ADVANCED MICROSCOPY LABORATORY

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A unique microscopy facility driving world-leading materials research.



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CRANN is one of the largest research institutes within Trinity College Dublin. It has significant infrastructure, and brings together over 300 researchers from across the Schools of Physics, Chemistry, Engineering, Medicine and Pharmacology.

The Institute is a co-host to the AMBER (Advanced Materials and BioEngineering Research) centre. AMBER is funded by Science Foundation Ireland and industry and provides a partnership between leading researchers in material science and industry to develop new materials and devices for a range of sectors, particularly the ICT, medical devices and industrial technology sectors. The centre is jointly hosted in Trinity by CRANN and the Trinity Centre for Bioengineering, working in collaboration with University College Cork and the Royal College of Surgeons of Ireland.

www.ambercentre.ie

INTRODUCTION

The ability to image and manipulate materials in the nanoscale range is critical for the integration of the highest quality materials science research into technological developments. Materials science is driving developments in diverse areas particularly ICT, energy, pharmaceuticals, medical devices and diagnostics.

However, with the proliferation of imaging and analytical techniques it can often be confusing to determine the most appropriate analysis method. CRANN's Advanced Microscopy Laboratory (AML) offers a unique spectrum of equipment housed within a single facility, designed to meet the needs for advanced manufacturing and materials research, whether for academic or industrial purposes.

The AML contains a suite of instruments that covers the entire resolution range from transmission electron microscopy, electron energy loss and energy-dispersive X-ray spectroscopies with spatial resolution up to the atomic scale, to scanning electron and ion beam microscopes for surface imaging and analysis. Many of these microscopy techniques not only support analysis but also can be used for fabrication purposes in the nanoscale range. All of these techniques are managed by a dedicated team of highly trained staff with many years of academic and industrial experience, who can advise on the most appropriate method.

The equipment suite includes the atomic resolution Nion UltraSTEM200 microscope, which was funded by Science Foundation Ireland. This is the most powerful microscope in Ireland and one of the top 10 microscopes in the world with atomic scale imaging and analysis capable of 0.78 Å spatial resolution (1 nA in a 1.4 Å probe) and 0.35 eV EELS Resolution at 200 kV and 1.4 Å spatial resolution and 0.4 eV, 0.35 eV EELS resolution at 40 kV. The Nion microscope is also capable of single-atom sensitivity EDXS.

The AML has an open-access policy and is available for variable rates to academic and industrial users, both nationally and internationally. We are committed to driving internationally competitive materials science research which will benefit the economy and society in Ireland and beyond, and we look forward to welcoming you to our facility.

Prof Valeria Nicolosi

Principal Investigator in AMBER and Trinity's School of Chemistry



Transmission Electron Microscopes (STEM/TEM)

This Page_Close-up of the Nion UltraSTEM, capable of imaging and spectroscopy with atomic resolution.

The Nion UltraSTEM

Operating acceleration voltage









FEI Titan 80-300kV FEG S/TEM

The FEI Titan 80–300kV FEG S/TEM (Scanning/Transmission Electron Microscope) is a powerful instrument capable of high resolution S/TEM imaging and nanoscale analytical materials characterisation. It is equipped with an EDAX EDX detector and a Gatan Tridiem spectrometer for EELS and EFTEM. These analytical capabilities provide information on a material's chemical composition, ideally suited to materials qualification, nano-metrology, device testing and characterisation of a wide variety of nanoparticles and chip based materials. Within the AML, the Titan acts as our high end analytical S/TEM. Combined EDX/EELS capabilities in STEM mode, and EFTEM, make it a powerful tool for local nano-compositional analysis. It also acts as a screening instrument for our Nion UltraSTEM 200. These capabilities are supported by our suite of sample preparation instrumentation.

TECHNICAL SPECIFICATIONS

Operating acceleration voltages 80 kV/300 kV

Information limit 0.1 nm

STEM Resolution 0.2 nm, bright field, dark field and high angle annular dark field detectors

EELS Energy resolution < 0.8 eV

Nanoscale Energy Dispersive X-ray (EDX) spectroscopy

Complimentary simultaneous EDX/EELS STEM based analysis

In-situ thermal, cryogenic, fluid and electrical capabilities.







03



01_FEI Titan 80-300 kV FEG S/TEM

02_STEM image of germanium nanowire showing twinned planes in the crystalline structure. Courtesy E. McCarthy

03_STEM image of germanium nano-fins fabricated from block co-polymer (BCP) selfassembled patterning. EDX mapping shows the elemental structure. Courtey E. McCerthy

JEOL 2100 LaB₆ TEM

The JEOL 2100 LaB₆ TEM is capable of high resolution imaging and is suitable for a wide range of materials, from nanomaterials to biological specimens. The choice of two operating voltages, 100kV and 200kV, allows scientists to image beam sensitive samples as well as more robust specimens. Single tilt, double tilt, and multi-specimen holders are available for use with this system.

