Focused Ion Beam (FIB) – its principles and applications for materials science

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Outline

1. Motivation to present FIB-SEM at CERN.
2. Dual beam machine
   2.1 Scanning electron microscope (FE-SEM)
   2.2 Focus Ion Beam
3. Application overview by FIB-SEM and TEM
   Characterization of Nb Coating in HIE-ISOLDE QWR Superconducting Accelerating Cavities.
4. Summary
1. Motivation to present FIB-SEM at CERN.

• Evolution of materials fabrication process → complex structure of today/future materials.

• Demand for advanced analysis → TE-VSC-VSM, TE-MSC-SCD and EN-MME.

• CERN electron microscopy equipment – standard tools on the market.

• Proposition to use the latest generation of equipment and collaboration with EPFL → the research institutes and services centers are equipped with dedicated SEM’s, FIB-SEM and TEM’s over the decade.
2. Dual beam machine FIB-SEM

SEM → Imaging and Analysis

• Secondary electron (SE), imaging, topography contrast
• Back scattered electrons (BSE), chemical contrast
• Low voltage → High resolution SE and BSE imaging
• Energy Dispersive Spectroscopy (EDS); point analysis and elemental mapping
2. Dual beam machine

FIB $\rightarrow$ Nano - machining

- Machining (sputtering $\sim$ milling)
- Chemically assisted deposition and etching (gas injection system)
- Ion beam induced imaging $\rightarrow$ SE and SI
- Micromanipulation of small objects
2. **FIB-SEM → nano laboratory**

Sample is at 52-55° tilt at coincidence point.
2.1 SEM

- Probe size vs interaction volume $\rightarrow$ resolution
- Imaging using several different signal $\rightarrow$ needed information

<table>
<thead>
<tr>
<th>Signal</th>
<th>Use</th>
<th>Typical resolution</th>
<th>Detector</th>
</tr>
</thead>
<tbody>
<tr>
<td>SE</td>
<td>Best surface sensibility</td>
<td>1-3 nm</td>
<td>In –column SE</td>
</tr>
<tr>
<td>BSE</td>
<td>Z-contrast</td>
<td>15 nm</td>
<td>In column BSE</td>
</tr>
<tr>
<td>BSE</td>
<td>Crystallographic information</td>
<td>15 nm</td>
<td>Electron Backscatter Diffraction Analysis (EBSD)</td>
</tr>
<tr>
<td>X-Ray</td>
<td>Semi-quantitative chemical</td>
<td>500 nm</td>
<td>Energy Dispersive Spectroscopy (EDS)</td>
</tr>
</tbody>
</table>
2.1 SEM – In–column detectors

In chamber SE detector

In-column SE-detector

In-column BSE-detector

5 kV, RSA 905, sample courtesy F. Leaux, C. Garion

In-column SE

In-column BSE
2.2 Focus Ion Beam

- Mainly developed in 1970’s and 80’s
  (Escovitz, Levi-Setti, Orloff, Swanson…)
- Used mostly by semiconductor industry, materials science and increasingly in the biological field
- Source: Liquid Metal Ion Source (LMIS)

Why Ga+?
→ Liquid around room temperature
→ Ionization and field emission
→ Atomic number 31 – low analytical interference
→ Long life of the Ga source (up to 1500 h)
2.2 FIB – 3 basic operation modes

a) Emission of secondary ions and electrons
   ➢ FIB imaging low ion current

b) Sputtering of substrate atoms
   ➢ FIB milling high ion current

c) Chemical interactions (gas assisted)
   ➢ FIB deposition
   ➢ Enhanced (preferential) etching

Other effects:
➢ Ion implantation - Ga atoms remain in the sample target
➢ Displacement of atoms in the solids – Induced damage
   → lattice defects: dislocations, interstitials, vacancies
➢ Heating
2.2 FIB - Imaging with ions

- **Detector biased positive**
- Emitted from top 5-10 nm (very surface sensitive)
- Typically 30kV 40pA
- Grounded metals very bright

- **Detector biased negative**
- Emitted from top 0.5-1nm
- Lower yield, so images noisier.

FIB secondary **ion** mode

FIB secondary **electron** mode
2.2 FIB - Imaging with ions

**Secondary ion mode**

- i-beam; 30 kV - 150 pA; SI
- Ni alloy – incipient intergranular corrosion

**Secondary electron mode**

- i-beam; 30kV - 50 pA; SE
- Nb$_3$Sn

- Material (sputtering) contrast – removal of oxide layer
- Orientation contrast - channeling effect
- Topographic contrast
- (Dis)advantage – slow damage of the surface
- Brilliant for the inspection of microstructures without prior chemical or physical etching.

