



University of  
South Australia

The  
*Wark*<sup>TM</sup>

Ian Wark Research Institute

ARC Special Research Centre for Particle and Material Interfaces

**Scientific Services**

Experience. The Difference.

unisa



# Introduction

Do you have problems with your flotation process, analysis needs, want a microfluidic chip or micro device manufactured or need advertising material designed and printed? If you answered yes you need to talk to us. If we can't help you will be referred to someone who can.

Scientific Services is a team of professionals, some of them with industrial experience, that have access to world class facilities and research staff within a research institute that is internationally recognised. Using our core expertise of particle and material interfaces with the Ian Wark Research Institute, Scientific Services offers a rapid turnaround consulting service with work programs and analysis tailored specifically for your requirements.

Our friendly staff will usually respond to your enquiry within 24 hours and you will receive an obligation free quotation that sets out what we

will do for you, how much it will cost and when you can expect delivery. Our customer service systems have been developed and refined over many years to provide you with superior and reliable service, every time. With a strong focus on communication, quality and personal attention, we provide solutions that are just one phone call away.

All enquiries and consulting projects are treated with the strictest confidence and we are well equipped to manage issues relating to intellectual property. We have standard non-disclosure agreements and will be happy to consider agreements provided by our clients.

Significant discounts are available for routine analysis of one or more samples, if conducted on a regular basis.

We also offer a longer term service to assist with research and development.

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# Scientific Services

Scientific Services hosts the SA node of the Australian National Fabrication Facility, a micro and nanofabrication facility offering design, development and fabrication of custom and off the shelf microfluidic chips and devices in a variety of materials. Specialist design and microfabrication services, combined with expertise in surface modification and functionalisation are also available. Microfabrication techniques for products in glass, metal, plastics and ceramics, coupled with a suite of specific inspection equipment, provide a full in-house fabrication and quality assurance service.

Working closely with experts in characterisation, the Minerals Technology team have comprehensive experience in mineral separation including flotation of ores containing base and precious metals, coal, oxide and industrial minerals together with a wide range of plant experience. Staff also have experience in magnetic and gravity separation as well as hydrometallurgical (leaching, solution treatment and recovery of values from solution) and pyrometallurgical processing of a wide range of materials.

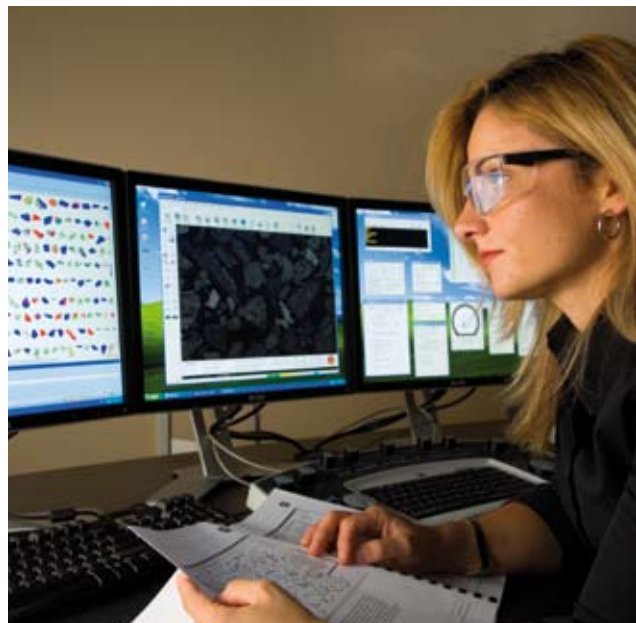
Materials analysis and characterisation is an area in which we have a significant level of expertise, with a particular focus on what happens at the surface of the material, recognised by the fact that The Wark hosts a number of surface analysis instruments of national significance within the Australian Microscopy and Microanalysis Research Facility. These instruments, one of which is the only one of its type in Australia, are available to assist with solving industry problems.

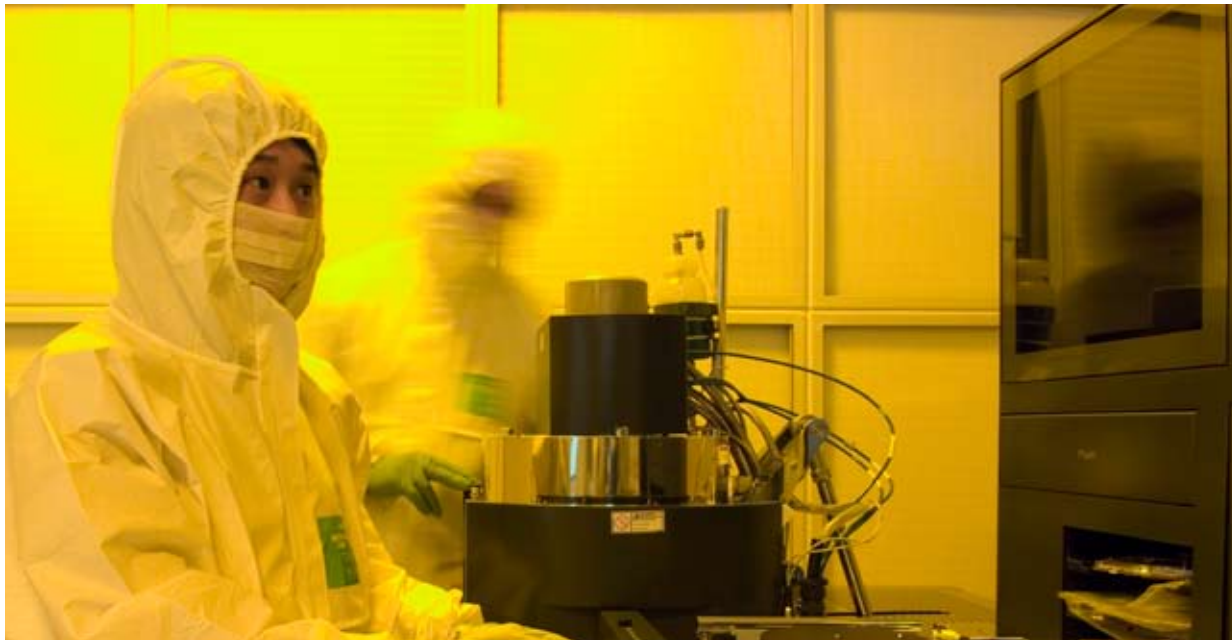
The chemical and physical nature of surfaces and materials influences factors such as suitability for specific applications, lifetime and performance. Surface analysis is particularly relevant in the consideration of coatings, adhesion, printability and resistance to corrosion and the study of specific minerals-related problems. The science is readily applied to a wide range of industrial processes, including product development.

The opportunities for industry are extensive and include, but are not limited to:

- Ceramics
- Metal forming and metal finishing
- Biomaterials and bio-medical
- Aerospace
- Automotive manufacturing
- Pharmaceuticals
- Polymer manufacture
- Whitegoods
- Catalysis: Petroleum industry

- Paints
- Composites
- Forensics
- Building materials
- Waste disposal
- Fertiliser/Agricultural chemicals
- Wood, paper and composite boards
- Glass manufacture
- Mineralogy
- Defence industries





Expertise in surface analysis, with a focus on minerals, colloids, polymers, biomaterials and micro and nanofabrication (and the interface between one material and another), has been applied in local, interstate and international industries.

Each investigation is individually tailored to the needs of the client.

The Wark has provided valuable information to manufacturers in many areas including:

- Paintwork defect identification
- Sources of paint delamination
- Treatment of metals prior to finishing
- Corrosion and fracture analysis
- Modified surfaces for printing and adhesion
- Particle analysis and identification
- Surface contamination
- Thin film analysis

Scientific Services also offers a fast and effective design service through its Visual Productions Unit, offering graphic design, technical drawing and posters at competitive prices.

# Mineral Processing Expertise and Equipment

The Wark manages a full range of equipment and expertise relevant to the minerals industry. The collective expertise of research and technical staff is coordinated and applied to short and medium term industrial consulting, as well as strategic research.

The Wark personnel have comprehensive experience in mineral separation including flotation of ores containing base and precious metals, coal, oxide and industrial minerals, together with a wide range of plant experience. Staff also have experience in physical (magnetic and gravity) and chemical (leaching and recovery from solution) processing of a wide range of materials.

