Australian Synchrotron
Turning bright ideas into brilliant outcomes

SYNCHROTRON POWDER DIFFRACTION

Kia Wallwork

Synchrotron Science for Cultural Heritage Materials, September 2010

OUTLINE

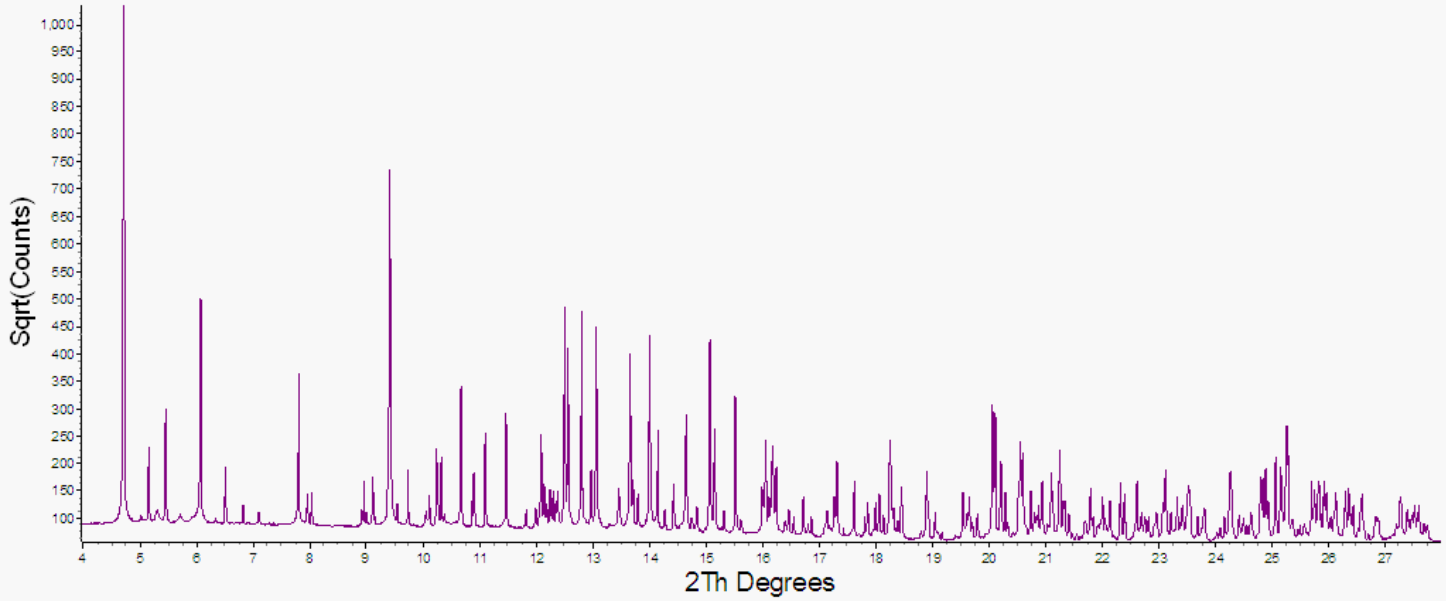
Powder Diffraction

- Introduction & Applications
- Beamline Capabilities
- Data Quality

Supported by
Australian Government
State Government Victoria AUSTRALIA

POWDER DIFFRACTION

- A technique for investigating atomic crystal structure
  - PD is not suitable for non-crystalline materials
- PD is not a spectroscopic technique
  - The peaks are not informative in isolation



