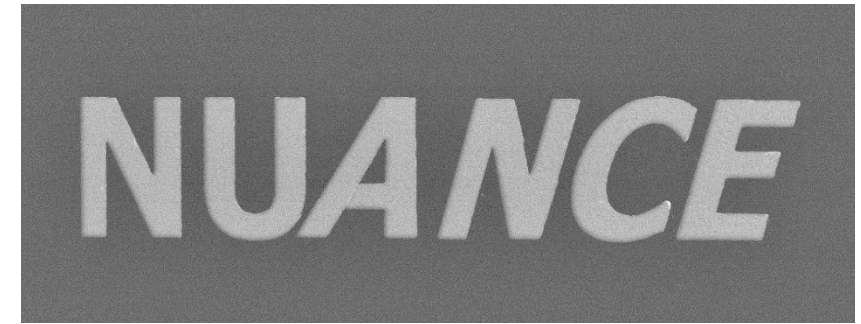
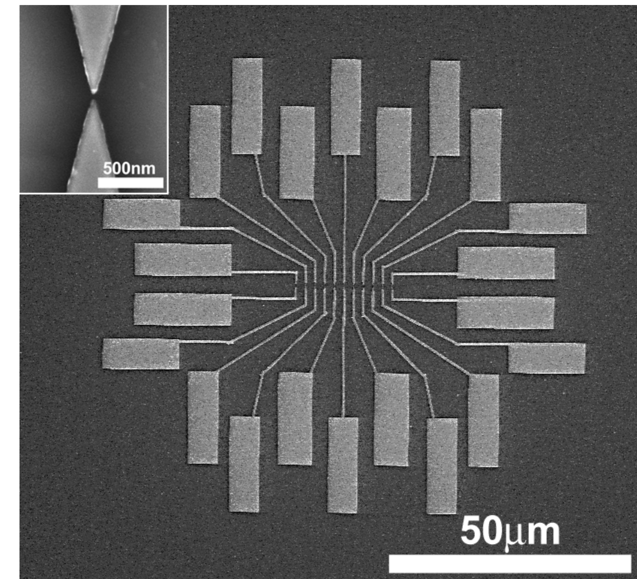