## Mineral Processing Equipment

- Sample preparation including crushing, grinding and screening, riffing to sub-samples
- Classification including hydrocyclone separation to 5 $\mu$ m
- Grinding and flotation at batch and 5 kg/h pilot scale, with automated reagent addition, regrind mill and column flotation
- Wet and dry magnetic separation
- Gravity separation using shaking table and centrifugal separators

## Plant Diagnostics

- Many plant surveys conducted at flotation plants in Australia and overseas since 1988
- Pulp and solution chemical analysis
- Flotation cell hydrodynamic analysis
- Modelling of plant circuits and individual cells
- Surface analysis of value and gangue minerals

## Analytical Facilities

- Particle sizing
- Scanning Electron Microscopy with Energy Dispersive X-Ray Analysis
- Time of Flight Secondary Ion Mass Spectroscopy
- FTIR, UV-Vis Spectroscopy
- X-Ray Photoelectron Spectroscopy
- X-Ray Diffraction
- Rheology determinations on slurries



# Colloid, Interface and Surface Analysis

## Analysis Techniques and Facilities Available to the Colloid and Interface Scientist

### Colloidal Studies

Scientific Services offer a range of colloidal characterisation equipment to aid scientists and industry alike including:

#### Particle Counter and Sizer.

Measures particle count and size information for particles suspended in a specified volume of liquid in the range of 0.5 to 500 $\mu$ m. This instrument can be used with very dilute samples.

#### Particle Sizer – Laser Diffraction

Provides average particle size results over a particle size distribution range of 0.02 $\mu$ m to 2000 $\mu$ m.

#### Zeta Potential and Particle Size Measurement

The Malvern Zetasizer Nano instrument can measure a combination of particle size (0.6nm to 6 $\mu$ m), zeta potential and molecular weight ( $1000^{10^2}$  x 10<sup>7</sup>Da). Measurements can be performed with little or no dilution while an auto titration unit allows pH sweeps or titration measurements to be conducted rapidly, reproducibly and economically.

#### The Colloidal Dynamics Acoustosizer

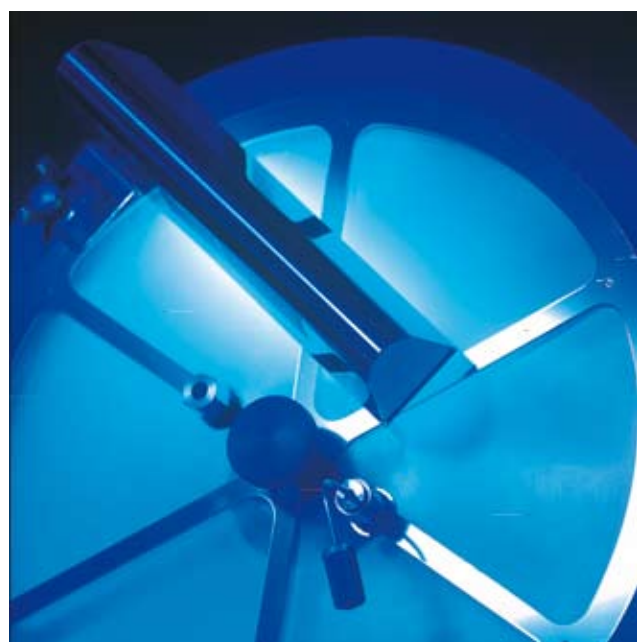
Allows measurement of Zeta potential in real systems (>5wt% solids, <38 $\mu$ m particle size). Also fitted with burettes to allow rapid titration measurements under automated software control.

#### Particle Contact Angle Meter

Measures the equilibrium capillary pressure (ECP) of liquids in packed particle beds (or in porous media) from which the particle contact angle can be derived. Ideal for particles possessing a narrow size distribution in the range 20 $\mu$ m to 500 $\mu$ m.

#### Shear Rate Controlled Rheometers

Provide rapid characterisation of mobile and viscous liquids, gels, pastes, slurries solids (in torsion) and molten polymers.



#### Stress Controlled Rheometers

Used in the characterisation of viscosity and elasticity of fluids (polymer melts, suspensions and emulsions) in a temperature range of -90°C to +400°C.

#### 300MHz Nuclear Magnetic Resonance (NMR)

spectrometer that has the capability to study both solution-state (by the majority of 1D and 2D experiments) and solid-state (cross polarisation/magic angle spinning) samples targeting nuclei such as <sup>1</sup>H, <sup>13</sup>C, <sup>15</sup>N, <sup>27</sup>Al, <sup>29</sup>Si and <sup>31</sup>P. NMR can be used for quantitative measurements and is ideally suited for product quality control, structure elucidation, evaluation of compound purity and for monitoring changes in materials that may occur over time.

#### *Unique Characteristics Include:*

- Multinuclear solution and solid state probes.
- Variable temperature capability for solutions (ranging from -80°C to +150°C)
- Solution-state NMR particularly applicable to the petroleum (motor oils, lubricant oils, recycled oils, greases, waxes and vegetable oils), pharmaceutical and polymer industries
- Solid-state NMR analyses cover a wide range of applications that cannot be addressed by solution-state experiments. Examples include the analysis of aluminosilicates and clays, polymers (EPDM, rubbers, nylons, polyurethanes, PVC), coals, soil, gaskets and seal materials, sugars and any other insoluble materials.

#### **Interfacial and Surface Analysis**

Interfaces and surfaces play an important role in many technical processes and products. Structures on the microscopic and molecular levels often have a strong influence on the macroscopic properties of products and developments. Listed below are the key surface analysis techniques available at The Wark to benefit the (bio) interface and surface scientist:

##### **Atomic Force Microscope**

This instrument measures the surface morphology of samples and forces between the probe tip and a surface. Experiments can be conducted in air or in solution. Contact and tapping modes are available. Two scanners are available, "E" scanner - 14 x 14µm XY scan range, 3µm Z scan range and the "J" scanner - 110 x 110µm XY scan range, 6µm Z scan range.

##### **Imaging Ellipsometer**

The Imaging Ellipsometer can follow dynamic phenomena in real time, such as the spreading of drops on liquid and solid surfaces, and surface chemical reactions. The system can also be used as a Brewster Angle Microscope for video intensity measurements at up to 100 frames per second. An ellipsometric image of the sample surface allows measurement of layer thicknesses smaller than the wavelength of the illuminating light.

##### **Wilhelmy Wetting Balance**

The Dataphysics DCAT 21 wetting balance can measure static and dynamic contact angles and interfacial tensions of powders, fibre bundles and plates using the Washburn, Wilhelmy plate and Dunoüy ring techniques. Measurement range of 1 to 1,000mN/m (0.1mg to 210g) while speed can be controlled over the range of 0.7µm/s to 500mm/min

**Optical Microscope equipped with an Olympus BH2 metallurgical microscope, Sony low light CCD camera and Optimus image analysis system.**

Used for measuring particle size, shapes and surface deposits (>1µm).

#### **Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)**

NanoToF utilises a pulsed primary ion beam, typically Gold (Au<sup>+</sup>), to remove atoms and molecules from the very outermost surface of the sample. This technique is used to analyse organic and inorganic materials (films, particles, cross sections). It provides information about the sample surface, however, it can also be used to monitor the variation of chemical composition with sputtering time (depth profile).

#### *Unique Characteristics*

- High surface specificity (<20 Å)
- High surface sensitivity (ppm to ppb detection limit)
- High mass resolution (M/ΔM for Silicon up to 9000)
- High lateral resolution (120nm)
- 30 keV Au<sup>+</sup> Liquid metal ion gun for high spatial resolution and high mass resolution
- Caesium (Cs<sup>+</sup>) ion gun that provides higher primary ion currents for trace elemental analysis and low voltage depth profiling
- 20 keV C60 ion gun that provides spectroscopy, imaging, low damage surface cleaning and sputter ion depth profiling for many organic and polymeric materials
- 5 kV argon/oxygen gas sputter ion gun for low voltage depth profiling

#### *Advantages*

- Non destructive
- Small surface required
- Unrestricted by charging effects (allows analysis of conducting and insulating materials)
- Able to detect the entire periodic table

#### **X-Ray Photoelectron Spectroscopy (XPS)**

XPS is the most commonly used technique for investigating elements in the outermost atomic layers of materials and is used to quantitatively determine valence states, bonding environments and the elemental composition of surface layers. This instrument has the ability to acquire spectra with high energy resolution and can perform chemical imaging at high spatial resolution down to dimensional scales of a few microns.