The multi-specimen holder supports 5 TEM grids allowing users to streamline their analysis and avoid lengthy sample exchange. The instrument also has an 80mm² XMAX EDX detector for elemental analysis.

TECHNICAL SPECIFICATIONS

Operating acceleration voltages 100 kV-200 kV

Information limit 0.14 nm Energy Dispersive X-ray (EDX) spectroscopy Stage tilt of ±60 degrees.

$01_JEOL 2100 LaB_6 TEM$ in use.

02_TEM image of gold nanoparticles bound to the surface of 210 nm diameter polystyrene particles. The gold nanoparticle binding sites show the location of transferrin protein within the polystyrene corona.

01





Scanning Electron Microscopes

This Page_Layered structure of bulk nickel hydroxides; taken by Zeiss ULTRA plus.

Zeiss ULTRA plus

This is a high resolution and analytical field emission Scanning Electron Microscope. Equipped with both InLens and SE2 detectors, users can obtain high resolution and topographical details of their samples, respectively. It also has an energy selective backscatter detector (EsB) for low kV compositional details of the samples. The Zeiss Gemini column gives very good resolution at low kV landing energies that allow surface details to be inspected and observed. The system also has STEM-in-SEM capabilities, which can give some basic bright field or dark field STEM imaging capability at 30 kV. A 20mm² Oxford Inca EDX detector enables elemental identification on samples with an energy resolution of 129 eV.

This SEM is a heavily utilised system that is used by all AML customers to give them further insight into their samples, particles, surfaces, layers and experimental results. A micromanipulator probing system can be connected to this SEM for probing capability.

TECHNICAL SPECIFICATIONS

Acceleration voltage range 0.1 kV-30 kV.

GEMINI® FESEM column capable of 1 nm resolution at 15 kV.

InLens, EsB, SE2, STEM and EDX detectors.

01_*Zeiss ULTRA plus in operation.*

02_MoS² crystals grown by chemical vapour deposition (CVD). Courtesy N. McEvoy

03_Ball of sodium dodecyl sulfate. Courtesy D. Hanlon

01







Zeiss ULTRA plus Cryo SEM

The Zeiss Ultra SEM can function as a standalone field emission SEM but also has the added advantage of cryo imaging capabilities. Cryo SEM offers the opportunity to capture high resolution images of frozen, fully hydrated samples in a condition very close to their native state.

Cryo SEM is effective for imaging samples that would be deemed 'difficult' in a standard high vacuum SEM. Liquid samples, high water content samples and unstable, beam sensitive samples can be quickly prepared and imaged at high vacuum resolutions. Additional benefits include the ability to expose internal structures by freeze fracture and controlled sublimation.

The food industry has been a very active user of cryo SEM over the years. Recently, other industries have become more aware of the advantages of this imaging technique. Cryo SEM has become an essential tool for pharmaceutical, cosmetics and healthcare industries, in both R&D and quality assurance.

The Ultra plus is also equipped with an 80mm² Oxford EDX detector (129 eV resolution).

TECHNICAL SPECIFICATIONS

Accelerating voltage range 0.1 kV-30 kV

GEMINI® FESEM column capable of 1 nm resolution at 15 kV.

InLens, SE2, AsB, EsB, and EDX detectors.

01_Zeiss ULTRA plus Cryo SEM.

02_SEM image of a cryo-prepared, freeze fractured plant stem. Courtesy D. Dooley

03_SEM image of a cryo-prepared, freeze fractured plant stem. Courtesy D. Dooley

04_Zeiss Ultra Plus image of a Bunch of boron doped carbon nanotubes (opposite). Courtesy C. Downing

01









10 — ELECTRON BEAM LITHOGRAPHY

Electron Beam Lithography

This Page_MOS2 flake contacted using electron beam lithography. Courtesy A. Bell, M. O'Brien, G.Duesberg

Zeiss SUPRA 40

The Supra FESEM is primarily used for high resolution electron beam lithography and imaging purposes, however this scope has been broadened with the recent installation of Bruker EDX and EBSD detectors, facilitating materials and elemental analysis. The Supra's GEMINI® FESEM column affords superb resolution down to 0.1 kV, a high efficiency In-lens SE detector, minimal adjustments when changing operating voltage and low magnetic fields at specimen level. A high-speed Raith beam-blanker/controller enables users to perform resist-based lithography of CAD designs.

