Sorby Nano Investigation Centre
Equipment List

The Sorby Nano Investigation Centre can offer your business a full range of cost effective micro and nano scale investigation capabilities. The latest microscopy techniques can be employed to analyse many cases, from the most demanding materials and environmental systems, to the most complex biological specimens.

Materials Analysis

- Analysis of microstructure and data interpretation
- Failure Mechanisms
- Composition Analysis
- Surface Analysis

Biological Analysis

- Ability to work with advanced biological specimens
- Expertise in biological systems and biofilms
- Data Interpretation

Environmental Analysis

- Advanced sample preparation
- Specialist Advice
- Data Interpretation

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1) Scanning Electron Microscopes (SEM)

The SEM is capable of producing high-resolution images of a sample surface. Due to the manner in which the image is created, they have a characteristic three-dimensional appearance and are useful for judging the surface structure of the sample. Several different types of electrons can be detected. Secondary electrons (generated by the specimen interaction with the electron beam) are the most common and primarily used for imaging. Back-scattered electrons (rarer, high energy electrons originating in the electron beam that are reflected or back-scattered out of the interaction volume) can be used to form an EBSD image which can determine the crystallographic structure and detail the substructure/microstructure of the material. The contrast between areas can be used to distinguish different chemical compositions since the brightness of the back-scattered electron image tends to increase with the atomic number. The FIB attachment can be used for milling TEM samples, surface layer analysis and imaging.

FEI Quanta 200 3d	Environmental, low vacuum SEM/FIB for 2D/3D characterisation Improved EDS analysis with high beam current Three imaging modes – high and low vacuum and environmental Max 3.5nm resolution in SE mode (high vacuum) Environmental stage to study in-situ dynamic behaviour of materials at different humidity levels High-current FIB enables fast material removal Live SEM imaging
FEI Inspect F (FEGSEM)	High-resolution, high vacuum field emission gun SEM Improved EDS and EBSD analysis with high beam current Max 1.2nm resolution in SE mode at 30kV Max 3.0nm resolution in SE mode at 1kV
FEI Sirion FEGSEM	FEGSEM with EBSD from HKL Technology Unique in-lens detection system Ultra-high resolution imaging in SE and BSE down to 500V Max 1.0nm resolution in SE mode at 30kV Max 2.0nm resolution in SE mode at 1kV
Philips PSEM 500 SEM	Fully motorised stage with SE and BSE detectors Be window EDS system (Na to U) and X-ray mapping Max 10 nm resolution in SE Digital image acquisition
JEOL JSM 6400 SEM	Fully motorised stage with SE and BSE detectors EBSD system from HKL Technology EDS system for microanalysis starting from Na

	Max 3.5 nm resolution in SE
	Digital image acquisition
Camscan Mk 2 SEM	X and Y motorised stage with SE and BSE detectors
	Capability of taking large samples
	Be window EDS system for microanalysis and X-ray mapping
	Manually controlled WDS spectrometer for light element analysis
	Max 7 nm resolution in SE mode
	Digital image acquisition

Fig. 1 FEI Quanta 3d SEM in the Sorby Nano Investigation Centre

2) Transmission Electron Microscopes (TEM)

The primary function of TEM microscopy is high magnification, since the use of an electron beam as opposed to a light source significantly increases the resolution (in the order of 1000s). Therefore objects of sub nanometre (10^{-9} m) can be imaged. The possibility for high magnifications has made the TEM a valuable tool in materials, medical and biological research. This is because fundamental properties, such as strength and electrical resistance, are intrinsically linked to atomic features and variations (such as grain boundaries and dislocations), and an increased knowledge of these properties is advantageous in material selection. Some practical examples include morphology (the size, shape and arrangement of the particles, as well as their relationship to each other on the scale of atomic diameters), crystallographic information (the arrangement of atoms, their degree of order and the detection of atomic-scale defects in areas a few nanometres in diameter) and compositional information (the elements and compounds the sample is composed of and their relative ratios, in areas a few nanometres in diameter, if equipped with an energy-dispersive spectrometer (EDS or EDX)).