Figure 8: Schematic of angular separation of BSE detection and detector layout

Electron Beam Lithography

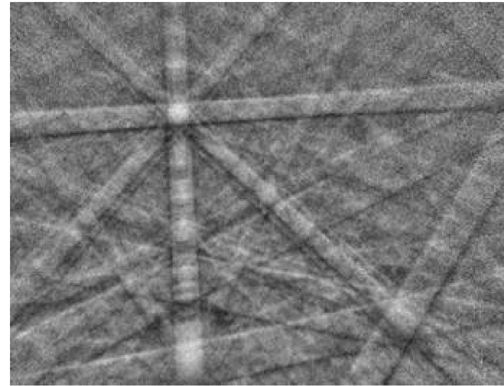


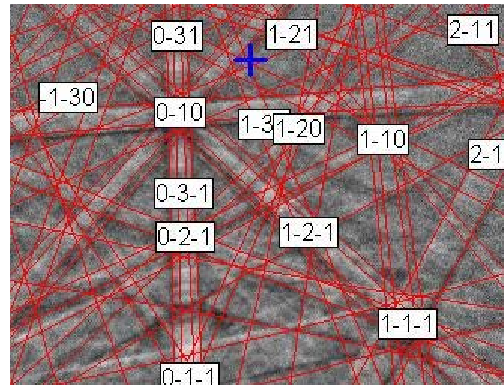


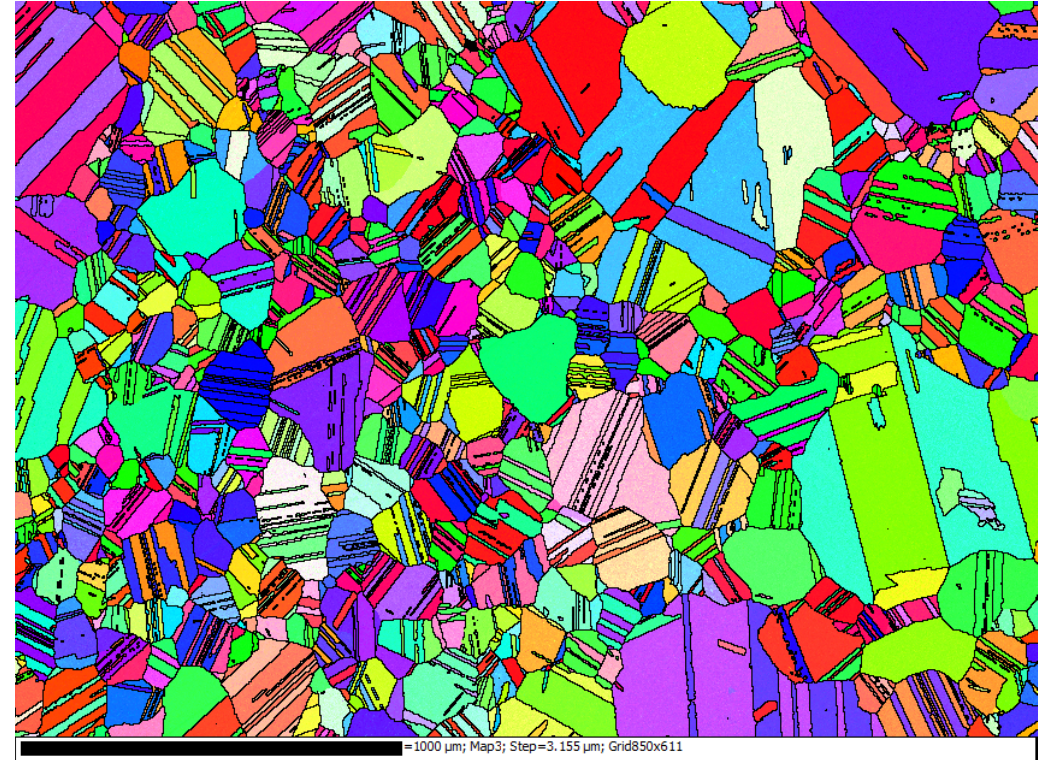


Microanalysis

- EDS/EBSD
- Electron Backscatter Diffraction
  - Used to understand structure, crystal orientation, phase differentiation, grain boundaries