TECHNICAL SPECIFICATIONS

GEMINI® FESEM column capable of 1 nm resolution.

Raith Quantum[™] high-speed beam blanker and software for precision electron beam lithography up to 30 kV.

Bruker XFlash® 6I30 EDX and e-FlashHR EBSD detectors, for materials and elemental analysis.

The AML boasts a class 10000 cleanroom with fume hoods, wet bench, and spinners for resist based lithography.

01_Zeiss SUPRA 40.

02_Crossed nanowires contacted by four silver lines using electron beam lithography on the Supra 40. Courtesy A. Bellew

01





Elionix ELS 7700

The Elionix ELS 7700 is a dedicated 75 kV electron beam lithography system. Equipped with a laser interferometer controlled stage, it is ideal for large area patterning as adjacent writefields may be seamlessly stitched together. Operating at 75 kV enables high precision lithography with reduced proximity effect. This is ideal for researchers looking to fabricate photonic waveguides, or other large area structures such as chess boards.

02

TECHNICAL SPECIFICATIONS

Dedicated electron beam lithography system. FESEM column operating at 75 kV.

Laser interferometer controlled stage enabling extremely precise X and Y movements.

01_*Elonix ELS 7700.*

02_MOS2 flake contacted using electron beam lithography. courtesy A. Bel, M. O'Brien, G.Duesberg 03_Close-up of Elonix ELS 7700 (opposite).





Focused Ion Beam (FIB) Technologies

This Page_Un-etched block copolymer PS:PMMA; from Zeiss ORION Plus. Courtesy A. Bell

Zeiss ORION Nanofab

Installed in November 2015, the ORION Nanofab is the only instrument of its kind in Ireland. Equipped with gallium, neon, and helium beams, the Nanofab combines high-resolution imaging with the fast, efficient milling capabilities of a FIB, for highprecision and high-throughput. Seamlessly switch between Ga- for removal of massive material, Ne- for high speed, high throughput nanofabrication, and He- for delicate, sub-10nm nanofabrication, and highresolution imaging. The tool is also well suited to imaging challenging sample sets such as polymer based systems and biological specimens, due to the absence of charging effects traditionally associated with SEM.

The Nanofab is also equipped with both Raith Multibeam and Fibics beam-blanker controllers and software, enabling users to translate complex CAD drawings onto their sample, either through ion milling, or ionbeam lithography.

TECHNICAL SPECIFICATIONS

Equipped with gallium, neon, and helium beams.

Capable of 0.5 nm resolution.

He- and Ne- ion lithography capabilities.

Raith Multibeam & Fibics beamblanker controllers.

01_Zeiss ORION Nanofab.

02_He-ion image of a nanowire spanning a SiO2 trench, in which a constriction has been milled by the He beam. courtesy A. Bellow

03_He- ion image of 13 nm lines of PMMA on SiO2 created using ion-beam lithography. Courtesy A. Bellew

01







Zeiss ORION Plus

One of the world's first installations of this exciting new technology, our Helium lon Microscope (HIM) affords the highest resolution surface imaging of bulk materials from a scanning microscope [0.4 nm], and bridges the gap in imaging resolution traditionally left between the scanning electron microscope [SEM] and the TEM. This is the only such tool installed in Ireland, and one of a handful in Europe.

The tool is well suited to imaging challenging sample sets such as polymer based systems and biological specimens, due to the absence of charging effects traditionally associated with SEM. Other exciting applications are dopant contrast mapping for the semi-conductor industry, and materials modification.

The HIM has a wide field of view, enhanced depth of focus, and excellent surface sensitivity.

TECHNICAL SPECIFICATIONS

Highly surface sensitive imaging to a resolution of below 0.4 nm.

Elemental analysis using backscattered ion spectroscopy.

Electron flood gun for charge compensationideal for biological samples.

01_Zeiss ORION Plus.

02_Breast cancer cell functionalized with gold nanoparticles. Courtesy A. Bell

03_Graphene flake imaged in PET. Courtesy A. Bell

01







Zeiss AURIGA Focused Ion Beam

The Carl Zeiss Auriga Focused Ion Beam (FIB) system is a versatile microscope tool for failure analysis, construction analysis, investigating embedded particles and features, implanting and deposition of selected materials and for in-situ TEM sample prep. The FIB is used widely in various industries such as semiconductor, ICT and medical devices. It is also used within academia and is having an impact in geology, mechanical, bone and biological disciplines. The FIB technique of cross sectioning is comparable to "key-hole" surgery for materials.

