



Flagships: FEI Nova NanoLab 200 DualBeam FIB and the FEI Helios NanoLab DualBeam FIB

High-resolution scanning electron microscopes with focused ion beams (FIBs), and energy dispersive X-ray spectroscopy and electron back scattered diffraction systems

With dual high-resolution electron and ion columns, these advanced microscopes offer a key capability in sub-nanometre-resolution imaging, in high-precision cross-sectioning by ion milling, and in elemental and orientational analysis. They also make possible 3-D image reconstruction by slice-and-view, script-driven large-scale prototype patterning and preparation of thin-foil TEM samples of difficult materials.

Scientific Drivers

Sub-structure investigation is critically important in current studies of materials. In many cases, examination of specimens through TEM is limited by the inherent difficulties in specimen preparation, particularly due to the hardness of the sample. The nature of the focused ion beam allows semi-automatic preparation of electron-transparent sections of these difficult samples for site-specific TEM investigation. The stages have a spatial precision better than one micrometre, and sections can be milled in one to two hours. This represents substantial time saving over conventional sample-preparation methods and allows studies to be performed on a number of materials for which TEM preparation is otherwise tedious and extremely difficult. Examples include surface-treated samples such as plated or chemically modified materials and mineral samples that are functionally graded, having soft and hard phases that would differentially polish or thin when prepared by other techniques.

Another exciting and emerging field in materials science is the analysis of both microstructure and chemical composition of site-specific features in three dimensions. The DualBeam FIB instruments provide a platform to do this, with extensive potential for a wide range of advanced scientific and engineering applications. The NanoLab DualBeam FIBs enable serial slices, only tens of nanometres thick, to be cut through a feature of interest, even if it is buried. The serially exposed cross-sections can then be imaged by field-emission scanning electron microscopy, and the recorded dataset of 2-D images processed to generate 3-D representations of the sample. Elemental analysis in three dimensions can be achieved in the same way by sequential milling of the sample and by mapping the exposed surfaces. FIB milling also allows the preparation of difficult surfaces for electron backscattered diffraction of mineral samples to show the crystallographic relationships between transformation phases.

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The ability to mill materials of differing hardness has made possible the investigation of bio-films on metal and mineral surfaces. Imaging of beam-sensitive materials at nanometre resolution is also possible by using the low-voltage (e.g., 1–2 kV) capabilities of the electron-beam column.

Some examples of projects enabled by the DualBeam FIBs include:

- The preparation of cross-sections through optical coatings on 'glass' lenses, which have been used as a defect analysis procedure in conjunction with conventional microtomy.
- Novel 3-D visualisations of material structures, such as internally oxidised steels or thermally sprayed surfaces.
- Determination of the preferential orientation of thin-film photovoltaic materials, which can be directly related to their efficiency of energy conversion.

References

P.R. Munroe, "The Application of Focused Ion Beam Microscopy in the Material Sciences." *Materials Characterization*, 60, 2-13 (2009).

M.Z. Quadir, M. Ferry, O. Al-Buhamad and P.R. Munroe, "Shear banding and randomization of the recrystallization texture in Al layers in a multilayered Al-based ARB sheet." *Acta Mater.*, 57, 29-40 (2009).

A.V. Nagasekhar, C.H. Caceres and C. Kong, "Characterization of intermetallics distribution in two and three dimensions in high pressure die cast AZ91 alloy." *Magnesium Technology 2009 Edited by TMS (The Minerals, Metals & Materials Society)*, 101-104 (2009)

J. Yao, D. Li, X. Zhang, C. Kong, W. Yue, W. Zhou and H. Wang, "Cubes of zeolite A with an amorphous core." *Angewandte Chemie*, 120, 8525-8527 (2008).

L. Han, J. Yao, D. Li, J. Ho, X. Zhang, C. Kong, Z. Zong, X. Wei and H. Wang, "Hollow Zeolite Structures Formed by Crystallization in Crosslinked Polyacrylamide Hydrogels." *J. Mater. Chem.*, 18, 3337-3341 (2008).

Z.H. Xie, N.M. Kilpatrick, E.K. Mahoney, M.V. Swain, P.R. Munroe and M Hoffman, "Transmission Electron Microscope

- Determination of the 3-D relationships of surface cilia in cystic fibrosis.
- Use of the FIB in forensic science to obtain the structural morphology, and elemental composition, of particles recovered from a crime scene. These data can be useful in assigning a possible source for this evidence.

Capabilities and the National Research Capacity

The versatility of the DualBeam FIBs in sample preparation, surface modification and high-resolution 3-D microscopy of a large range of materials adds substantially to the AMMRF's national research capability. Adelaide Microscopy at the University of Adelaide and the EMU at UNSW, where these two instruments are housed, both provide easy access to the equipment and technical support staff for all users, making these instruments available to all Australian researchers as part of the AMMRF.

Characterisation of Molar-Incisor-Hypomineralisation." *J. Mater. Sci: Materials in Medicine*, 19, 3187-3192 (2008).

A. Muhktar, D.L. Zhang, C. Kong and P. Munroe, "Variation in Hardness of Ultrafine Grained Cu-Al₂O₃ Composite Hollow Balls and Granules Produced by High Energy Mechanical Milling." *Materials Forum*, 32, 105-109 (2008).

A.J. Haq, P.R. Munroe, M. Hoffman, P.J. Martin and A. Bendavid, "Berkovich Indentation of Diamond-like Carbon Coatings on Silicon Substrates." *J. Mater. Res.*, 23, 1862-1869 (2008).

R.K. Singh, Z.H. Xie, A. Bendavid, P.J. Martin, P. Munroe and M. Hoffman, "Effect of Substrate Roughness on the Contact Damage of DLC Coatings." *Diamond and Related Materials*, 17, 975-979 (2008).

Z.H. Xie, M. Hoffman, P. Munroe, A. Bendavid and P. Martin, "Deformation Mechanisms of TiN Multilayer Coatings Alternated by Ductile or Stiff Interlayers." *Acta Mater*, 56, 852-861 (2008).

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