#### Unique Characteristics

- Multiple X-ray source (mono Al and Mg/Al dual anode)
- Cold and hot stages
- Elemental mapping (spatial resolution < 10  $\mu\text{m}$ )
- Small area analysis
- Depth profiling via ion beam etching
- Unrestricted by charging effects (allows analysis of conducting and insulating materials).

#### Advantages

- Surface sensitive (< 100  $\text{\AA}$ , variable using angle resolved XPS)
- Quantitative analysis
- Elemental and chemical state identification
- Non destructive



#### High Resolution Scanning Electron Microscope (SEM)

This instrument can produce images of a surface with a finely focused electron beam. The achievable magnification of the SEM is typically around several hundreds of thousands times, enabling detection of features that are 50nm apart. This instrument is equipped with an energy dispersive x-ray spectrometer (EDS) to perform quantitative elemental analysis.

#### Synchrotron Radiation Analysis

The Wark routinely accesses the Australian Synchrotron facility in Melbourne together with other facilities around the world as required including BESSY (Berlin, Germany), the Advanced Photon Source (Chicago, USA), Aladdin (Madison, Wisconsin USA), NSRRC (Taipei, Taiwan), the Photon Factory (Tsukuba, Japan), Elettra (Trieste, Italy) and the Canadian Light Source (Saskatoon, Canada). The unique, controllable properties of synchrotron light enable a large range of analyses that are conventionally difficult due to detection limits, spatial and/or spectral resolution and light intensity. Some measurements may only be performed using a synchrotron source.

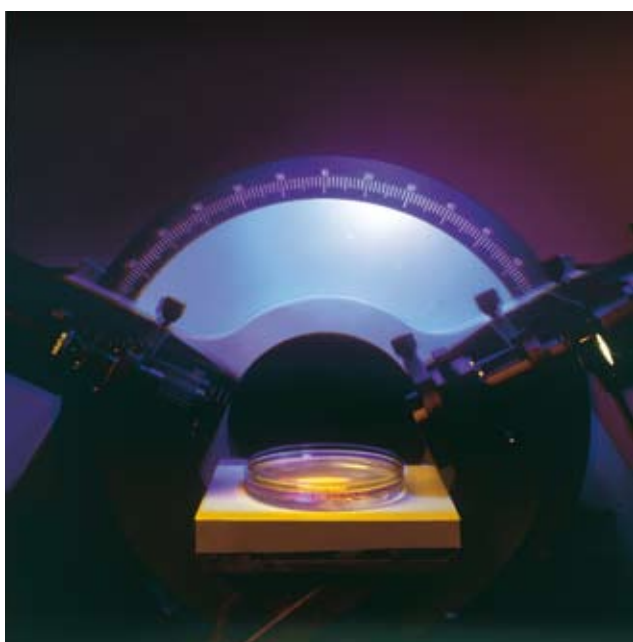
Techniques commonly employed include synchrotron radiation XPS (SR-XPS), soft and hard X-ray absorption spectroscopies (NEXAFS/XANES, EXAFS), imaging photoemission and adsorption (PEEM, XPEEM) micro-X-ray fluorescence spectroscopy ( $\mu$ -XRF), Infrared (IR) spectroscopy and imaging, Small and Wide Angle X-ray Scattering (SAXS/WAXS) and X-ray microtomography ( $\mu$ -CT). Synchrotron techniques are especially powerful when combined with the conventional array of advanced instrumental measurements available at The Wark.

#### Fourier Transform Infrared Spectrometer (FTIR)

Used to identify a variety of surface chemical functional groups, analyse molecular adsorbed species and oxidation. The FTIR can be used in a number of modes including: Photo-acoustic, DRIFT, Transmission and ATR.

#### Nanoindenter

This is an excellent tool to measure the mechanical properties of thin surface films on nanometer length scales. More specifically, the nanoindenter makes a small indentation on the surface of a material to measure



properties such as wear, thickness, hardness and elasticity. Elastic modulus and deformation behaviour are determined from an analysis of the force vs. displacement response measured during indentation of the material.

## Surface Modification

Modification of surfaces allows for the production of far superior products in terms of reduced wear, increased corrosion resistance, improved optical properties and better biocompatibility to name a few. Obviously, as surface modification methods improve, there are many more possible applications of such surface tailoring methods. Listed below are the key surface modification facilities available at The Wark to benefit interested parties:



### Multi-Station Vacuum Coating Unit

This instrument enables fabrication of single or multiple layer structures of thin film coatings of a wide range of compositions. One of the three stations contains a microwave-powered plasma deposition unit allowing deposition of plasma polymer coatings at unprecedented rates of up to 100nm/sec.

Such high deposition rates makes it feasible to consider economic application of plasma polymer coatings of several  $\mu\text{m}$  or even several tens of  $\mu\text{m}$  thickness. The other two stations enable deposition of ceramic and metallic coatings. Dual vapour delivery can be used to deposit alternating coating structures or coatings with graded compositions. The larger deposition chamber can hold and coat substrates with dimensions of up to 400 x 500mm.

### Plasma Reactors

Used to promote an assortment of surface reactions/modifications via the introduction of various gases and vapours. They are also used as an elegant surface cleaning tool.

### UV Spectroirradiator

A 300W xenon lamp generates a continuous range of wavelengths between 220nm and 700nm which can be utilised in different ways. Firstly as 'white' light (the full, unfiltered range of wavelengths) and secondly as monochromatic light by 'dialing-up' the wavelength of interest. Particularly useful in studying the photochemistry of modified photo-responsive surfaces.

# Mini Pilot Plant

The Wark operates a sophisticated Flotation Mini Pilot Plant. The Mini Pilot Plant is unique in Australia due to its extremely small scale that affords the plant a degree of control and flexibility that is impossible to achieve with conventional pilot plants.

This new facility enables The Wark to test and evaluate new separation processes for a wide range of mineral commodities, using very small sample sizes (and less energy), and providing results at a significantly lower cost than full-scale pilot plants, which are often not feasible or accessible. This makes the Mini Pilot Plant an important tool, not only for mineral research but also for commercial ventures.

The Mini Pilot Plant has the ability to not only monitor but, most significantly, to closely control the chemical and physical conditions in flotation that affect optimal mineral separations on a continuous basis. Clients are able to assess potential processing options at the pre-feasibility stage of a mine site development, thus minimising the risks of expensive metallurgical errors and wasted energy as the mine develops, and therefore also minimising environmental impacts.

The plant is equally applicable to development of material processing systems across a broad range of industry sectors with similar processing needs, including energy (coal), soil remediation and plastics recycling.

The Mini Pilot Plant fills the long gap between laboratory scale tests and larger pilot plant trials. It is able to operate in continuous mode with roughing, regrinding and column flotation at a feed rate of only 5kg/h.

The low throughput rate of solids allows for maximum data recovery with minimum sample and resource input. This means much lower costs and increased flexibility when compared to conventional pilot plant trials. The Mini Pilot Plant is specifically designed to make use of samples as small as 100-300kg such as may be obtained from exploration drilling campaigns.