The system has a Gallium based ion beam column along with a field emission SEM

column, together they are often referred to as a dual beam system. The FIB is used to cut and the SEM to view and it also has live milling capability. The FIB can also be used to image and is especially useful for grains structure analysis due to ion channelling.

The FIB can be used to mill into samples in a controlled manner or deposit platinum or tungsten metal lines. The Zeiss FIB also has a low kV energy capability which helps to minimize any damage to samples especially for TEM sample prep. There is a Raith based system installed on the system for lon beam Lithography techniques.

02

TECHNICAL SPECIFICATIONS

GEMINI® FESEM column capable of 1 nm resolution at 15 kV.

Accelerating voltage range from 0.2 kV – 30 kV lon column technology with ~3 nm resolution lon beam currents range from 0.5 pA – 40 nA

01_Zeiss AURIGA Focused Ion Beam.

02_*TEM lamella targeting a nanowire cross* section. Courtesy D. Daly

03_Cross section and grains on a medical device. Courtesy D. Daty







FEI Strata 235DB

The FEI Strata 235DB is a dual beam FIB/ SEM system equipped with multiple electron detectors, and also includes a secondary ion detector.

The system has a digital scan board to help with complex milling patterns and it also has electron and ion beam lithography capability. The FIB has a 4 cartridge gas injection system (GIS) for either enhanced etching, or deposition of metal/insulator for protection layers and/or circuit editing.

TECHNICAL SPECIFICATIONS

lon beam currents range from 1 pA-20nA. lon beam resolution is ~7 nm. SEM resolution is ~5 nm. SEM accelerating voltage range 1kV-30 kV.

01_Operating the FIB Strata 235DB.
02_SEM image of a FIB cut circuit edit carried out on an integrated circuit chip. Courtesy D. Daty
03_FEI Strata 235DB (opposite).

01







AML HIGHLIGHTS

Excellence in Science

The AML has enabled over 15 Nature and Science group publications since 2013, including Science, Nature Materials, and Nature Communications.

Industrial Engagement

47 unique industry contracts in 2015; routinely supporting 20 longer term industry projects. Routinely accessed by 11 Industry researchers based full time in AMBER and external industry staff in SMEs and MNCs. Industry hot-desk facility.

Training & Development

Over 10,000 person hours of training provided to users since 2010. 285 unique users trained on Scanning Electron Microscopy; 178 users trained on Ion Microscopes; 110 users trained on Transmission Electron microscopy.

Funding Diversification

The AML draws funding from national (HEA, SFI, EI, Teagasc, EPA), and international sources (FP7, H2020, ERC) and Industry.

Driving research to benefit the economy and society

- -2D materials for energy storage.
- New materials for carbon dioxide capture to reduce greenhouse emissions.
- Magnets for better data storage technologies.
- -Cells for more effective cancer treatment.
- -Diatoms for more effective water monitoring
- -Blood platelets for enhanced drug delivery.

Open Access

Supporting all-Ireland microscopy access for Universities, Institutes of Technology, Semi-States and Industry.

Pushing Resolution Limits

Highest spec STEM in Ireland (Nion Ultra STEM200); highest resolution ion microscopes in Ireland (Carl Zeiss Orion; Carl Zeiss Auriga).

Experienced Support Staff

Blue chip industry experience including FEI, Hitachi, HP, Intel, Xilinx, and smaller HPSUs. Academic experience from worldrenowned Universities.

Supporting spin-outs

AML microscopes are used by Trinity campus company Adama Innovations to pattern diamond for applications such as metrology for the ICT industry and anti-counterfeit technologies.

Supporting Multi-disciplinary Research

Working with academics across Physics, Chemistry, Bioengineering, Geology, Immunology, Medicine, and Zoology.

International Placements

The AML has hosted placements of Post-Docs, Industry, and Undergrad students from across the world including Beijing University, the Netherlands and Cal Tech.

Public Engagement

Range of structured outreach and schools' activities, from primary school teachers and students to older people.

CONTACT

Our Advanced Microscopy Laboratory is open for business, for academics or industry users in Ireland and internationally. Get in touch to discuss access rates and logistics.

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DIRECTIONS: From CRANN: Walk towards

Grand Canal Docks for approximately 10 minutes. Once at Grand Canal Quay, turn right and enter through security gate on right. By Luas: Take the Red line to the Spencer Dock Stop, cross the Liffey using the Samuel Beckett Bridge to Pearse St, Grand Canal Quay. By Dart: Grand Canal Dock Station or Pearse

Advanced Microscopy Laboratory

Unit 27-29, Trinity Enterprise Centre, Grand Canal Quay, Dublin 2, Ireland.