JEOL 2200F	Ultra high point resolution of 0.14nm at 200kV
	Aberration corrected
JEOL JEM 3010 TEM	Ultra-high point resolution of 0.19nm at 300kV
	EDS detector for microanalysis of elements starting from B.
	High Resolution High speed CCD Camera for digital images and recording dynamic experiments
FEI Tecnai 20 TEM	Fully computerised, 0.24nm resolution at 200kV
	Computerised stage for accurate specimen position control
	EDAX EDS system qualitative and quantitative microanalysis
	Digital CCD Camera for high-quality image recording
Phillips EM 420 T	Max 120kV TEM, resolution approaching 0.34 nm
	EDS detector to analyse elements starting from Na
Phillips EM 430	300kV TEM with twin lens configuration for very high tilt
	Lattice resolution down to below 0.2nm
	EDX-system and digital recording capabilities

Fig. 2 FEI Tecnai 20 Transmission Electron Microscope at Sheffield University

3) Optical Microscopes

The optical microscopy uses light and a system of lenses to magnify images of small samples. Optical microscopes are the oldest and simplest of the microscopes, but the resolution is limited by the wavelength of light, which is why today this technique is often used in conjunction with EM.

Confocal laser scanning microscopy (CLSM or LSCM) is a technique for obtaining high resolution optical images. Its key feature is its ability to produce in-focus images of thick specimens, a process known as optical sectioning. Images are acquired point-by-point and reconstructed with a computer, allowing three-dimensional reconstructions of topologically complex objects. CLSM is widely-used in numerous biological science disciplines, from cell biology and genetics to microbiology and developmental biology.

Confocal microscopy equipment can be adapted for Raman spectroscopy. The Raman Effect involves the interaction of monochromatic light with matter. Laser light causes atoms in the molecules to vibrate, the analyses of which provides a fingerprint and reveals information about chemical structure and physical state of the sample.

Zeiss LSM 510 META	Point scanning laser confocal microscope Fully automated with optoelectronic Z drive to create 3D images Two single channel detectors, a polychromatic multi-channel detector and seven laser lines (ranging from 405 to 633 nm). High-sensitivity photo-multiplier tube detection, providing 12-bit confocal images up to 2048 by 2048 pixels
Horiba LabRAM HR800	Unique DUAL PATH optics with Raman spectroscopy Fast, simple switching between UV and VIS/NIR regions Wide spectral range, applied to PL and Raman spectroscopy 800mm focal spectrometer for a resolution of 1 cm^{-1}/pixel in UV Automated operations with motorized XYZ plots and mapping UV laser wavelengths from 244 to 785nm

Fig. 3 Horiba LabRAM HR800 Raman Confocal microscope in the Sorby Nano Investigation Centre

4) Electron Microprobe

The electron microprobe is an analytical tool used to non-destructively determine the chemical composition of small volumes of solid materials. It uses a high-energy focused beam of electrons to generate X-rays characteristic of the elements present within a sample volumes 1 to 3 micrometres across and can quantitatively analyze elements from boron to plutonium at routine levels as low as 100 parts per million (ppm). Chemical composition is determined by comparing the intensities of characteristic X-rays from the sample material with intensities from known composition (standards).

CAMECA SX-51

Four wavelength dispersive spectrometers

Microprobe can perform automated line scans and elemental maps

Qualitative and quantitative analysis of elements from Be to U

Detection limit of 100 – 500 ppm

Sample size: 25 mm diameter, 20 mm height

Fig. 4 Cameca SX-51

5) X-ray Photoelectron Spectroscopy (XPS)

XPS is a surface specific technique that employs the photoelectric effect. This is a phenomenon in which electrons are emitted from matter after the absorption of energy from electromagnetic radiation such as x-rays or visible light. It is highly surface specific due to the short range of the photoelectrons that are excited from the solid. The energy of the photoelectrons leaving the sample is measured to give a spectrum with a series of photoelectron peaks. The electron binding energy of each peak is characteristic of an element. XPS can therefore be used to obtain a number of characteristics; elements and their quantity within the first 10nm of the sample surface, the identification of any contamination, chemical states and the thickness of one of more thin layers (1-8nm) of different materials on the surface.