The Mini Pilot Plant consists of the following modulated components, which can be used to create numerous process configurations:

<ul style="list-style-type: none"> <li>• 10 - 15kg Batch Rod Mill</li> </ul>	<ul style="list-style-type: none"> <li>- Stainless steel construction</li> <li>- Variable speed drive</li> <li>- Electro-hydraulic tilt system to facilitate loading and discharge</li> </ul>
<ul style="list-style-type: none"> <li>• Slurry Holding Tank</li> </ul>	<ul style="list-style-type: none"> <li>- All stainless steel construction</li> <li>- Variable speed agitators</li> <li>- Recirculation pump system</li> <li>- Continuous pH monitoring</li> <li>- Able to operate at elevated temperatures</li> </ul>
<ul style="list-style-type: none"> <li>• MPP Feed Tank</li> </ul>	<ul style="list-style-type: none"> <li>- All stainless steel construction</li> <li>- Variable speed agitators</li> <li>- Recirculation pump system</li> <li>- Magnetic flow meter</li> <li>- Automatic slurry feed sampler</li> </ul>
<ul style="list-style-type: none"> <li>• Continuous Flotation Machine (CFM)</li> </ul>	<ul style="list-style-type: none"> <li>- Twelve 1.7L Denver style cells (crowders can be used to reduce cell volume to 1L)</li> <li>- Variable speed agitators</li> <li>- Digital air flow controllers</li> <li>- Launder water with flow measurement</li> <li>- Continuous pH monitoring</li> <li>- pH and <math>E_H</math> control</li> </ul>
<ul style="list-style-type: none"> <li>• Flotation Column</li> </ul>	<ul style="list-style-type: none"> <li>- Clear PVC column body - 7.5 cm <math>\varnothing</math> x 1.5 m high</li> <li>- Adjustable froth height</li> <li>- Automatic level control</li> <li>- Digital air flow control</li> <li>- Digital wash water flow control</li> </ul>
<ul style="list-style-type: none"> <li>• Regrind Mill</li> </ul>	<ul style="list-style-type: none"> <li>- Continuous stirred mill</li> <li>- Stainless steel chamber and shaft</li> <li>- Variable speed drive</li> <li>- Stainless steel or ceramic grinding media</li> </ul>
<ul style="list-style-type: none"> <li>• Reagent Dispensing System</li> </ul>	<ul style="list-style-type: none"> <li>- 16 precision metering pumps</li> <li>- Fully controlled by the PLC data system</li> </ul>



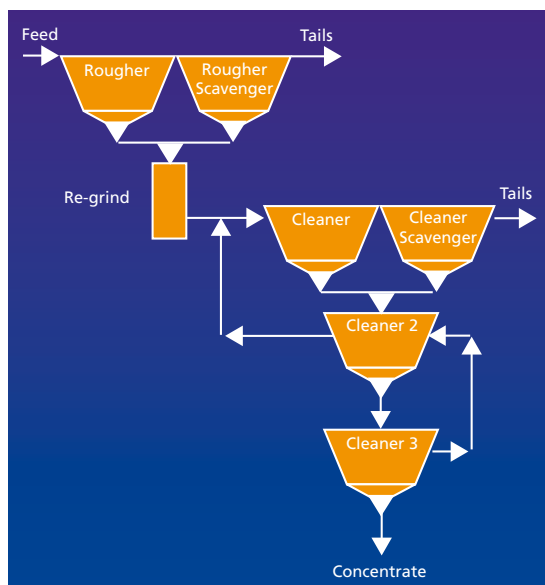
The benefits over laboratory scale testing are considerable, with operating procedures and parameters emulating industrial practices. This means that all data and results strongly represent likely plant experiences and can be used to design the full-scale processing plant with confidence

Virtually every aspect of the system is either controlled or at least monitored by the PLC data system from the feed flow into the cells to the regrind mill speed. This allows operators to spend more time optimising and stabilising the system rather than recording data from the separate units.

Data is automatically logged and can be retrieved at any time for data processing. Critical process parameters can be selected and monitored via a real-time graphing system.

The versatile nature of the Mini Pilot Plant enables operators to change configurations to represent almost any process flowsheet.

The ease at which the Mini Pilot Plant can be configured allows for examination of various process flow designs to optimise the mineral separation process. Feed and final products are monitored continuously throughout the trial and used to determine process stability.



Owing to the flexibility of the setup, once the process has reached steady state, the intermediate product streams are easily accessible, allowing for precise sampling. The information and data obtained from these trials are invaluable in determining the efficiency of the process.

The Mini Pilot Plant is able to simulate the continuous operation of a basic rougher / scavenger / cleaner circuit with a level of confidence that exceeds those from standard locked cycle tests.



## Minerals Processing Expertise

The Wark personnel have comprehensive experience in all areas of mineral separation from flotation of ores containing base and precious metals to physical (magnetic and gravity) hydrometallurgical and pyrometallurgical processing of a wide range of materials.

Together with a wide range of plant experience, this enables the staff at The Wark to get the very most out of the Mini Pilot Plant.

Clients can also choose to either take charge of the technical decisions when running trials or to let The Wark staff manage all aspects of the trials and take advantage of this wealth of knowledge.

## Analytical Facilities

A distinct advantage of performing trials using the Mini Pilot Plant is the extensive range of analytical services available at The Wark. This provides the ability to examine samples with minimal degradation and fast



turn around times. The following facilities as well as highly skilled operators are available at The Wark:

- Particle Sizing
- Scanning Electron Microscope with Energy Dispersive X-Ray Analysis
- Time of Flight Secondary Ion Mass Spectroscopy
- FTIR, UV-Vis and AAS
- X-Ray Photoelectron Spectroscopy
- X-Ray Diffraction
- Rheology
- XRF Analysis
- QEMSCAN

## Polymer Identification and Characterisation

Scientific Services offers a range of polymer identification and characterisation equipment available to industry including:

- **300MHz Nuclear Magnetic Resonance Spectrometer (NMR)** An important technique used to obtain physical, chemical, electronic and structural information about a molecule for samples both in the solution and solid state. The sample can be organic or inorganic in nature. The following nuclei can be targeted: Solution-state  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$ ,  $^{27}\text{Al}$ ,  $^{29}\text{Si}$  and  $^{31}\text{P}$ . Solid-state  $^{13}\text{C}$ ,  $^{15}\text{N}$ ,  $^{27}\text{Al}$ ,  $^{29}\text{Si}$  and  $^{31}\text{P}$ .
- **Fourier Transform Infrared Spectrometer (FTIR)** Used to identify a variety of chemical functional groups, analyse molecular adsorbed species and oxidation. The FTIR can be used in a number of modes including: transmission, DRIFT and photo-acoustic. Measured spectra can be referenced to a computer spectral database to help identify unknown materials.
- **Raman Spectroscopy** Used to identify and characterise materials such as polymers, minerals and forensic samples. Characteristic molecular vibrational frequencies provide a fingerprint with which the Raman Spectrometer can identify the sample at hand. This instrument also possesses a surface mapping capability with  $2\mu\text{m}$  resolution.
- **UV-Visible-Near Infrared Spectrometer** An important technique for studying molecular adsorption, reaction kinetics (solutions and surfaces) and molecular identification.

## Thermal Analysis

Thermal analysis is the application of a precision controlled temperature program to a material and measurement of the response of that material within the  $-150^\circ\text{C}$  to  $+1500^\circ\text{C}$  temperature range. Instruments available to industry include:

- **Thermogravimetric Analyser (TGA)** TGA measures weight changes in a material as a function of





temperature (or time) under a controlled atmosphere. Its principal uses include measurement of a material's thermal stability and identification of unknown composition. Modulated TGA (MTGA) is also available and provides information regarding sample degradation and kinetics thereof.

- **Differential Scanning Calorimeter (DSC)** Measures the amount of energy absorbed or released by a sample as it is heated, cooled or held at constant temperature. Measures heat flows which are associated with transitions in the material as a function of temperature and time. Melting point, glass transition temperature and crystallinity temperature can be obtained. Modulated DSC (MDSC) is also available and allows weak transitions to be measured precisely such as heat capacitance and thermal conductivity. Furthermore, Photo DSC and Pressure DSC are also available and provide information regarding the photo-curing of materials and the curing of solvent based coatings respectively.
- **Simultaneous Differential Thermal Analyser (SDT)** SDT provides both TGA and DSC information in a single run from room temperature up to 1450°C.
- **Dynamic Mechanical Analyser (DMA)** DMA measures the mechanical properties (bending, tension, compression, and shear) and phase behaviour of blends and composites as a function of time, temperature, and frequency.

### Rheology

Rheology enables detailed characterisation of Non-Newtonian Fluids (polymer solutions and melts, suspensions, emulsions and foams) including the determination of flow curves (viscosity vs shear rate), yield stress values, dynamic measurements of viscoelasticity, thixotropy, normal forces and pipe flow. Instruments available to industry include:

- **Shear Rate Controlled Rheometers** Provide rapid characterisation of mobile and viscous liquids, gels, pastes, slurries solids (in torsion) and molten polymers.
- **Stress Controlled Rheometers** Used in the characterisation of viscosity and elasticity of fluids (polymer melts, suspensions and emulsions) in a temperature range of -90°C to +400°C.