Atom column align with the ion trajectory = higher penetration
→ less sputtering and SE electrons

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## 2.2 FIB - Milling

- Sputter rate for a 10x10x5 μm box in Cu
- Typical ion current (high) 45 nA (up to 100 nA)
- Sputter yield for Cu: 0.25 μm³/s.nA

### Table:

<table>
<thead>
<tr>
<th>Material</th>
<th>Sputter rate</th>
<th>High current</th>
<th>Middle current</th>
<th>Low current</th>
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</thead>
<tbody>
<tr>
<td>Volume</td>
<td>[mm³/s.nA]</td>
<td>45 nA</td>
<td>10 nA</td>
<td>500 pA</td>
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<td>500 μm³</td>
<td>0.27</td>
<td>0.7</td>
<td>3</td>
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<tr>
<td>Si</td>
<td>0.3</td>
<td>0.6</td>
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<td>Al</td>
<td>0.08</td>
<td>2.3</td>
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<td>Al2O3</td>
<td>0.61</td>
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<td>GaAs</td>
<td>1.5</td>
<td>0.1</td>
<td>1</td>
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<td>Au</td>
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<td>6</td>
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<tr>
<td>TiN</td>
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<td>C</td>
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<tr>
<td>Ti</td>
<td>0.18</td>
<td>1.9</td>
<td>8</td>
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<tr>
<td>Cr</td>
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<td>0.6</td>
<td>3</td>
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<tr>
<td>Fe</td>
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<td>Ni</td>
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<td>0.7</td>
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<td>67</td>
</tr>
<tr>
<td>Cu</td>
<td>0.12</td>
<td>1.5</td>
<td>7</td>
<td>139</td>
</tr>
<tr>
<td>Mo</td>
<td>0.32</td>
<td>0.6</td>
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<td>52</td>
</tr>
<tr>
<td>W</td>
<td>0.12</td>
<td>1.5</td>
<td>7</td>
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<tr>
<td>MgO</td>
<td>0.15</td>
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<td>6</td>
<td>111</td>
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<tr>
<td>TiO</td>
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<tr>
<td>Fe2O3</td>
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<tr>
<td>Pt</td>
<td>0.23</td>
<td>0.8</td>
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<tr>
<td>PMMA</td>
<td>0.4</td>
<td>0.5</td>
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</tbody>
</table>

**Volume:** 500 μm³

**Time:** Volume/sputter rate = 200 s = 3 min

- Not the final time !!!
- Why?
2.1 FIB - Milling

- Less damage (curtain effect) at cutting surface for small currents
- Better resolution for small current but high currents mill faster → series of decreasing currents need to be used
- Redeposition → milling strategy is important → “milling mode / deposition mode”
2.2 FIB - Deposition

- Nozzle $\rightarrow$ local gas atmosphere
- Decomposition of precursor gas $\rightarrow$ CVD
- Deposition without surface damage $\rightarrow$ only e-beam
- Deposited material $\rightarrow$ a mixture of Ga, C and the depo species (C, Pt, W, XeF$_2$,...)
3. Application overview by FIB-SEM and TEM → Research and development TE, EN dept. and CIME.

HIE-ISOLDE upgrade project
→ Boost the radioactive beam energy from 3MeV/u to 10MeV/u by using SC linac.

Quarter-wave resonator (QWR):
Nb thin film sputtered on 3D forged OFE Cu substrate
3. Research and development TE, EN dept. and CIME.

DC-bias sputtering baseline parameters are:

- 0.2 mbar Ar pressure, 8 kW powered Nb cathode, -80 V biased cavity
- Substrate temperature rising from 300°C up to 630°C (below the bake out temperature of 650°C) on the inner conductor during a coating step.
- Coating process lasts 4 days and is done in 14 steps of 25’ coating + 5h35’ cool down to 300°C each, leading to a net coating time of 6h
3. Research and development TE, EN dept. and CIME.

Film thickness (XRF) and RRR profile, courtesy A. Sublet
3. Research and development TE, EN dept. and CIME.

FIB-SEM surface morphology

Inner conductor:
- flat grains
- apparent grain boundaries
- grain size ~ few µm

Outer conductor:
- very fine plate-like structure
- grain size ~ few 100 nm
3. Research and development TE, EN dept. and CIME.

Basic application – Cross-section
4 steps:
1. Deposition of protective layer.
2. The removal of the material by sputtering using the staircase-like pattern with high Ga+ beam current to form the large trench.
3. Reduction of the beam current and milling to reduce redeposition of sputtered material onto the sample surface.
4. Imaging of the final surface.

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3. Research and development TE, EN dept. and CIME.

FIB-SEM cross section imaging
3. Research and development TE, EN dept. and CIME.