APPLICATIONS OF POWDER DIFFRACTION

1. Phase identification

2. Quantitative phase analysis

3. *In situ* examination of reaction processes

    e.g. effect of heat treatment

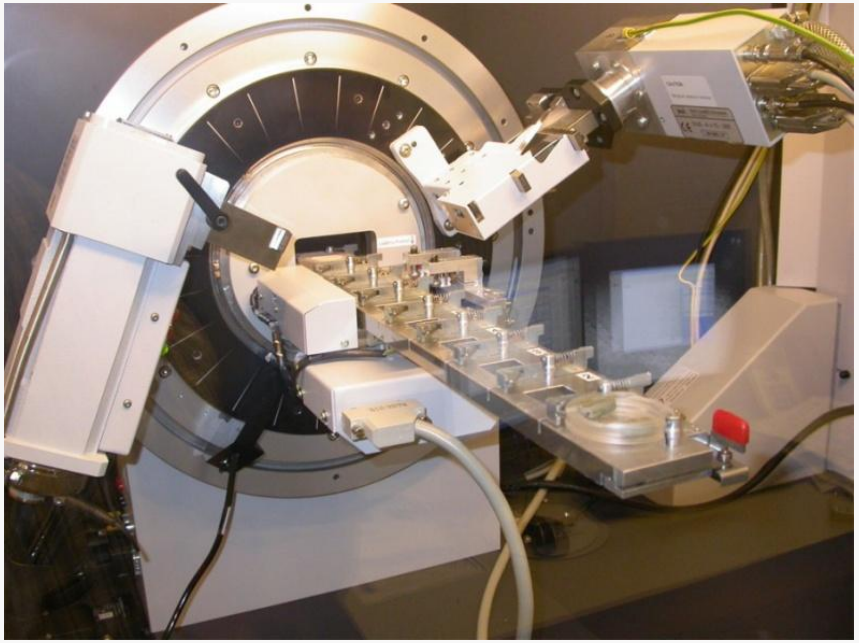
Supported by
Australian Government
State Government Victoria AUSTRALIA

TRADITIONAL X-RAY SOURCES

Vital for thorough studies

Advantages

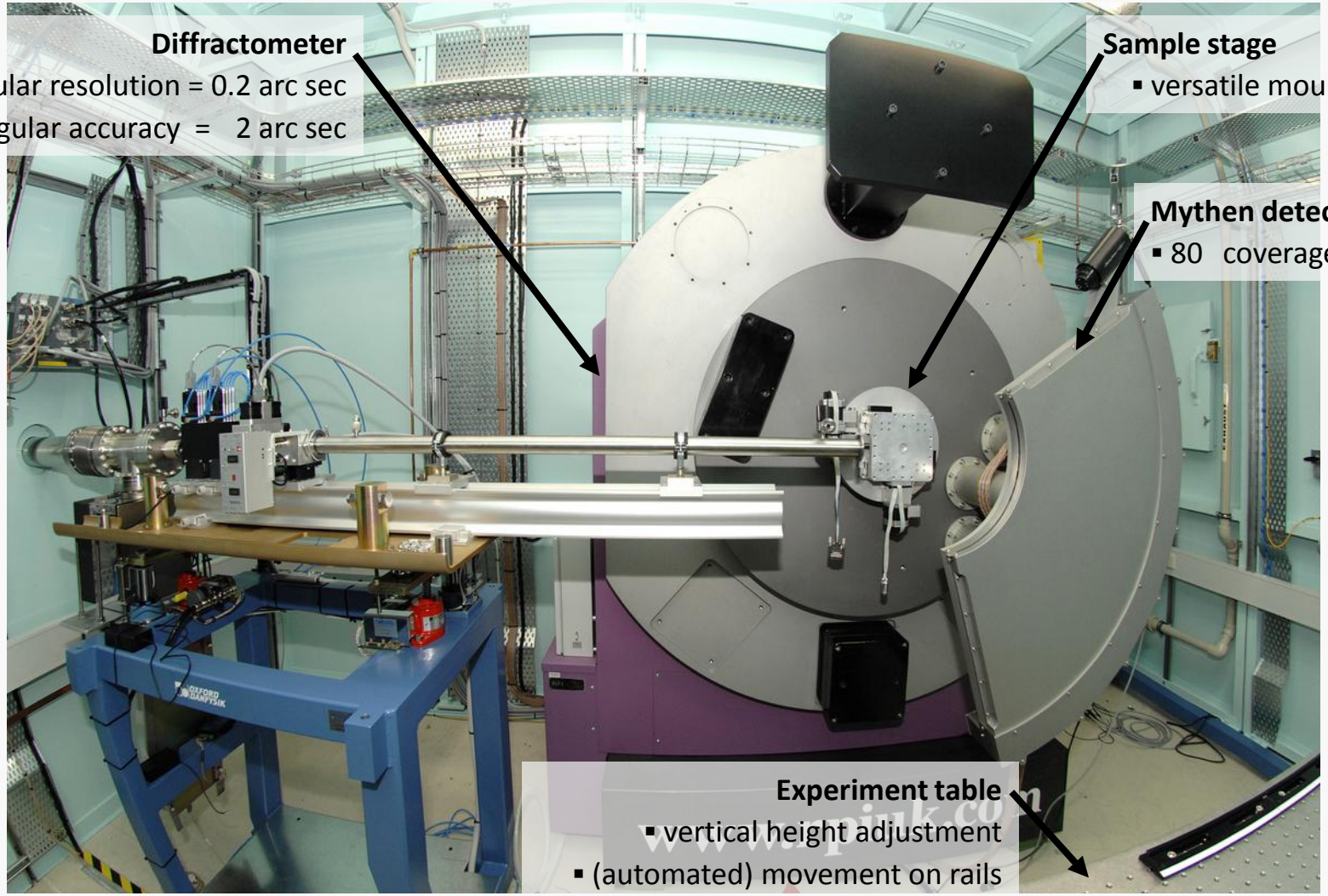
- Convenient to access
- Low cost
- Excellent for routine phase identification
- Quantitative analysis
- Some structure solution possible



