Shielded FIB-FE-SEM: Evaluation, technical modification and implementation

HOTLAB 2017 - Annual meeting on Hot Laboratories and Remote Handling
September 17-22, 2017, Mito, Japan
Outline

- Introduction
- Instrument
  - FE-SEM
  - FIB
  - Additional Equipment
  - Analysis
- Nuclearization – Adaptation for use with radio-active Materials
  - Shielding: Containment & Glovebox
  - Loading System
  - Internal Shielding
  - Modifications for Maintenance
- Time Schedule
- Planned Field of Application
- Summary
Introduction – a Brief history

- SEM in PSI Hot-Laboratory since more than 40 years
- Cambridge S4-10 until 1991
- 1992: Purchase of the DSM 962 from Zeiss including Pionier Si(Li) Detector & Voyager EDS Analysis System from Tracor-Noran
- EDS Software replaced with Thermo-Noran System SIX in 2002
- Spare parts difficult to find and Maintenance therefore expensive
- 2010 decision to replace with a shielded SEM/FIB
- 2015 Order placed, 2017 implementation
Zeiss Crossbeam XB 540
- Gemini II FE column
- 4 detectors for imaging
- Large Chamber
- Capella ion gun
- Gas injection system
- Plasma cleaner
- Micromanipulator

EDAX TEAM Pegasus 3D analysis system
- “Octane Super” SDD
- “TEXS WDS” spectrometer
- “DigiView 5” EBSD-Camera
FE-SEM: Chamber, Stage & Specification

**Column:**  
Gemini II Shottky Field Emitter  
Long source lifetime of 15’000h – 20’000h  
0.02 – 30 kV, high current up to 300 nA  
M: 17x – 1’000’000x

**Resolution:**  
2 nm at 1 kV, 1 nm at 30 kV

**Chamber:**  
Large (inner diameter = 330mm, height = 270mm)  
18 ports, 2 IR cameras

**Stage:**  
High precise super-eucentric  
X, Y: 100 mm; Z: 50 mm; R: 360° continuous  
T: -4° to 70°; M: 13 mm  
all 6 axes motorized; sample weight up to 0.5 kg
<table>
<thead>
<tr>
<th>Detector Type</th>
<th>Characteristics</th>
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<tbody>
<tr>
<td>SESI Scintillator photomultiplier system (similar to ET)</td>
<td>Imaging with Secondary Electrons and Secondary Ions</td>
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<tr>
<td>YAG BSE Crytur Rebeka™ YAG(^1) BSE-detector, retractable</td>
<td>Material contrast, excellent S/N ratio, Sensitive in low energy (down to 200 eV)</td>
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<tr>
<td>In-Lens SE High spatial resolution</td>
<td></td>
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<tr>
<td>In-Lens EsB Energy specific separation of BSE in low energy</td>
<td>Nano-scale compositional information, high spatial resolution, Sub-surface information visible, Not sensitive to charging, less sensitive to edge contrast</td>
</tr>
</tbody>
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\(^1\)Yttrium-Aluminium-Garnet
<table>
<thead>
<tr>
<th>Equipment</th>
<th>Description</th>
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<tbody>
<tr>
<td>Capella FIB</td>
<td>Gallium liquid-metal Ion-Source</td>
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<tr>
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<td>Lifetime up to 3000 μAh, high current stability</td>
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<td></td>
<td>0.5 kV – 30 kV, 1 pA – 100 nA</td>
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<td>300x – 500kx, 3 nm resolution (30 kV)</td>
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<td>Multi-GIS</td>
<td>Multiple injector needle system (4)</td>
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<td>C and Pt</td>
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<td>integrated charge compensation, dry N$_2$-injection</td>
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<tr>
<td>Plasma Source</td>
<td>Sample cleaning (low voltage imaging)</td>
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<td>Vacuum cleaning</td>
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<td>Micromanipulator</td>
<td>Autoprobe Model 200.2 from Omniprobe</td>
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<td>3 axes positioning stage, 100 nm step size</td>
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EDS

