10. advanced SEM

**SEM**
- Low voltage SE imaging
- Condition of the surface, coatings, plasma cleaning
- Low voltage BSE imaging
- Polishing for BSE, EDX and EBSD, effect of ion beam etching/polishing

*Figure: Tension d'accélération (kV) vs. Resolution (nm)*

- Low voltage, high resolution:
  - Observation of the "real" surface
  - Non-metallic samples
  - Reduced beam damage
- High voltage, high resolution:
  - Edge effects
  - Charging effects
  - Beam damage

**Latest generation of SEM**

Field emission gun
monochromators
beam boosters, beam deceleration,
Lens-design: In-lens, Semi-in-lens, immersion lens
Short working distance

Detectors:
Everhard-Thornley (SE)
In-column (through the lens, in-lens, "in-beam")
Low Voltage BSE detection,
energy filtering (separation of materials and topography contrast)

Analytical SEM:
SDD EDX detectors (high throughput, large collection angle)
High-speed EBSD detectors
Beam currents of several 100 nA
No specimen preparation needed: Low kV imaging of non-conducting, low density samples

Al₂O₃ Nano-crystals

FEI Magellan
Operator: Ingo Gestmann
Samples: Marco Cantoni

Carbon nano-tubes (MWCT)

FEI Magellan
Operator: Ingo Gestmann
Samples: Marco Cantoni
SEM low kV imaging

No specimen preparation needed:
Low kV imaging of non-conducting samples

Liquid filled organic membranes

Zeiss Nvision 40
Marco Cantoni

MSE-636 2014 advanced SEM Marco Cantoni
SEM low kV imaging

Purely organic specimen: non-conductive, low density:
Metal coating

HeLa Cells, Graham Knott
Marco Cantoni, Nvision 40

SEM low kV imaging

“Easy” sample:

SC wire
Nb$_3$Sn in Cu matrix
SEM low kV imaging

"Easy" samples:

Everhard-Thornley detector (SE)

Solid state BSE detector

SEM low kV BSE imaging

2keV, In-Column EsB detector

Solid state BSE detector
SEM low kV imaging

“Easy” samples:
Contamination by hydro-carbons

- contamination “spoils” imaging at low kV
- How to avoid (at CIME):
  - plasma cleaning of the sample before inserting
  - Plasma clean the chamber at each insertion (multi-user environment)
**SEM: Preparation for analytical EM**

**Nb\textsubscript{3}Sn multifilament superconducting cable**

\textit{Nb\textsubscript{3}Sn superconductor multifilament cable: 14'000 Nb\textsubscript{3}Sn filaments (diameter ~5um) in bronze matrix}

Solid State BSE detector
20kV acceleration voltage

EDX maps

Sn
Cu
Nb

**Mechanical polishing <-> Ar ion beam polished**

**SEM BSE/EDX/EBSD**

Mechanical polishing:
- Grains of harder phase incorporated in softer matrix
- Deformed microstructure at surface reduces formation of Kikichi lines in EBSD

After Ar ion polishing (Gatan PIPS)
Ion polishing

in-chamber
ET-detector
SE

in-column
"InLens"
SE-detector

in-column, "energy-selective" EsB
BSE detector

SEM low kV BSE imaging

Nb$_3$Sn multifilament Superconductors
Materials & orientation contrast

NVision 40
1.8kV EsB detector
FIB/SEM low kV BSE imaging

**Nb$_3$Sn multifilament Superconductors**

**Materials & orientation contrast**

Interaction volume

**Blue:** scattered electrons

**Red:** backscattered electrons

(leaving the sample surface)

**Goal of final polishing:**

Removal of the damaged surface layer

mechano-chemical polishing (EBSD) or ion polishing

**escape depth of BSE:** Nb$_3$Sn

- 30kV: 800nm
- 5kV: 50nm
- 1kV: < 5nm

Monte-Carlo Simulation CASINO v2.42
New HR-SEM at CIME

• Starting point: XL-30 SFEG SIRION (since 2001)
• First semi-in-lens HR-SEM:
  in-lens (through the lens) detection of SE and BSE