### Surface Chemical Composition

The following characterisation techniques provide important chemical information concerning polymer surfaces, thin-film coatings and defects:

- **Time-of-Flight Secondary Ion Mass Spectrometer (TOF-SIMS)** The technique has ultra-high sensitivity to surface layers (one atomic thickness), and detection of atomic concentrations in the high ppb range. Surface layers of insulating materials, including minerals, polymers, organic, and biological materials, can be analysed readily (including depth profiling). The instrument also allows imaging of secondary ions to submicron spatial resolution.
- **X-Ray Photoelectron Spectrometer (XPS)** Exceptional technique commonly used for investigating elements in the outermost atomic layers of materials and is used to quantitatively determine valence states, bonding environments and the elemental composition of surface layers 1-5nm in depth. XPS also enables both depth profiling and surface element and chemical state mapping. This instrument has the ability to acquire very high energy resolution spectra and can perform chemical imaging at high spatial resolution down to dimensional scales of a few microns.

- **Scanning Electron Microscope (SEM)** This instrument can produce images of a surface with a finely focused electron beam. The achievable magnification of the SEM is typically around several hundreds of thousands times, enabling detection of features on the nanometer scale. This instrument is equipped with an energy dispersive x-ray analyser (EDX) to perform quantitative elemental analysis.
- **Nanoindenter** This is an excellent tool to measure the mechanical properties of thin films on nanometer length scales. More specifically, the nanoindenter makes a small indentation on the surface of a material to measure properties such as wear, thickness, hardness and elasticity. Elastic modulus and deformation behaviour are determined from an analysis of the force vs. displacement response measured during indentation of the material.

### Particle Sizing and Light Scattering

Of particular importance to the polymer scientist is the ability to analyse polymers in solution or in the emulsion state. Listed below are a range of particle sizing and light scattering equipment available to industry:

- **Particle Counter and Sizer** Measures particle count and size information for particles suspended in a specified volume of liquid.
- **Particle Sizer - Laser Diffraction** Provides average particle size results over a particle size distribution range of  $0.02\mu\text{m}$  to  $2000\mu\text{m}$ . This can be performed in both aqueous and organic based dispersion media.
- **Zeta Potential and Particle Size Measurement** The Zetasizer Nano instrument can measure a combination of particle size ( $0.6\text{nm}$  to  $6\mu\text{m}$ ), zeta potential and molecular weight ( $1000 - 2 \times 10^7$  Da). Measurements can be performed with little or no dilution.





### Polymer Processing Facilities

- Brabender mixing facilities and extrusion equipment are available for examining polymer processing, melt and flow behaviour, coatings and various manufacturing processes.
- **Compression and Injection Moulding equipment** Compression and injection moulding is the process that moulds molten plastic, through compression, into the desired shape of the moulding. Injection moulding is the most commonly used process for moulding plastics because it gives a good surface finish and can be used for very complex mouldings.

Other forms of analysis pertinent to the polymer industry provided by Scientific Services include:

- **Tensile Testing Machine** Measures tensile strength of a sample, or more specifically, the force required to pull a sample to the point where it breaks. Can provide further information such as yield strength, ultimate strength and breaking strength
- **Accelerated UV Weathering Instrument.** Used to simulate the exposure of polymer samples to external weather conditions. Incorporates both light and moisture exposure
- **Optical Microscope** equipped with an Olympus BH2 metallurgical microscope, Sony low light CCD camera and Optimus image analysis system. Used for measuring particle size, shapes and surface deposits (>1µm)

# Surface Analysis and Characterisation

## PHI TRIFT V Nano Time-of-Flight Secondary Ion Mass Spectrometry (NanoToF)

NanoToF utilises a pulsed primary ion beam, typically Gold (Au<sup>+</sup>), to remove atoms and molecules from the very outermost surface of the sample. This technique is used to analyse organic and inorganic materials (films, particles, cross sections). It provides information about the sample surface, however, it can also be used to monitor the variation of chemical composition with sputtering time (depth profile).

### Unique Characteristics

- High surface specificity (<20 Å)
- High surface sensitivity (ppm to ppb detection limit)
- High mass resolution (M/ΔM for Silicon up to 9000)
- High lateral resolution (120nm)
- 30 keV Au<sup>+</sup> Liquid metal ion gun for high spatial resolution and high mass resolution
- Caesium (Cs<sup>+</sup>) ion gun that provides higher primary ion currents for trace elemental analysis and low voltage depth profiling
- 20 keV C60 ion gun that provides spectroscopy, imaging, low damage surface cleaning and sputter ion depth profiling for many organic and polymeric materials
- 5 kV argon/oxygen gas sputter ion gun for low voltage depth profiling

### Advantages

- Non destructive
- Small surface required
- Unrestricted by charging effects (allows analysis of conducting and insulating materials)
- Able to detect the entire periodic table

## X-Ray Photoelectron Spectroscopy (XPS)

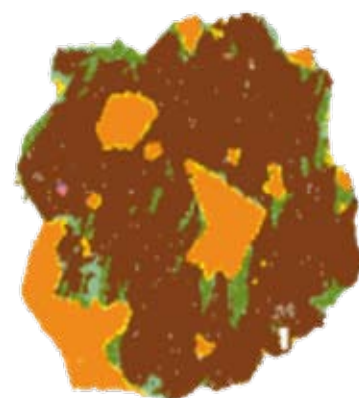
XPS (Kratos Axis Ultra) is the most commonly used technique for investigating elements in the outermost atomic layers of materials and is used to quantitatively determine valence states, bonding environments and the elemental composition of surface layers. This instrument has the ability to acquire spectra with high energy resolution and can perform chemical imaging at high spatial resolution down to dimensional scales of a few microns.

### Unique Characteristics

- Multiple X-ray source (mono Al and Mg/Al dual anode)
- Cold and hot stages
- Elemental mapping (spatial resolution < 10 μm)
- Small area analysis
- Depth profiling via ion beam etching
- Unrestricted by charging effects (allows analysis of conducting and insulating materials)

### Advantages

- Surface sensitive (< 100 Å, variable using angle resolved XPS)
- Quantitative analysis
- Elemental and chemical state identification
- Non destructive



150.0 μm

Orange	Chalcopyrite	Red	Dolomite
Yellow	Pyrite	Blue	Calcite
Brown	Magnetite	Green	Mg-Fe-silicate
Grey	Goethite		

Mineral map of a particle as obtained using QEMSCAN®

## QEMSCAN®

QEMSCAN® is the Quantitative Evaluation of Minerals using a Scanning Electron Microscope. This fully automated instrument integrates electron beam technology with the latest liquid nitrogen-free energy dispersive x-ray detectors (EDX). QEMSCAN® is a powerful analytical technique particularly orientated towards the study of rocks, cores, plant products and dust relating to the mining industry. The technique is equally applicable to the study of particulates from other sources. Integrated iDiscover™ software controls the operation of the measurements and presents the generated data for analysis.



### Unique Characteristics

- Backscatter electron imaging for mineral and phase analysis
- False colour images of particles representing mineralogy and texture
- Capable of analysing a wide range of samples, from thin sections of a core sample to soil and sand, from manufactured products to dust

### Advantages

- Capable of achieving 200,000 counts/second, using EDX detectors
- Quantitative and repeatable
- Quick mineral identification
- Provides mineral association, locking and liberation characteristics, elemental deportment, particle texture, shape, size and density distribution information

### Sample Preparation

- Sputter coater and thermal evaporator for SEM specimen preparation
- Struers Microtome for precision cutting
- Struers TegraSystem for polished cross-sectioning

## Scanning Electron Microscopy (SEM)

The Scanning Electron Microscope can produce images of a surface with a finely focused electron beam. The achievable magnification of the SEM is typically around several hundreds of thousands times, enabling detection of features that are 50 nm apart.

### Unique Characteristics

- Schottky field emission source for high resolution imaging
- Backscatter electron imaging for phase analysis
- Large analysis chamber to accommodate large samples
- Windowless energy dispersive X-ray spectrometer (EDS) to perform quantitative elemental analysis (down to Boron)
- Digital output of microscope images and EDS results

### Advantages

- Elemental mapping of the surface area of interest
- Area and spot analysis
- Bulk elemental analysis (up to ppm detection limit [EDS])

## Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is used to identify a variety of chemical functional groups, analyse molecular absorbed species, oxidation, reaction kinetics and adsorption densities. The FTIR can be used in a number of modes including: transmission, DRIFT, ATR and photo-acoustic.