“Octane Super” Silicon Drift Detector (SDD), LN-free
Crystal active area 60 mm²
Energy resolution 126 eV (Mn)
Stable energy resolution at high collection speeds
High count rates allow fast mappings
TEAM Software

WDS

TEXS WDS spectrometer with capillary optics for maximum efficiency in low energy and superior low energy resolution (< 40 eV)
5 diffractors allow analysis of almost complete periodic table (≥ Be)
Integrated in TEAM Software
EBSD

DigiView EBSD Camera
Foreward BSE detector
Fully integrated into TEAM-Software

TEAM™

One software for three analysis systems
EDS spectrum analyses, multipoint analyses, linescans, phase mapping, qualitative and quantitative element mapping, drift correction
WDS full spectrum energy scan, qualitative & quantitative analyses
EBSD orientation & phase analyses
3D option for EDS & EBSD
Technical constructions and modifications with the goal to...

- protect human health & life
- protect instrument from fast ageing
- allow a safe and clean operation and maintenance
- transfer radioactive material
- load and unload active and/or contaminated samples

Requirements:

- limited floor load → affect dimensions of hot cell
- available space → affect access to instrument (sample material, maintenance)
- dividing the instrument → elongation of cables and tubing's affect signal quality, vacuum quality and pumping time, gas pressures
- access to all parts of instrument → possibility to move the instrument and replace it on exact position
The goals with the requirements are realized with:

- optimized hot cell and integrated glove box
- material and sample transfer ports
- protection of sensitive detectors with intern shutters
- new constructed sample loading system
- attachable maintenance box
- instrument mounted on wheels to move on a guide rail
- Limitation of floor load
- Hotcell as **large as necessary** and as **small as possible**
- 10 cm Lead walls $\rightarrow$ 17 tons
- 20 cm steel door $\rightarrow$ 7 tons
- 30 cm lead glass window $\rightarrow$ 336kg
- 5 gloves, 1 tele-manipulator
- handling of high-active and contaminated sample material
- double cover lock, La Calhène lock (for material transfer)
Nuclearization – Material & sample transfer

Transport flask - Double Cover Lock
Nuclearization – Material & sample transfer

La Calhène - Double Cover Lock
Nuclearization – Sample loading system
Detector inserting opens shutter → ready for analysis
Retracting closes the shutter → Detector protected
Detector inserting opens shutter → ready for analysis
Retracting closes the shutter → Detector protected
Nuclearization – Guiding rail

- Instrument on wheels
- Move on a guiding rail
- Exact re-positioning after move
- Necessary for sample transfer system
Nuclearization – Maintenance Box

- Protection by open front door of the potentially contaminated sample chamber
- Provide cleaning work and maintenance in sample chamber
- Tightly attachable to instrument
- Equipable with 4 gloves
- Good compromise for access, visibility and protection
Schedule Start-Up

- September 2017: installation & last modifications finished
- October 2017: start-up & training
- 1st quarter 2018: inactive operation & testing
- 2nd quarter 2018: low-active materials testing
- 3rd quarter 2018: definitive start of operation
Sample preparation for...

- ... TEM
- ... intern and extern beamlines (e.g. SLS, Swiss-FEL, μ-XAS...)

... from different materials like...

- ... cladding material
- ... fresh/irradiated fuel
- ... other active or inactive material

Analyses of fresh prepared surfaces for ...

- ... crystalline structures
- ... strain orientation
- ... size and composition of SPP
- ...
A complex project like this can only be realized ...

... if the goals and needs are clearly defined in advance.
... if everything is carefully prepared.
... with an excellent collaboration between supplier, manufacturer, engineers and customer.
Therefore my thanks go to ...

• Gloor Instruments
• Défi Systems
• Zeiss, EDAX and Oxford Instruments

... for the excellent collaboration and support...

... and of course to the helpful staff of the PSI Hot-laboratory