Limitations

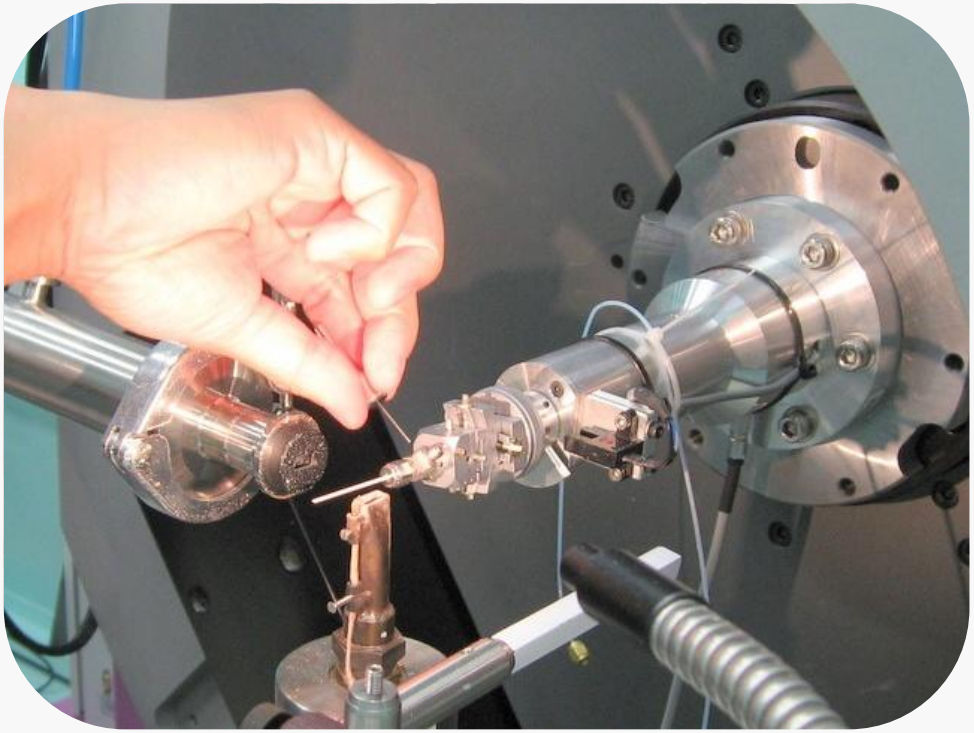
- Poor peak resolution
- Low intensity source
- Speed of data acquisition
- Limited sample ancillaries / stages