Nickel-yttria stabilized zirconia, solid oxide fuel cell (SOFC) anode
3. Research and development TE, EN dept. and CIME.

**e9 FIB tomography**

Unexpected presence of pores lead us to FIB tomography in order to visualize distribution of the pores and quantify their volume. Accelerating voltage during acquisition was set to 1.85 kV. 3D ATLAS software was used during the **16 hours experiment and 788 images** was acquired.

4170x576x301 (7.8nm slices) SESI detector 1.85 kV, M. Cantoni and B. Bartova

Pore reconstruction in a section (500x500x301) of Nb coating

→ 1% pore volume fraction
Distribution of the porosity in the sample
Possibly different phases distribution
3. Research and development TE, EN dept. and CIME.

i9 FIB tomography

3 kV, other additional information were not specified

→ 0.2 % pore volume fraction
3. Research and development TE, EN dept. and CIME.

Advanced application – TEM lamella preparation
3. Research and development TE, EN dept. and CIME.

**(S)TEM**

- In STEM mode the focused probe is scanned across the specimen in a raster and the image is built up one image point or pixel at a time as opposed to the whole image formed parallel at one time in CTEM.
- HAADF STEM is a valuable tool for the study of chemical homogeneities in materials containing elements of sufficiently different atomic numbers.

**Tecnai Osiris**

- 200 kV Accelerating voltage
- 0.18 nm HAADF STEM resolution
- A-Twin pole piece with Super-X EDX (4 SDD detectors and 0.9 srad solid angle)
3. Research and development TE, EN dept. and CIME.

e9 STEM imaging and EDS analysis

- HAADF STEM image with corresponding mapping of the Nb Coating.
- Oxygen layer at the top as well as around pores was revealed.
- No other inhomogeneity was detected.
3. Research and development TE, EN dept. and CIME.

e9 EDS at Cu/Nb interface

• Detailed mapping at the interface revealed presence of max 20 nm sized Cu precipitates.
• The precipitates are randomly scattered along the Cu/Nb interface and were found up to 200 nm far from the interface.
• Oxygen enrichment at the interface and around the porosity is detected.

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3. Research and development TE, EN dept. and CIME.

**e9 Orientation mapping**

- Technique based on collection of precession electron diffraction patterns and cross-correlation with the simulated template.
- Grains in the coating show no preferential orientation.
- The very small grains close to the interface cannot be index because of grain overlap.
- For the grain size characterization plain view sample at well defined height of the coating is needed.
- XRD data measurement are needed for comparison of results.

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3. Research and development TE, EN dept. and CIME.

**i4 STEM imaging**

- HAADF STEM image revealed the epitaxial growth.
- BCC structure deals with the fast deposition by mis-orientation of the layers by max 1-4°.
- Density of dislocation is changing.
- Orientation mapping will be performed to clarify the layers orientation.
3. Research and development TE, EN dept. and CIME.

**i4 STEM EDS measurements**

- The mapping of first layer revealed the presence of porosity that is filled with Ar
- Presence of Cu precipitates
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• Answers to some questions that are here for a long time
• This systematic characterization of the film efficiently highlight the regions to be improved and help to target the hardware/process parameters to adjust (bias voltage to densify the film, cathode geometry, etc.)
• Combined together these material science and SC/RF approaches will help to tune the key parameters to achieve the best performances of Nb-coated SRF cavities
4. Summary.

• Preliminary results on Nb coating led to the proposition of FIB-SEM acquisition at CERN → June 2014
• The acquisition approved → December 2014
• EN-MME, TE-VSC, TE-MSC and BE-RF
• Testing of the machines → December 2014, January 2015
• Procurement procedure → 2015
• FIB-SEM at CERN → beginning 2016
Acknowledgments - CIME

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ZEISS Nvision 40

FEI Tecnai Osiris

JEOL 2200FS
Acknowledgments – TE-VSC and TE-MSC

Collaboration on Nb coating:
Alban Sublet, Sergio Calatroni, Mauro Taborelli

Collaboration on MgB2:
Michinaka Sugano, Amalia Ballarino, Marina Garcia, Julien Hurte

Collaboration on Nb3Sn:
Patrick Alknes, Bernardo Bordini, Christian Scheuerlein
Acknowledgments – EN-MME-MM
Thank you for your attention!
NanoMegas ASTAR

- The electron beam is scanned in combination with beam precession through the sample area of interest.
- A number of electron diffraction spot patterns are acquired at high speed using a dedicated fast CCD camera.
- Local crystal orientation are obtained by comparing all individually obtained ED spot patterns via cross-correlation matching techniques with ED templates.
- Resolution is determined by electron probe size and can reach 1nm on orientation maps with TEM-FEG microscopes.