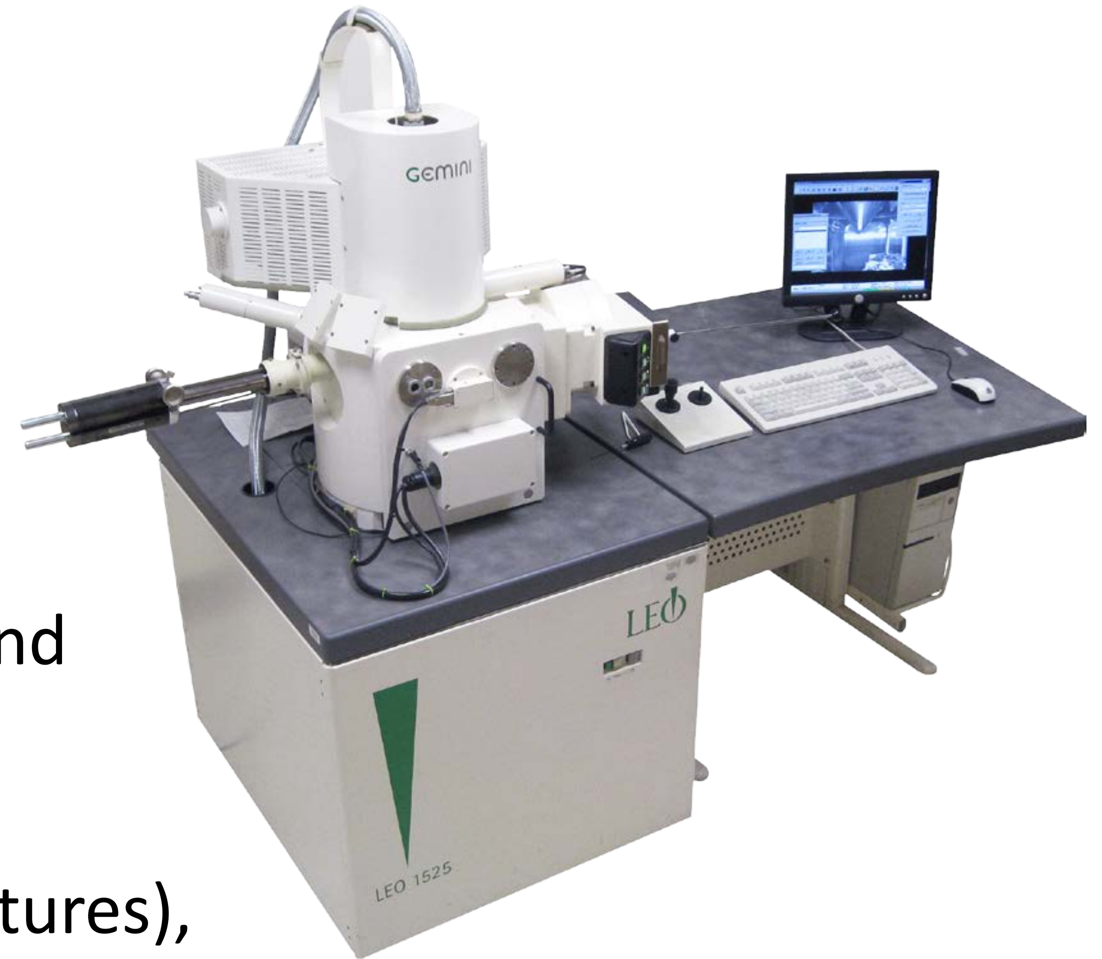




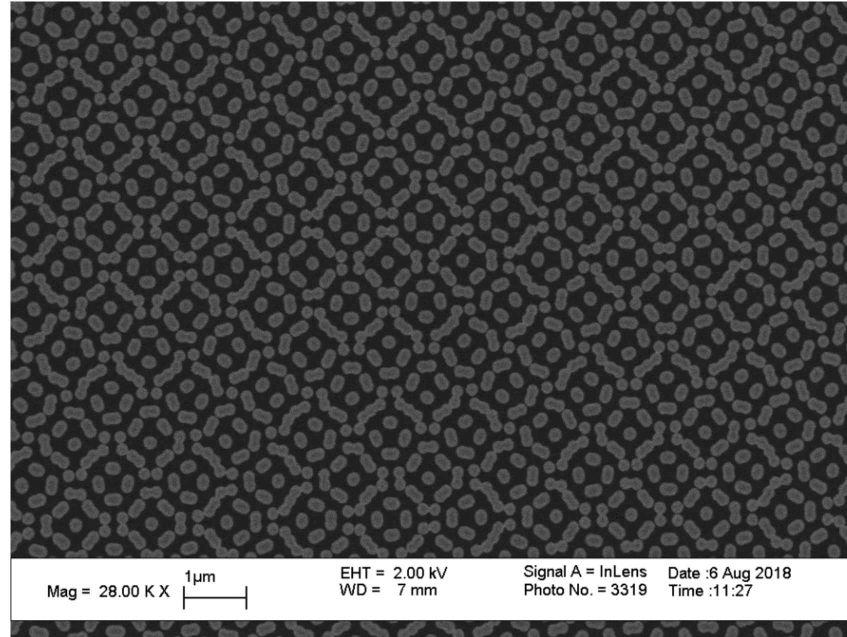


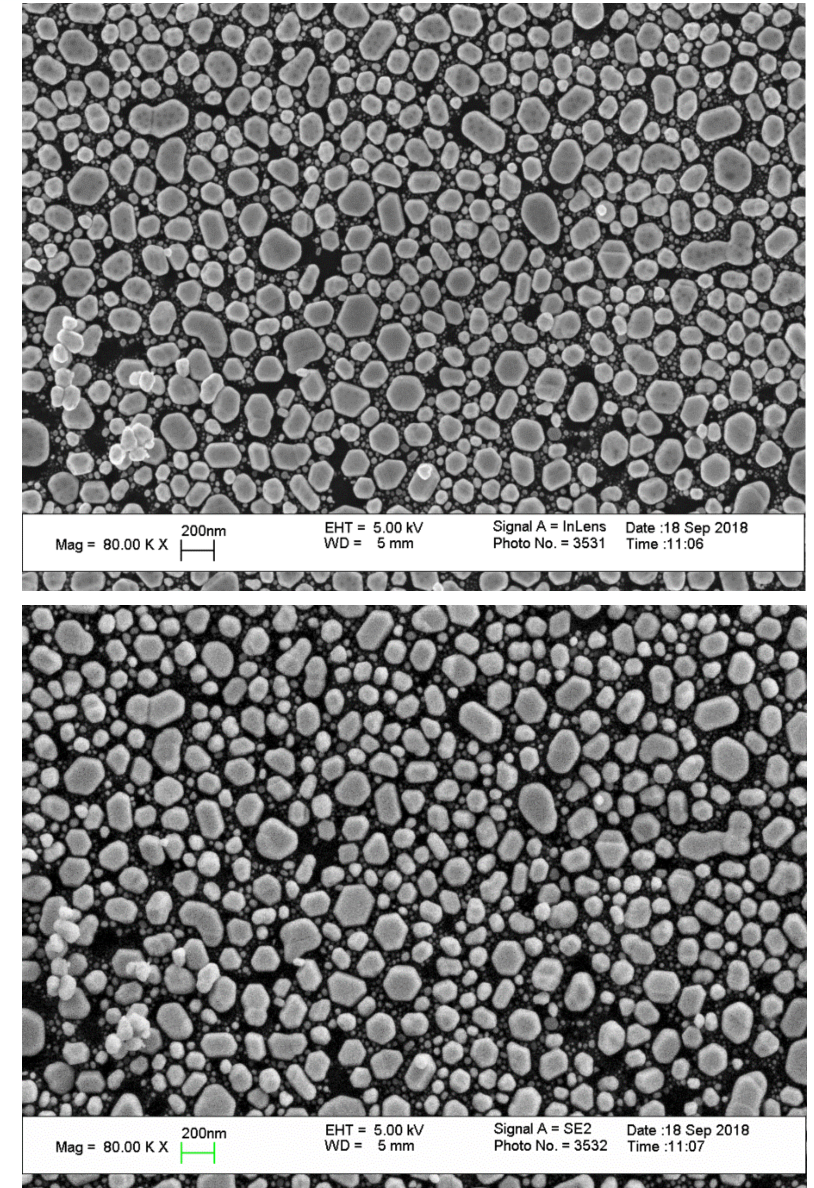
LEO Gemini 1525

- Schottky FEG
- Gemini column, Upper SE
- E-T SE and Robinson BSE
- Spatial resolution of 1.5nm at 20kV and 3.5nm at 1kV
- Applications: General purpose medium/high-res imaging (10 nm features), high tilt-range stage, magnetic samples



- High resolution imaging
    - Electrostatic lens alters landing voltage
- Magnetic samples
    - Electrostatic lens
- High tilt of sample stage/Large chamber
- Basic SEM imaging





Things to Consider

- Resolution
  - Size of the features of interest
- Type of Imaging
  - SE, BSE, STEM
- Type of Sample
  - Conductive, Non-conductive, beam sensitive
- Type of Analysis
  - EDS, EBSD, WDS, eBL

SEM Facility Instruments

Hitachi S3400N-II SEM

- Low mag imaging
- VP-SEM
- BSE detector
- EDS

Hitachi S4800-II cFEG SEM

- High Mag imaging
- BSE – SE filtering
- EDS

Hitachi SU8030

- High Mag imaging
- BSE – SE filtering
- EDS
- STEM imaging

LEO Gemini 1525 SEM

- High Mag imaging
- BSE

FEI Quanta 650 ESEM

- Basic SEM imaging
- ESEM/Low Vac Mode
- EBSD/eBL/EDS
- Directional BSE detector

NUcore

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