

What's Brilliant and BRIGHT at the Australian Synchrotron

Professor Michael James





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OPAL Research Reactor Lucas Heights, Sydney





Australian Synchrotron Clayton, Melbourne



Just down the Hume Highway...





Lucas Heights

875 km 9 hrs drive 1½ fly

Clayton

The Australian Synchrotron







The Australian Synchrotron...



Is an electron accelerator...

When electrons travel through magnetic fields at relativistic speeds they generate intense beams of synchrotron light (Infrared, visible and X-rays)

The Australian Synchrotron...





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The Storage Ring...





How is light produced in a Synchrotron?





Synchrotron light produced by a bending magnet source is **collimated**, **tunable**, **polarized** and up to a *million times* brighter than the sun.

How is light produced by a Wiggler?



Wiggler: Incoherent broad spectrum emission



Higher energy and higher flux than a bending magnet

Superconducting Multipole Wiggler





How is light produced by an Undulator?





Light produced by the Australian Synchrotron can be a *billion times* brighter than light from the sun.

Our Science





Australian Synchrotron: Areas of Impact



Research at the AS covers a broad range of fields:

Biosciences and Health; Earth and Environmental Science, Advanced Materials; Engineering and Manufacturing; Energy and Sustainability Science; Chemistry; Physics; Agriculture and Food processing; Cultural Heritage and Archaeology; Beamline and Technique Development; and Accelerator science.



The Australian Synchrotron at a Glance



140 staff supports 5000 hours of User Operations

10 Beamlines

6,000 Registered Users

1000 User Experiments per year

5,600 User Visits per year

3,250 Journal Publications from Users or Staff ~ 500 Journal publications per annum.

450 Journal Publications with Impact Factor > 7

1,500 Protein Structures in Protein Data Bank

> 800 Graduate Theses





10 Operational Beamlines







Infrared Microscope (IRM) Terrahertz / Far-IR Spectroscopy (THz/Far-IR) Soft X-ray Spectroscopy (SXR) X-ray Absorption Spectroscopy (XAS) Powder Diffraction (PD) SAXS / WAXS Macromolecular Crystallography (MX1) Micro-focused Crystallography (MX2) X-ray Fluorescence Microscopy (XFM) Imaging and Medical Beamline (IMBL)

Structural Biology & Health: MX1 & MX2





Immunology





Bacterial Structure



Cancer & Leukaemia



Tuberculosis



Toxins



Herbicides



Malaria



Alzheimer's disease



ACTIVE INACTIVE

Antibiotics



17 nm

Virology

Ansto

Venetoclax is now being used to treat Chronic Lymphocytic Leukaemia.

"There has not been a new effective treatment for Leukaemia for the past 50 years."



The US FDA recently granted approval for *Venetoclax*

Also approved in Australia, Europe and some Asian countries.



Australian Government

Chemical Crystallography for Advanced Materials



Kuan Sun, *et al.*, A molecular nematic liquid crystalline material for high-performance organic photovoltaics, *Nature Communications*, 6, 6013 (2015).



Crystal structure and molecular packing solved using MX2 beamline



OPV performance approaching 10% PCE in printable flexible films





A state-of-the-art Eiger 16M detector is now operational on MX2.

The ACRF Eiger 16M can take data ~10x faster with quality that can lead to huge improvements in structural resolution.

\$2M support from the



Don't forget to upgrade your computing and data storage systems...



Infrared Microspectroscopy





Bruker FT-IR spectrometer and Hyperion 2000 IR Microscope

- Transmission
- Reflection
- Grazing Angle Objective
- Attenuated Total Reflection (ATR)
- Micro-compression Cell
- Linkam FTIR600 sample stage
 -196 °C to 600 °C
- Sample stages for the study of living biological cells

Motorised stage allows raster mapping Spatially resolved "spectral maps" (~3 -8 µm resolution)

Studying Bone Quality After Drug Treatment





Fluorescence micrographs showing 6x15 µm regions from where Infrared spectra were taken.

Intermittent administration of parathyroid hormone (PTH) is used to stimulate bone formation in patients with osteoporosis.

Bone deposited during PTH treatment undergoes normal collagen maturation and normal mineral accrual.



C. Vrahnas, et al. Bone, 93, 146, 2016

THz/Far-IR Spectroscopy





IFS 125 / HR Brüker spectrometer utilises a Michelson interferometer with an optical path length of 942 cm.

Continuous spectral coverage from the THz to the mid-IR region.

- Gases
- Surfaces
- Materials
- Cultural Heritage
- Forensic studies
- Protein & higher order structures

Can conduct studies of gaseous materials under a range of extreme environments

Astrochemistry Using THz Spectroscopy





Unlike Earth, where water drives the weather, on Titan water is frozen meaning it doesn't play a role in its atmosphere.

Weather on Titan is controlled by methane (5%) and nitrogen (95%).