Resolution in UHR mode: 1.5 nm at 10 kV (or higher); 2.5 nm at 1 kV
Best “SEM” at EPFL: FIB
Nvision@CIME
Two different contrasts with one scan: parallel detectors

**Specifications**

**SEI Resolution**
- 1.0mm guaranteed at 15kV
- 1.5nm guaranteed at 1kV, in GB mode
- 2.5nm guaranteed at 1kV, in SEM mode
- 0.8nm guaranteed at 30kV, in STEM mode
- 3.0nm guaranteed at 15kV, 5 nm, 8.5mm WD

**Magnification**
- Low: 25x to 19,000x
- High: 100x to 1,000,000x at 4x5 photo size

**Maximum Sample Size**
- 100mm diameter, 50mm (H) or 290mm diameter, 20mm (H)

**Accelerating Voltage**
- 0.1 to 30kV

**Probe Current Range**
- 1 pA to ≥ 200 nA

**Standard Detectors**
- In-lens (SEI) secondary-electron detector
- Below-the-lens (LEI) secondary-electron detector
- Low angle backscatter electron detector (LaBE)

**Optional Detectors**
- Retractable in-lens BSE detector
- Retractable solid-state or PMT-type STEM
- In-column Faraday cup (PFD)
**GENTLE BEAM (GB)**

- Semi-in Lens Objective Lens
- Objective Lens Pole Place
- r-Filter

**Gentle Beam Mode**

- Gun kV – GB Bias V = kV @ sample

**Reduces the effect of charging**

**Provides ultralow kV**

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**Electron beam resolution**
- Site survey required to determine attainable resolution
- Resolution @ optimum WD
  - 0.8 nm at 15 kV
  - 0.8 nm at 2 kV
  - 0.9 nm at 1 kV
  - 1.5 nm at 200 V
- Resolution @ coincident point
  - 0.8 nm at 15 kV
  - 0.9 nm at 5 kV
  - 1.2 nm at 1 kV

**Maximum horizontal field width**
- E-beam: 1.5 mm at beam coincident point (WD 4 mm)

**Landing energy range**
- 50 V – 30 kV

**Probe current**
- E-beam: 1 pA to 22 nA
Introducing...
MERLIN™
– Analytical power for the sub-nanometer world –

MERLIN™
– Analytical power for the sub-nanometer world –

Gemini II column
Low current configuration (Max. probe current 40 nA):
For high resolution investigations:
+ 0.6 nm at 30 kV (STEM mode)
+ 0.8 nm at 15 kV at optimal WD
+ 1.4 nm at 1 kV at optimal WD
+ 2.4 nm at 0.2 kV at optimal WD
+ 3.0 nm at 20 kV at 10 nA @ WD = 8.5 mm
MERLIN™
- Analytical power for the sub-nanometer world -

- High stability field emitter cathode
- Maximum probe current 300 nA
- Beam Booster
- Brightness of the electron probe maintained for low landing energies
- Energy selective Backscatter detector (EsB)
- In-lens Secondary Electron detector
- GEMINI® II final lens
- Proven GEMINI® final lens design
- New double condenser lens for highest probe current possibilities (300 nA)
- Beam booster technology maintains brightness of all electron probes including low landing energies
- True on-axis in-lens SE and BSE detectors
MERLIN™
- Analytical power for the sub-nanometer world -

In-lens SE (Secondary Electron detector)

Topographical information with on-axis in-lens SE detector

Complete detection system:
- Unique double in-lens detection
- Acquisition of pure secondary and backscatter electron signals
- Separation of compositional, topographical and crystalline surface information

MERLIN™ II design

Complete detection system:
- Unique double in-lens detection
- Acquisition of pure secondary and backscatter electron signals
- Separation of compositional, topographical and crystalline surface information

Energy filtering grid

EsB (Energy selective Backscatter detector)

Compositional contrast with on-axis in column EsB detector

Complete detection system:
- Unique double in-lens detection
- Acquisition of pure secondary and backscatter electron signals
- Separation of compositional, topographical and crystalline surface information