# Cryogenic Dual Beam Nova 200

The cryogenic dual beam instrument is dedicated mainly to analysis of electron-sensitive materials (soft materials). The equipment core is based on the Nova Nanolab 200 model, but upgraded with a cryo-setup that allows the analysis of materials at low temperatures. This capability consists of a cryo-transfer setup and a cryo-chamber with embedded sputtering system. For example, this system permits to *in situ* generate controlled fractures on quenched soft materials, avoiding the mechanical damage associated with room temperature fractures.

In addition to the study of the material in its original state (porosity, embedded nano-objects, internal heterogeneities, etc.), any internal distribution of materials can be determined by using the Focused Ion Beam (FIB) to produce cross-sectional surfaces. A combined strategy between this equipment and the Helios Dual Beam Model 650, also available at our Center, is being conducted to produce series of ion-cuts of biological materials embedded in epoxy. These images are being used to produce three-dimensional (3D) information of material distributions. Appropriate software for compositional analysis based on Energy-Dispersive X-ray micro-analysis (EDX) is also included in this equipment. Additionally, the equipment also holds an Omniprobe nanomanipulator for lamellae preparation as well as 5 gas injectors.

The expertise of our scientific and technical staff is also offered to researchers from public and private research centers and also to professionals from industrial sectors that require the use of this instrument.

## *What can be done with this instrument?*

**Image** (resolution 1.4 nm)/ **Analysis**:

By using the different detectors available within this instrument, the following information can be obtained:

- Image with secondary electrons and topography by means of an ETD/TLD (Everhart-Thornley/ Thru-the-Lens Detectors).

- Image (back scattered electrons) and composition by using a BSED (Back Scattering Electron Detector).
- Image with secondary ions, sensitive to crystallographic direction.
- Elementary Chemical Analysis by EDX (Energy-Dispersive X-ray micro-analysis).
- STEM (scanning-transmission) images.

**Nanofabrication** (lateral dimension between 50 nm and tens of microns)

- FIB: focused ion beam; etching of a predesigned motif over the sample.
- FEBID/FIBID: focused electron/ion beam induced deposition.

PRECURSOR GASES

$(CH_3)_3(CpCH_3)Pt$, $Co_2(CO)_8$, $W(CO)_6$, TEOS + $H_2O \rightarrow SiO_2$, Selective Carbon Mill ($MgSO_4 \cdot 7H_20$)

**Micromanipulation**

- Lamellae preparation in conventional mode.
- Thinning at low temperatures (samples for TEM observation).
- Nano-manipulator (Omniprobe).

**Low Temperature**

- Fast freezing and cryo-fracture of materials. Samples can be fractured in the -180 to -150 ºC range. The observation can be made between -130 and -140 ºC ± 1 ºC.

## *Sample requirements*

- Non-conductive samples need metallization, which can also be done in our Centre.
- Conductive and non-conductive samples as bulk, films, powder (compacted), etc. can be studied.
- Samples should be compatible with high vacuum conditions.
- Samples in the 1 mm to 100 mm range can be studied. They should be less than 10 mm thick.
- Use of the cryo-option allows the measurement of liquid samples, semi-liquids and beam sensitive samples, polymers, resins, MOFs (metal-organic frameworks), etc.