The two chemicals react slowly with sunlight to form cyanide gas and other chemicals.

"Titan: 1.2 billion km away.

Cyanide rain falls from the skies and temperatures are around -180 °C."

Far-IR spectra can be obtained using a collisional cooling cell and compared with data from the Cassini probe.

C. Ennis, *et al. Physical Chemistry Chemical Physics*, **19**, 2915 (2017).

Imaging and Medical Beamline





138m - High Resolution Phase Contrast Imaging 34m – Fast Imaging and Computed Tomography (CT)

22m - High Dose Irradiation, MRT

Conventional Imaging vs Phase Contrast using IMBL





Conventional Radiograph



Phase contrast imaging provides exquisite structural detail at video speed.

CT on Large Objects – No Worries...





Upgraded Computed Tomography Stages

Enables *Phase Contrast* CT of heavy (~80 kg) and large (> 1m) objects

> Photon Energy: Up to 300 keV

Beam Dimensions: 300mm x 20mm

Resolution: 5-10 mm



HOME » NEWS » WORLD NEWS » AUSTRALIA AND THE PACIFIC » AUSTRALIA

Australia scientists develop 'bionic spine' which could help paralysed patients walk

Device can be inserted in the brain – without brain surgery – and could allow paralysed patients to operate robotic limbs "using thought alone"





nature biotechnology

Thomas J. Oxley, *et al. Nature Biotech.*, **34**, 320 (2016).

Powder Diffraction Beamline







New Sample Robot

New robotic sample changer & auto-alignment system Hot air blowers (1000 ° C), furnaces (2300 ° C), cryostream (80 K) and cryostats (10 K) Gas & fluid flow cells, DAC (30 GPa), hydrothermal reactions Batteries, electrochemical cells

Making Better Batteries





Li, Na and K ion batteries are key technologies for power storage

Charge / discharge \rightarrow phase formation and stability at the cathode by in situ Powder Diffraction and X-ray Absorption Spectroscopy

High throughput battery measurement system now routine.

Studies of electrode and electrolyte chemistry.

Able to collect rapid data with realistic charge rates.

You Have More Chance of Being Hit by a Meteorite



Ancient iron micrometeorites suggestive of an oxygen-rich Archaean upper atmosphere



Accepted theory on O₂ concentrations in early Earth's upper atmosphere

Oxidation of small micrometeorites passing through upper atmosphere

Separating the effects of geological oxidation

Powder Diffraction beamline used to characterise oxidised species from ~25 µm diameter samples

Andrew G. Tomkins, et al., *Nature*, **533**, 235 (2016).



SAXS/WAXS beamline generated > 100 publications in 2015 & 2016 and more than 65 already in 2017.

> Polymers, Organic solar cells and electronics Advanced drug delivery systems Catalysis & Metal Organic Frameworks Surfactants, and Liquid Crystal systems Food and Agricultural products Nanomaterials Protein structures and complexes



Old System for Solution Handling for Protein SAXS





- Up to 96 samples automatically
- ~3.5 min/sample

Disadvantages:

- Requires ~50-100µL of sample
- Even under flow (~5 μL/s) still havs capillary fouling and radiation damage

Co-Flow is the Answer





- Large improvement in dose efficiency (12-fold)
- Improved measurement statistics (2-fold)
- Reduced sample volume
 - 5-10 μL (routine) down from 50-100 μL
 - 2 μ L (minimum) down from 30 μ L
- Sample fouling gone
 - only very minor buffer contamination can occur
 - much more stable day to day User operations
- Throughput should be able to be increased ~3-4 fold for static samples &

at-least 2-fold for Size Exclusion Chromatography.

Flow sample only in centre of capillary

Nigel Kirby et al., *Acta Cryst. D*, **72**, 1254 (2016).

Polymer-Based Organic Photovoltaics & Electronics



Researchers use the Soft X-ray Spectroscopy beamline (NEXAFS) to determine molecular orientation, and the SAXS/WAXS beamline (GIWAXS) to study polymer crystallinity in semiconducting polymer devices.









Plenty of great publications in: Nature Communications, Applied Materials Interfaces, Advanced Materials, Advanced Energy Materials, Nano Energy, Chemistry of Materials,...