### Unique Characteristics

- 400-4000 wavenumber range
- Photo-acoustic (PA) mode
- Attenuated Total Reflection (ATR) mode for thin and small volume samples

### Advantages

- Analyse aqueous solutions
- Analyse solid samples (powder, pellets or chunks)
- Non destructive
- Detects molecular functional groups
- Complementary analysis technique to Raman spectroscopy

## X-Ray Diffraction (XRD)

X-ray diffraction can provide information based on vertical  $\theta$ - $\theta$  Bragg-Brentano law by using a fine focused X-ray beam bombarded on a sample surface. The sample remains horizontal during measurement, which allows analysis of powders. Depth profiling is also possible using GA-XRD (Glancing-Angle X-Ray Diffraction).

### A wide range of analytical techniques

X-ray diffraction is a versatile and non-destructive technique that reveals detailed structural and chemical information about the crystallography of materials.

- Identification of single or multiple phases in an unknown sample
- Quantification of known phases of a mixture
- Crystallography – solving crystal structure
- Surface and thin film analysis (surface roughness and film thickness)
- Texture analysis
- Residual stress analysis



### **A broad range of materials**

Natural or manufactured materials of organic or inorganic chemistry can be characterised with this technique. Some typical materials are listed here:

- Chemicals
- Pharmaceutical substances
- Environmental dusts
- Asbestos
- Rocks
- Clay minerals
- Zeolites
- Polymers
- Metals
- Cement and building materials
- Forensics
- Pigments

### **X-Ray Fluorescence (XRF)**

XRF is an analytical method for determining the chemical composition/elemental analysis of all kinds of materials. The materials can be in solid or liquid form. The method is fast, accurate, non-destructive and usually only requires a minimum of sample preparation. Applications are very broad and include the metal, cement, oil, polymer, plastic, glass, pharmaceutical and food industries along with mining, mineralogy and geology.

#### **Advantages**

- Quantitative bulk elemental analysis (calibration is required with your specific matrix)
- Extremely fast qualitative analysis
- Detect elements in the range from sodium to uranium
- Auto-sampler enables high sample throughput and fast turnaround once calibration has been established

### **Synchrotron Radiation Analysis**

The Wark routinely accesses the Australian Synchrotron facility in Melbourne together with other facilities around the world as required including BESSY (Berlin, Germany), the Advanced Photon Source (Chicago, USA), Aladdin (Madison, Wisconsin USA), NSRRC (Taipei, Taiwan), the Photon Factory (Tsukuba, Japan), Elettra (Trieste, Italy) and the Canadian Light Source (Saskatoon, Canada). The unique, controllable properties of synchrotron light enable a large range of analyses that are conventionally difficult due to detection limits, spatial and/or spectral resolution and light intensity. Some measurements may only be performed using a synchrotron source.

Techniques commonly employed include synchrotron radiation XPS (SR-XPS), soft and hard X-ray absorption spectroscopies (NEXAFS/XANES, EXAFS), imaging photoemission and adsorption (PEEM, XPEEM) micro-X-ray fluorescence spectroscopy ( $\mu$ -XRF), Infrared (IR) spectroscopy and imaging, Small and Wide Angle X-ray Scattering (SAXS/WAXS) and X-ray microtomography ( $\mu$ -CT). Synchrotron techniques are especially powerful when combined with the conventional array of advanced instrumental measurements available at The Wark.

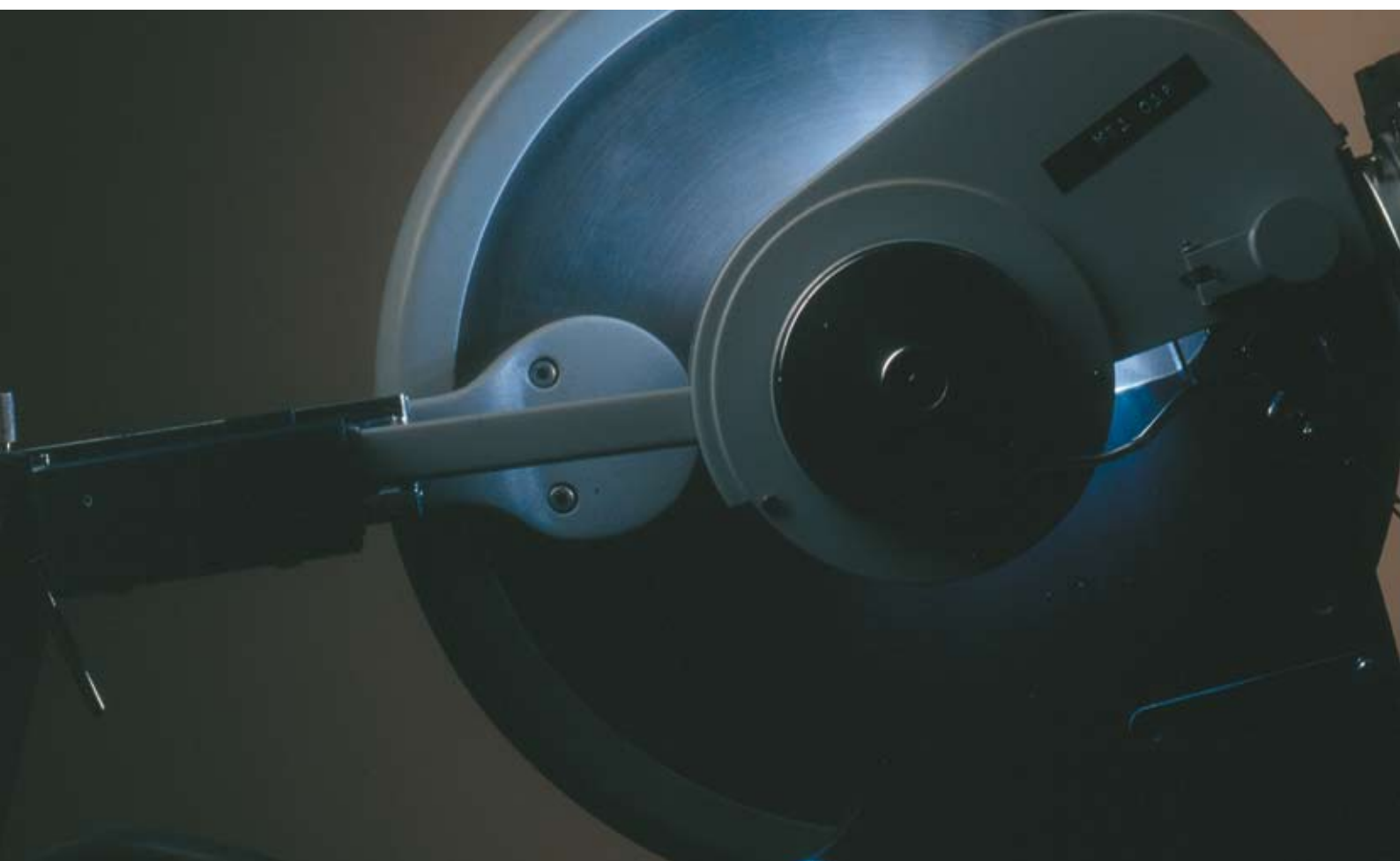




## Nuclear Magnetic Resonance Spectroscopy (NMR)

NMR spectroscopy uses radiofrequency radiation to induce transitions between different nuclear spin states of samples residing in a static magnetic field. The 300MHz NMR spectrometer (Bruker-BioSpin) has the capability to study both solution-state (by the majority of 1D and 2D experiments) and solid-state (cross polarisation/magic angle spinning) samples targeting nuclei such as  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$ ,  $^{27}\text{Al}$ ,  $^{29}\text{Si}$  and  $^{31}\text{P}$ .

- Solution-state NMR particularly applicable to the petroleum (motor oils, lubricant oils, recycled oils, greases, waxes and vegetable oils), pharmaceutical and polymer industries
- Solid-state NMR analyses cover a wide range of applications that cannot be addressed by solution-state experiments. Examples include the analysis of aluminosilicates and clays, polymers (EPDM, rubbers, nylons, polyurethanes, PVC), coals, soil, gaskets and seal materials, sugars and any other insoluble materials



NMR can be used for quantitative measurements and is ideally suited for product quality control, structure elucidation, evaluation of compound purity and for monitoring changes in materials that may occur over time.