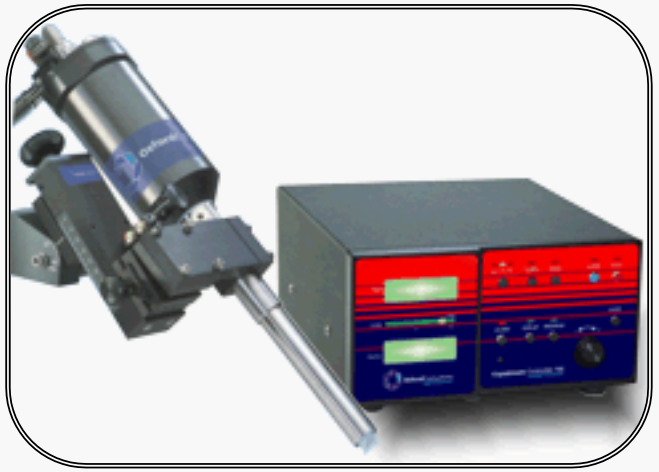
POWDER DIFFRACTION BEAMLINE END STATION



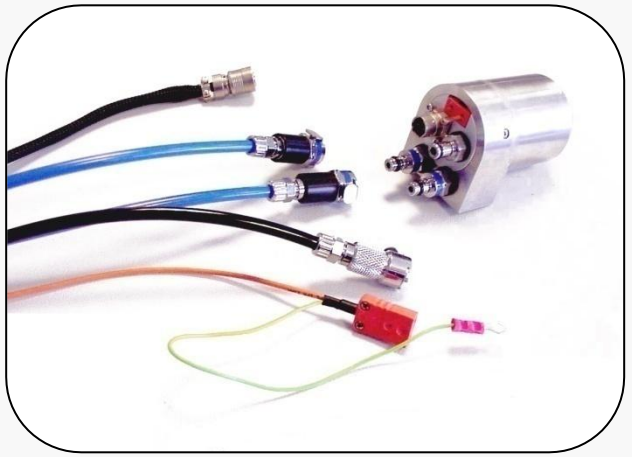
Australian Synchrotron
Turning bright ideas into brilliant outcomes
Supported by
Australian Government
State Government Victoria AUSTRALIA