## *Technical Specifications*

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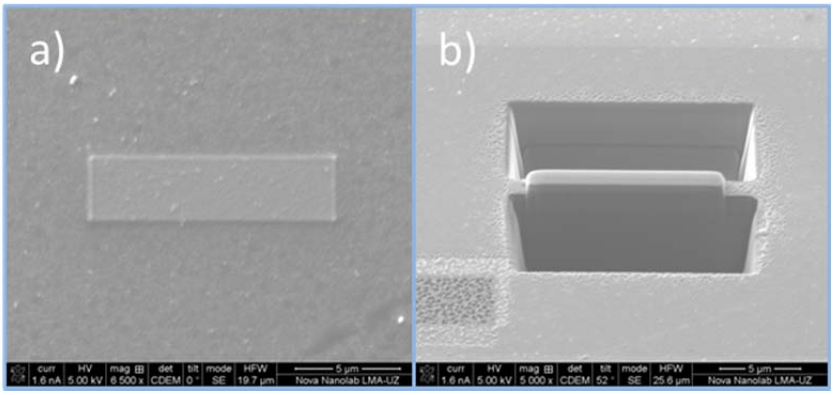
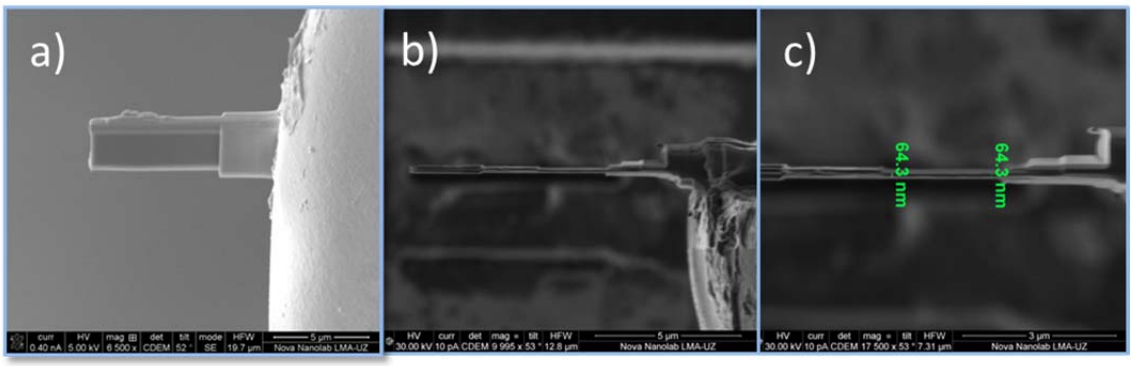
|---|---|
| **Electron beam resolution** | 2.5 nm at 1 kV, 1.4 nm at 15 kV |
| **Ion beam resolution** | 7 nm at 30 kV |
| **Landing Voltage Range** | E-beam: 200 V-30 kV<br>I-beam 2kV-30kV |
| **Probe current** | E-beam 1.4 pA (1kV) up to 37 nA (30kV)<br>I-beam: 1 pA up to 20 nA at 30 kV |
| **High Precision 5-axes motorized stage** | XY: 50 mm<br>Z: 25 mm<br>T: -10 to +60<br>R= 360° (continuous) |
| **Chamber vacuum** | <2.6 x $10^{-6}$ mbar (after 24 h pumping) |
| **Sample Size** | Maximum size: 150 mm diameter with full rotation( larger samples possible with limited rotation).<br>Weight: max 500 g (including the sample holder). |
| **Cryo-option<br>Model PPT2000 with Cryo-transfer from Quorum Technologies** | |
| **Transfer** | After freezing and vacuum transfer the sample is placed on the preparation chamber cold stage. Stage temperature is normally set to between -130°C and -140°C (precisely controlled to with +or-1C) |
| **Fracturing** | Sample can be fractured using either the cooled probe or cryo knife Tools. |
| **SUBLIMATION (ETCHING):** | Water (ice) can be sublimed (etched) from the sample by raising the stage temperature (typically to between -80°C and -100°C. |
| **COATING** | Sample is sputter coated with Pt or C and then transferred in to the SEM cold stage. |

## *Images*

Lamellae preparation and thinning at low temperature.

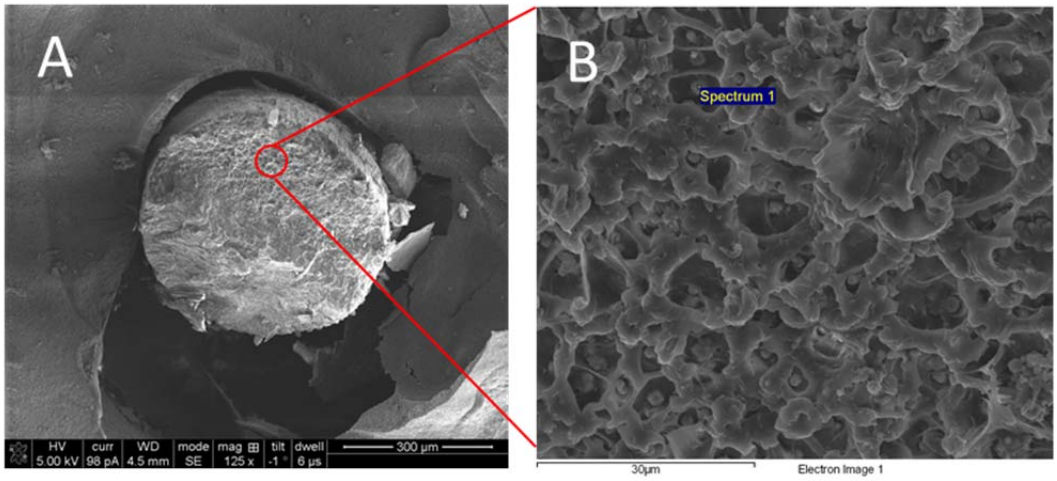


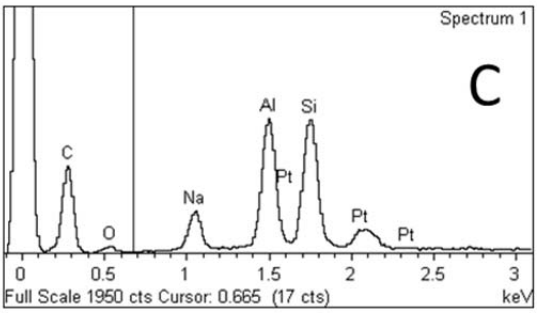

a) Pt deposited by e- over multilayer organic magnetic tunnel junctions b) View of milling by i+ beam during the process to make lamella previous to the lift out.




a) Electron image of lamella of organic magnetic tunnel junctions on the grid. b) Final view of the lamella, ion image c) details and thickness of lamella by ion image, the final process was made at low temperatures (-160 ºC)

Cryo-Fracture in organic materials**.**




(A) General overview of polymers after of cryofracture. (B) Details about the internal structure and distribution of Zeolite (C) show the spectral analysis and of specific zeolite areas, demonstrating the presence of the aluminum and Silicon typical in the zeolite.