### Unique Characteristics

- Multinuclear solution and solid state probes
- Variable temperature capability for solutions (ranging from  $-80^{\circ}\text{C}$  to  $+150^{\circ}\text{C}$ )

### Advantages

- Only small quantities of sample required (10-100mg for solution. 0.5-1.0g for solid)
- Non destructive
- Complementary analysis technique to FTIR spectroscopy
- Reasonably priced

# Microfluidics and Nanofabrication

The Microfluidics and Nanofabrication section is the South Australian node of the Australain National Fabrication Facility (ANFF-SA).

Our capabilities focus on the design and fabrication of microfluidic chips and micro-engineered structures, coupled with their subsequent functionalisation characterisation.

Our fabrication facilities are complemented by the surface coating and functionalisation facilities at The Wark, so we can produce next-generation microengineered devices with enhanced performance, through precise tailoring of both surface geometry and chemistry.

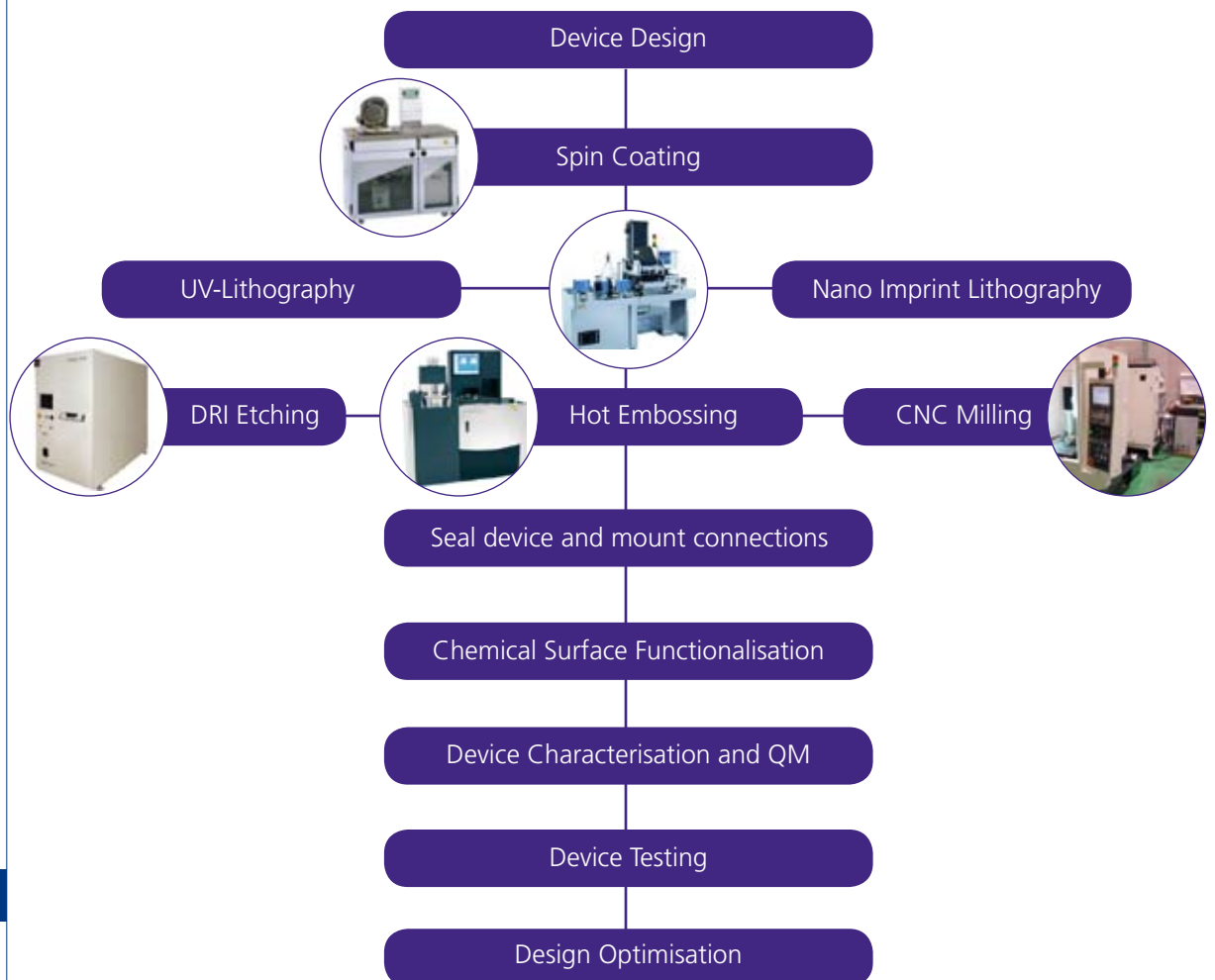
Using the multi-million dollar state-of-the-art suite of equipment in the our clean rooms, we have the ability to tailor fabrication to individual needs, whilst still providing that essential cost effective solution you demand.

## New Fabrication Capabilities in Australia

Here in Scientific Services we are focused on developing new and improved microfabrication methods and capabilities in order to better service our customers.

We can take your design concept and translate it into a microengineered device, or we can provide fundamental concept and proof of principle assistance. With significant industrial consulting experience behind us we offer flexibility and will provide a rapid response to your requirements. Please feel free to contact us to further discuss your requirements.

### Fabrication of a Device





Here are just some examples of our equipment and recently fabricated devices to give a snapshot of our capabilities.

## Micro CNC Milling

### Kira 2M SuperMill

The Kira SuperMill-2M has a positional accuracy of 0.01  $\mu\text{m}$  (or 10 nm) with double spindles, a 50,000 rpm main spindle and a 120,000 rpm finishing spindle. It offers tool makers and researchers alike an outstanding next generation high speed precision milling capability.

This new technology was developed in collaboration with the University of Tokyo and was installed at The Wark early in 2009.

Designed specifically for single part and small series production milling of steel, non-ferrous, plastics and ceramics, the simultaneously controlled 3-axis system allows true interpretation of three-dimensional structures providing capability in metals, polymer or glass:

- State-of-the-art Fanuc linear motors with the Fanuc 31i nano CNC control.
- Temperature controlled environment within the room to 1°C with additional temperature compensation to the equipment.
- Blum laser tool measurement system offering precision tool geometry and wear monitoring
- Maximum work piece dimensions of 270 x 200 x 170 mm
- Maximum load capacity of 4 Kg
- Automatic tool changer (ATC) with an 8 holder magazine.

*Machining capability for:*

- Micro injection moulding tools
- Hot embossing tools
- Micro channels and features



## Bonding and Hot Embossing

The Wark has an integrated Substrate Bonding System that allows both Hot Embossing and Bonding.

Hot Embossing is the stamping of a pattern into a polymer or glass softened by raising the temperature of the material just above its glass transition temperature. The non-consumable stamp used to define the pattern may be made in a variety of ways including micromachining using our Kira Micromill Machining Centre. The benefits of this technique are the potential to fabricate the low cost devices with micron-scale features.

Bonding is one of the most critical and challenging steps in the fabrication of a microfluidic device. This is the final step that seals the device to allow fluid flow through the channels, and is most simply performed by the addition of a cover plate of the same material. Failure to achieve a good hermetic seal will leave a device with the potential to leak.