SAMPLE ANCILLARIES – CAPILLARY SAMPLES

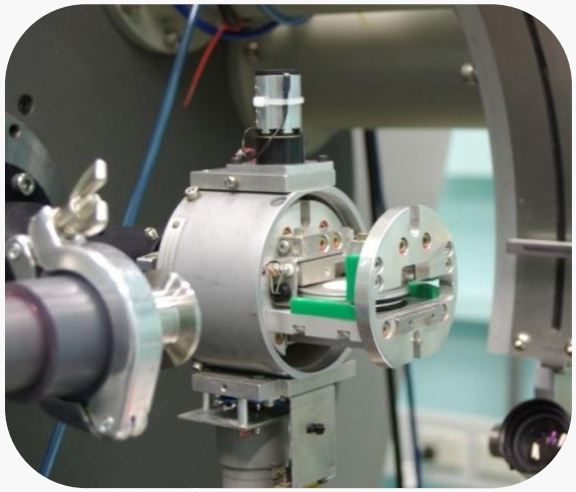


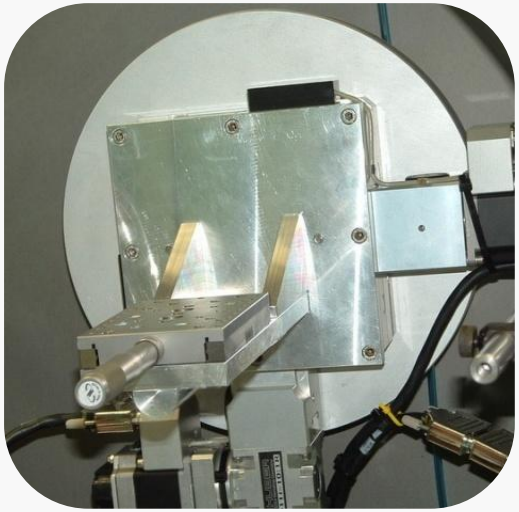


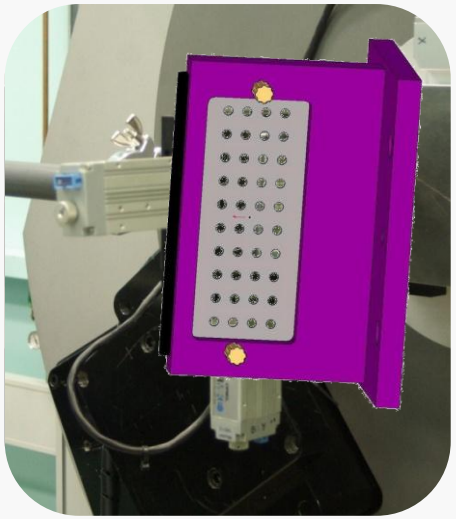
Temperature range = 80 – 1,173 K

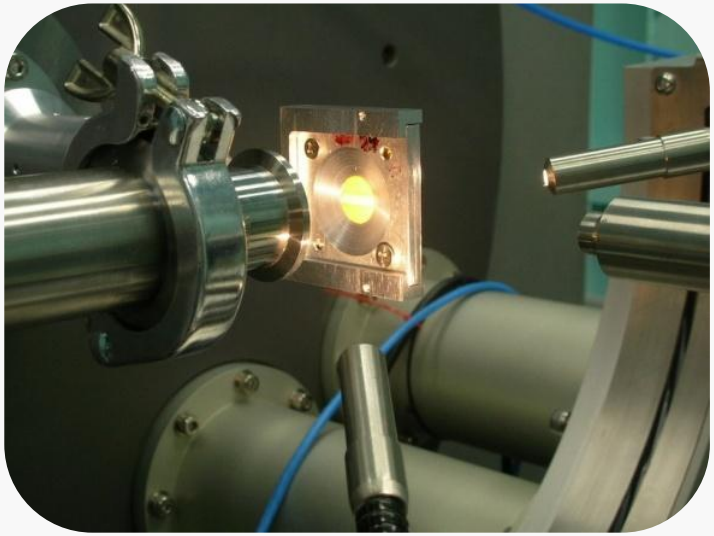


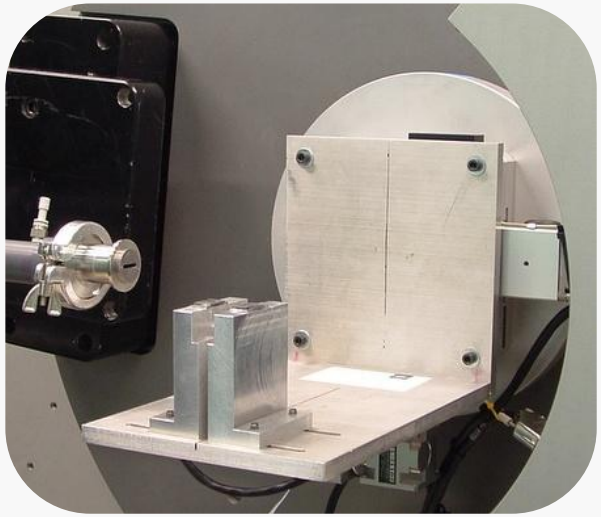
SAMPLE STAGES – VARIOUS MOUNTING METHODS