Kratos Axis Ultra

High resolution, high vacuum (10^{-8} to 10^{-10} mbar)
Two X-ray sources, monochromatic Al-K and a dual (Mg and Al) non-monochromated
Hemispherical analyser and delay line detector (DLD)
Patented magnetic immersion lens improves capture and sensitivity
Spectra can be obtained from surface areas <15μm
Parallel XPS imaging with spatial resolution of 6μm

Fig. 5 Kratos Axis Ultra at the Kroto Research Institute, University of Sheffield

6) Secondary Ion Mass Spectrometry (SIMS)

The bombardment of a surface by a primary ion beam creates secondary ions released from the surface, called splutter. The mass spectrometry of the emitted ions constitutes secondary ion mass spectrometry (SIMS) and gives information and quantitative data on the composition. It is widely used for the analysis of trace elements in solid materials, especially semiconductors and thin films. The spluttering of the sample can be maintained into the bulk of the material, and layers up to 1000nm thick can be depth-profiled (known as dynamic SIMS). The main advantages of SIMS over other composition analysis techniques include the ability to identify all elements and in very low concentration levels. Disadvantages of the technique is first, the limited sampling capacity due to limited beam diameter (1-200 micrometers) and secondly, the destructive nature of the sampling.

Ion-TOF V

High resolution, high vacuum
Reflectron time of flight analyser allows very high mass resolution
Excellent image capability that can obtain a full SIMS spectra at a spatial resolution of 300nm
Analysis of specific spectral features

Fig. 6 Ion-TOF V SIMS at the Kroto Research Institute, University of Sheffield

7) Glow Discharge Optical Emission Spectroscopy (GDOES)

Glow Discharge Optical Emission Spectrometry (GDOES) is mainly used for the elemental analysis of bulk solids. It is an analysis technique that can be used for surface, elemental composition vs. depth profiles and bulk analysis of almost any material or coating, conductive or non-conductive. In glow discharge, accelerated ions splutter the surface, leading to some surface atoms being ejected. These are excited by collisions inside an anode, away from the surface producing an optical emission spectrum characteristic of the sample composition. It is possible with GDOES to measure signals from the first atomic layers of the surface down to depths of more the 100µm. For example, this technique can provide a useful tool for understanding of the mechanisms and processes involved in the formation of surface and subsurface layers in thermomechanically processed material.

Horiba GD-ProfilerT HR

Depth resolution to < 1nm
Pulsed RF Source to handle conductive and non conductive layers
HDD detectors allow an instantaneous dynamic range
Elements can be de determined at the ppm in one layer and at +99% in the next
Large sample chamber for wide range of applications

Fig. 7 Horiba GD-ProfilerT HR at the University of Sheffield

8) Scanning Probe Microscope (SPM)

Atomic Force Microscopy (AFM) is a very high-resolution type of scanning probe microscope. It has a demonstrated resolution of fractions of a nanometre (1000 times better than the optical diffraction limit) and is an important tool for imaging, measuring and manipulating matter at the nano scale. Information is gathered by "feeling" the surface with a mechanical probe; therefore a true 3D surface profile is recorded. Forces that can be measured include mechanical contact force, Van der Waals forces, capillary forces, chemical bonding, electrostatic forces, magnetic forces etc. Samples viewed by AFM do not require any special treatments (such as metal/carbon coatings) that would irreversibly change or damage the sample and it can work perfectly well in ambient air or even a liquid environment, making it possible to study biological macromolecules and even living organisms. The major disadvantage with AFM is the image size, which is on the micrometre scale as opposed to a SEMs order of millimetres, in other words the resolution is too high.