### **EVG 520 Hot Embosser/Bonder**

- Configurable for all wafer bonding processes such as anodic, thermo compression, fusion bonding, or low temperature plasma bonding
- Can handle wafers up to 200 mm
- Fully programmable bond/embossing process parameters
- Independent top and bottom heating and cooling after bonding

#### *Capability for:*

- Hot embossing polymers and glass – a unique Australian Capability!
- Bonding glass

### **Deep Reactive Ion Etching**

#### **ULVAC 520NLD Deep Reactive Ion Etcher**

- From several 10nm to several 10  $\mu\text{m}$  etching
- Extremely high uniformity by control of magnetic coil field

#### *Etching capability for micro/nano fabrication*

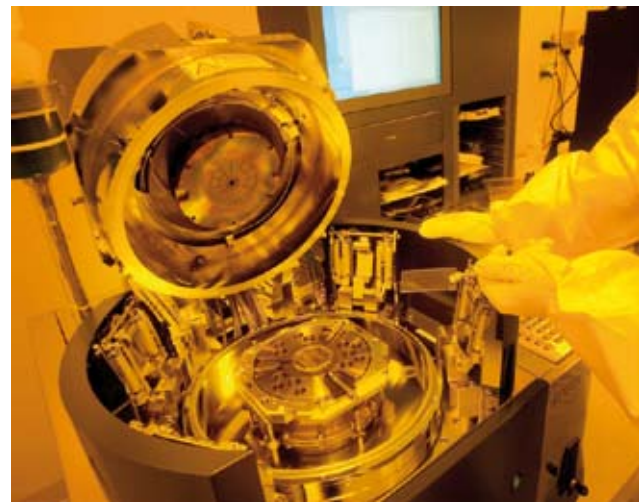
- Dielectric materials, Quartz,  $\text{TiO}_2$ ,  $\text{LiNbO}_3$
- Optical devices (Arrayed Waveguide Gratings, Optical Amplifier, etc)
- Micro-Total Analysis Systems ( $\mu\text{-TAS}$ )
- Microfluidic chips

### **UV Lithography**

The state of the art NanofabricationSA facility allows the team to conduct its lithographic processes in a class 100 clean room.

Lithography uses chemicals to create images and fabricate the desired features.

In this process a mask is used to transfer a design into a substrate that has been prepared and coated with a chemical called a photoresist. The substrate and photoresist are exposed to UV light through the mask which transfers the feature design. Following developing, the features can be permanently formed into the substrate via an etching process, after which the photoresist is stripped away leaving the substrate and features ready for bonding.





An Australian  
Government Initiative  
National Collaborative  
Research  
Infrastructure Strategy



Government  
of South Australia  
Department of Further  
Education, Employment,  
Science and Training



ANFF-SA  
Australian National  
Fabrication Facility  
[www.anff.org.au](http://www.anff.org.au)

Specific equipment includes:

**Karl Suss Delta 80 Spin Coater**

- Multiple substrate types and sizes with different media for various applications
- Wafer size up to 200 mm and substrates up to 150 by 150 mm
- Spin speed from 1 up to 7,000 rpm
- Fully programmable process parameters including a dispense arm with different resist pumps, syringe systems and media nozzles for accurate and precise dispensing

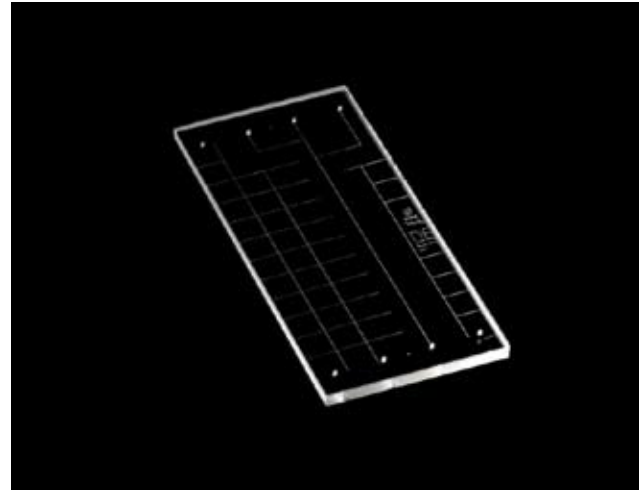
**EVG 620 Mask Aligner with nanoimprint lithography (NIL) function**

- Optical double-side lithography and precision alignment up to 150 mm wafer sizes
- Ultra-soft wedge compensation together with a computer controlled contact force between the mask and wafer ensures that both yield and mask lifetime are dramatically increased
- High resolution top and bottom side splitfield microscopes ensure < 1 µm alignment
- Imprint processes with stamps to substrates ranging from small chip size pieces up to 150 mm in diameter
- Uniform contact force for high yield large area printing
- NIL function allows for sub 100 nm features at low cost, high throughput and high resolution

**Our Facilities and Equipment**

28 m<sup>2</sup> ISO class 6 (class 1000) and 45 m<sup>2</sup> ISO class 5 (class 100) cleanrooms with ISO class 4 (class 10) work areas

- Süss Delta80+ Gyrset Spin Coater
- EVG620HE Mask Aligner with UV-NIL capability
- Ulvac ND-570 Deep Reactive Ion Etching (DRIE) system
- EVG520 Substrate Bonder and Hot Embosser
- Kira SuperMill 2m

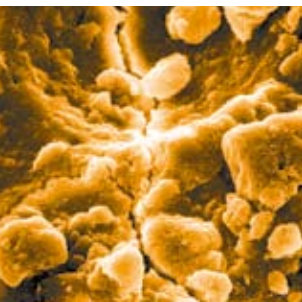


**Characterisation and Quality Management**

- Microfluidic chip test station
- VEECO optical profilometer
- SEM
- AFM
- XPS
- ToF-SIMS

# AIB Labs Bio-Interface Facility

Bio-interface Techniques and Instrumentation applicable to the SA Biotechnology Industry



## XPS

- Measures the elemental composition (atomic percentages) of materials surfaces and is quantitative

### Applications in Biotechnology

- Surface analysis of materials, lab ware and devices
- Quality control for fabrication of surface-treated lab ware, eg tissue culture polystyrene
- Detection of adventitious, surface-bound contaminants interfering in manufacturing processes and biological assays
- Detection of biological molecules adhering to surfaces of lab ware, devices, etc
- Troubleshooting for materials surface problems

## ToF-SIMS

- Used to image and record organic and inorganic mass spectral data of solid materials
- Qualitative surface chemistry comparison between samples
- Extremely surface sensitive (10-20Å), sub-micron imaging resolution, ppm-ppb detection
- Four ion sources allow for greater range of analytical, imaging and depth profiling capability

### Applications in Biotechnology

- Latest C<sub>60</sub> ion source for analysis of polymers (high mass fragments) and depth profiling of organic materials
- Surface analysis of materials, lab ware and devices
- Detection of manufacturing materials problems and adventitious surface-bound contaminants
- Detection of biological molecules adhering to solid surfaces

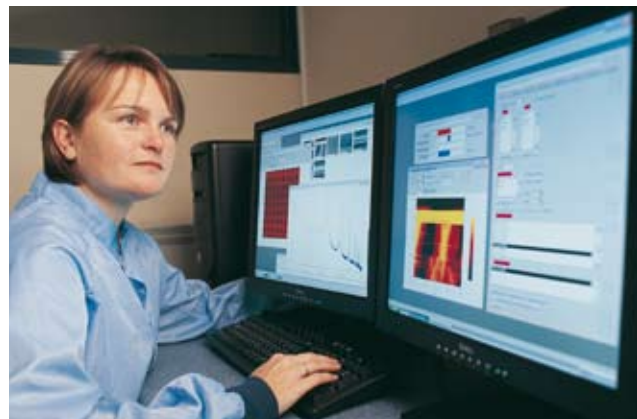
Often used in tandem with XPS which is a complementary and powerful combination

## AFM

- Measures the surface topography of materials down to nanometre resolution (Scanning Electron Microscopy)
- Can analyse samples in air and under water
- Vacuum not necessary

### Applications in Biotechnology

- Detection of topography defects in manufacturing processes of lab ware, diagnostic chips, etc
- Imaging of biological matter (eg cells) attached to solid device surfaces
- Detection and analysis of inhomogeneities in materials, for example uneven curing of contact lenses



# Visual Productions

See your science

The Visual Productions Unit produces professional and responsive material that is tailored to the needs of the client.

Bank SA, Australia Post, SA Lotteries, Port Adelaide Enfield Council, Signal Point Tourist Centre, SA Tourism and the University of Adelaide.

Initially established to facilitate the needs of academic staff and students, our professional design service is now available to industry and members of the public. Our designer has produced work for a diverse range of external clients including Ansett Airlines,

We can design and print annual reports, newsletters, pamphlets/brochures, conference posters, banners, invitations, logos, bookmarks, charts, flow charts, graphs, diagrams, spot illustrations, cartoons and more.



Surface Analysis, Colloids, Interface, Polymer Analysis and Characterisation



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**Confidentiality Statement**

The Wark ensures the complete confidentiality of your projects. Confidentiality agreements are available if required.

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