Soft X-ray Spectroscopy Endstation

Measurement

UHV transfer



Prep

Chamber

- XPS
- SPECS Phoibos[™] 150 energy analyser

NEXAFS

- Total electron yield (TEY)
- MCP Total Fluorescence Yield (TFY)
- Channeltron Partial Electron Yield (PEY)

- Argon ion gun
- Dual cell thin film growth facility
- Organic Material Evaporator (RT to 300 C)
- Crucible Evaporators (300 to 1400 C)
- Electron Beam (rod) evaporator (to 2500 C)
- Low Energy Electron Diffraction (LEED)
- Quartz Crystal Microbalance
- Heating/cooling of sample: -160 C to 1600 C
- Gas Dosing (to 10⁻⁵ mbar)
- Kelvin Probe, 4 point probe

Soft X-ray Spectroscopy Endstation





SPECS Phoibos[™] 150 energy analyser

NEXAFS

- Total electron yield (TEY)
- MCP Total Fluorescence Yield (TFY)
- Channeltron Partial Electron Yield (PEY)

- Diamond & Si semiconductors
- Topological insulators & Dirac Semimetals
- Photo-catalysts
- Novel electronic materials
- Organic Semiconductors & OPVs

ARPES @ Australian Synchrotron Toroidal analyser

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03

Offline (VUV lamp) Online (SXR beamline)

"Super Resolution" Microscopy: Coherent Soft X-ray Ptychography





Michael Jones, et al., *Scientific Reports* **4**, 6796 (2014).

"Super Resolution" Microscopy: Coherent Soft X-ray Ptychography





Data taken using La Trobe University

Soft X-ray Imaging Endstation

Michael Jones, et al., Scientific Reports 4, 6796 (2014).

X-ray Absorption Spectroscopy Beamline

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- High brilliance, micro-focused beam.
- Various sample types:

liquids, solids, films, glasses, soft matter, biological tissues and cells,...

- Almost all metals and heavier elements (Ti to Pu)
- Large concentration range: ~100 ppb to ~10%
- p = 1...600 bar; T = 5...1500 K

(~\$1M monochromator upgrade in progress)



Studies of metals in Environmental Science, Advanced Materials, Earth Science Minerals Formation & Processing,...

Multi-Anvil Press ("D-DIA") – XAS Hutch C







The D-DIA allows study of materials under extremes of temperature & pressure

(~Upper Earth Mantle)



The widespread use of silver nanoparticles (Ag-NPs) results in their movement into wastewater treatment facilities and into agricultural soils.

Ag-NPs entering soils via wastewater pose a low risk to plants due to their conversion to Ag_2S in the wastewater treatment process.

> Peng Wang, et al., Environmental Science and Technology, **50**, 8274 (2016). Environmental Science: Nano, **4**, 448 (2017).





Nanoparticles and the Environment (XAS).

XFM - For the Study of Metals in Biology, Environment, Earth Sciences,...



Simultaneous access to 10⁺ elements 7 > 14 ~ Si

High sensitivity - sub-ppm; sub-mM; 1e-12 g/s

Native contrast - no dyes or contrast agents necessary

Quantitative

Non-destructive / minor damage

Extended penetration & Depth of Field

- study intact cells & sections

Sensitive to **chemical speciation** via XANES spectroscopy





XFM Microprobe for Mapping Metals Now Approaching Megapixels/sec







Maia Detector – Rev C (Improved ΔE ; Elements down to Si)

Fast Scanning Stages Up to 200 mm/sec

> 100,000 pixels/second



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XFM: Scanning Metals in Biology





Katherine Ganio, et al. *Analyst*, **141**, 1434 (2016).

Multimodal Whole "Animal" Imaging With X-ray Fluorescence Microscopy



The X-ray Fluorescence Microprobe reveals elemental composition within *C. elegans* @ 2 μ m resolution using a Maia Detector



Super-resolution (~50 nm) using Coherent X-ray Ptychography

> Phase + Fluorescence (Cu)



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Dr Gawain McColl (unpublished)





478,349 diffraction patterns in a single "fly scan" in 3 hours

(Previously, a similar "step scan" would take an additional ~26 hours)

Leaf stem from nickel hyperaccumulator tree from Borneo (Nickel shown in red) Dr Antony van der Ent (University of Queensland).

X-ray Fluorescence Microscopy **Unlocks Hidden Treasures**



Paper in *Scientific Reports* describing analysis of Degas' "Portrait of a Woman".

Associated media release triggered a massive social and conventional media response.

Coincided with major Degas Exhibition at National Gallery of Victoria

Aus. Synchrotron

Aus. Synchrotron

bbc in/2aDobyE

Researchers describe reveal of hidde by Impressionist Edgar Degas to @E 'What's really exciting is we now have one more Degas for the world to see' Story: @BBCNews bbc.in/2aMdPUv



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l of detail elieve it w Damworld

Dr. Amanda Caples @Vic LeadSci

Masterful example of creative arts @creat



Aus. Synchrotro



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10:29 AM - 5 Aug 2016

International Coverage > 1400 Media Articles

TV – News, Catalyst Radio **Newspapers** Web

Australian Synchrotron

BRIGHT: New Beamlines for the AS







\$114 million program to design and construct the next 8 beamlines.

- Micro-Computed Tomography
- Medium Energy XAS
- Advanced Diffraction and Scattering
- Bio-SAXS
- High Performance Macromolecular Crystallography
- Micro Materials Characterisation
- Multi-Modal Nanoprobe

Program to Commence in July 2017

Any Questions?...







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