DI Dimension 3000	X-Y scan range of 90μm, and Z range greater than 6μm
	Low noise performance, characterises and controls tip movement
	Nanoscope 3D controller combining analogue and digital control
	Large sample stage provides full range of SPM techniques
	1.5μm optical resolution, computer controlled illumination and video capture
	150μm to 675μm viewing area, motorised zoom and focus

9) Specimen Preparation

Even with the latest techniques available, specimen preparation is crucial in obtaining good results for microscopic analyses. This can be time consuming and sometimes painstaking, but is key to achieving the required specimen finish and outcomes.

For example, TEM specimens need to be thin enough for the beam to pass through (a few tens of nanometres to a micron in thickness) and provide a representative region, but must not be altered by the preparation technique. The sample should also be strong enough to be handled and be resilient enough to be examined in the microscope. All of these requirements are not easily met, and the degree of difficulty can range from trivial to very skilful.

For the study of surface morphology, bulk specimens are normally used, and therefore the preparation of SEM specimens is far simpler than that for TEM. The aim of specimen preparation for SEM is to create a flat, smooth clean surface. In most cases this is achieved through various grades of grinding and polishing. If etching is required, this must be adequate enough to reveal the true microstructure without distortion, smearing, pitting etc. Where the final surface quality of the specimen is imperative (such as EBSD) electro-polishing may be necessary.

For effective viewing of a specimen in the SEM, the surface needs to be electrically conducting. This is to allow the surplus of surface electrons that builds up during operation to be conducted away. For clean metal specimens, there are no problems providing that there is a path to earth. However for non-conductors such as ceramics, polymers and biological materials is usual to coat the specimen with a thin (approx. 10nm) conducting layer of gold or carbon. This is usually easily and rapidly done by splutter coating. However care must be taken so that the coating does not mask surface details or interfere with other modes or contrasts e.g. X-ray emission. The examination of polymers and biological materials in the SEM may present other problems, such as specimen degradation by beam heating or radiation damage. These problems tend to be individual in nature and need to be tackled specifically.

Equipment

Gatan Duo Mill Ion Beam Milling Equipment (IBM) (X2)

Gatan Precision Ion Polisher (PIPS)

Precision ion polisher designed to produce high quality, TEM specimens. High milling rates at shallow angles to less than 1 degree

Dimpler

The dimpling technique is applicable to many materials and yields a higher quality specimen with a broader, near electron transparent area which reduces ion milling times. The technique is automated so that the most demanding specimen can be reduce to < 10 microns routinely and repeatedly. Real time measurement of tool height, specimen thickness, and tool run out allow the user to precisely control the dimpling process.

Emitech C evaporation unit

Coating unit that allows non-conductive materials to be coated with carbon, which is conductive and easily and evenly distributed. Specimens can then be viewed in the SEM.

Gold Sputter unit

Similar to the carbon coating unit, but gold sputter is used instead. The unit supplies a multi-directional deposition of sputtered atoms that will form an even coating on the surface of the specimen. This electrically conductive thin coating will be representative of the surface of the specimen. It will inhibit charging, reduce thermal damage, and improve secondary electron mission which are beneficial for Scanning Electron Microscopy.

Various polishing and cutting equipment

10) Glossary of Terms

AFM	-	Atomic Force Microscopy
BSE	-	Back Scatter Electron
EBSD	-	Electron Back Scatter Diffraction
EDS	-	Electron Dispersive X-ray Spectroscopy
EDX	-	Energy Dispersive X-ray Analysis
EM	-	Electron Microscopy
ESEM	-	Environmental Scanning Electron Microscope
FEGSEM	-	Field Emission Gun Scanning Emission Microscope
FIB	-	Focussed Ion Beam
GDOES	-	Glow Discharge Optical Emission Spectroscopy
NIR	-	Near Infrared
PL	-	Photoluminescence
SE	-	Secondary Electron
SEM	-	Scanning Electron Microscope
SIMS	-	Secondary Ion Mass Spectrometry
SPM	-	Scanning Probe Microscope
TEM	-	Transmission Electron Microscope
UV	-	Ultraviolet
VIS	-	Visible
WDS	-	Wavelength Dispersive X-ray Spectrometry
XPS	-	X-ray Photoelectron Spectroscopy