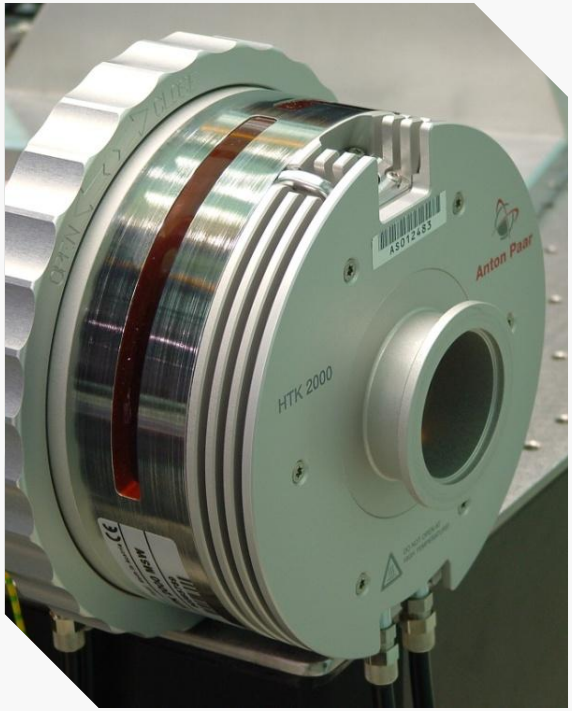




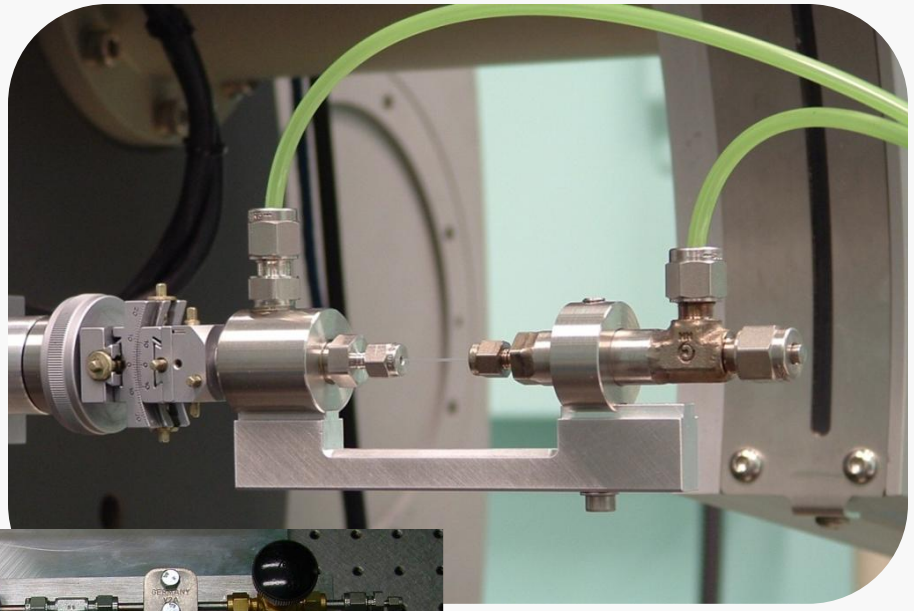


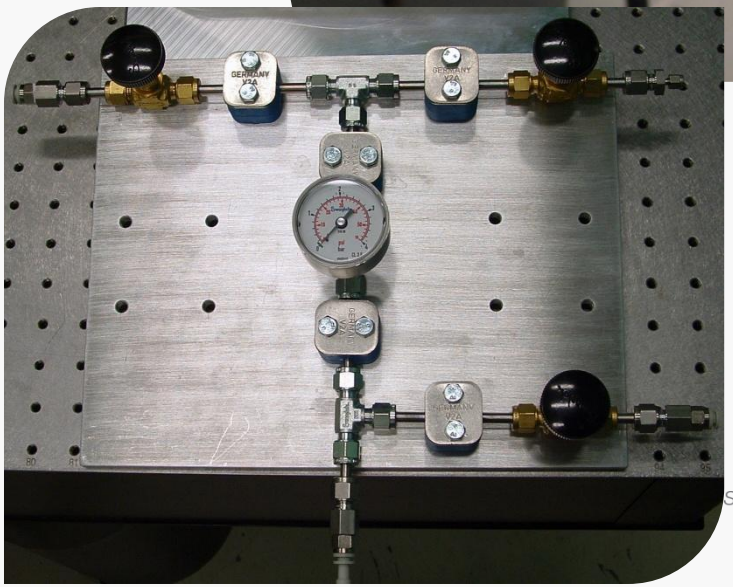
MECCANO & STICKY TAPE

SAMPLE ANCILLARIES – FURNACE & GAS FLOW



Furnace temperature range = 298 – 2,573 K



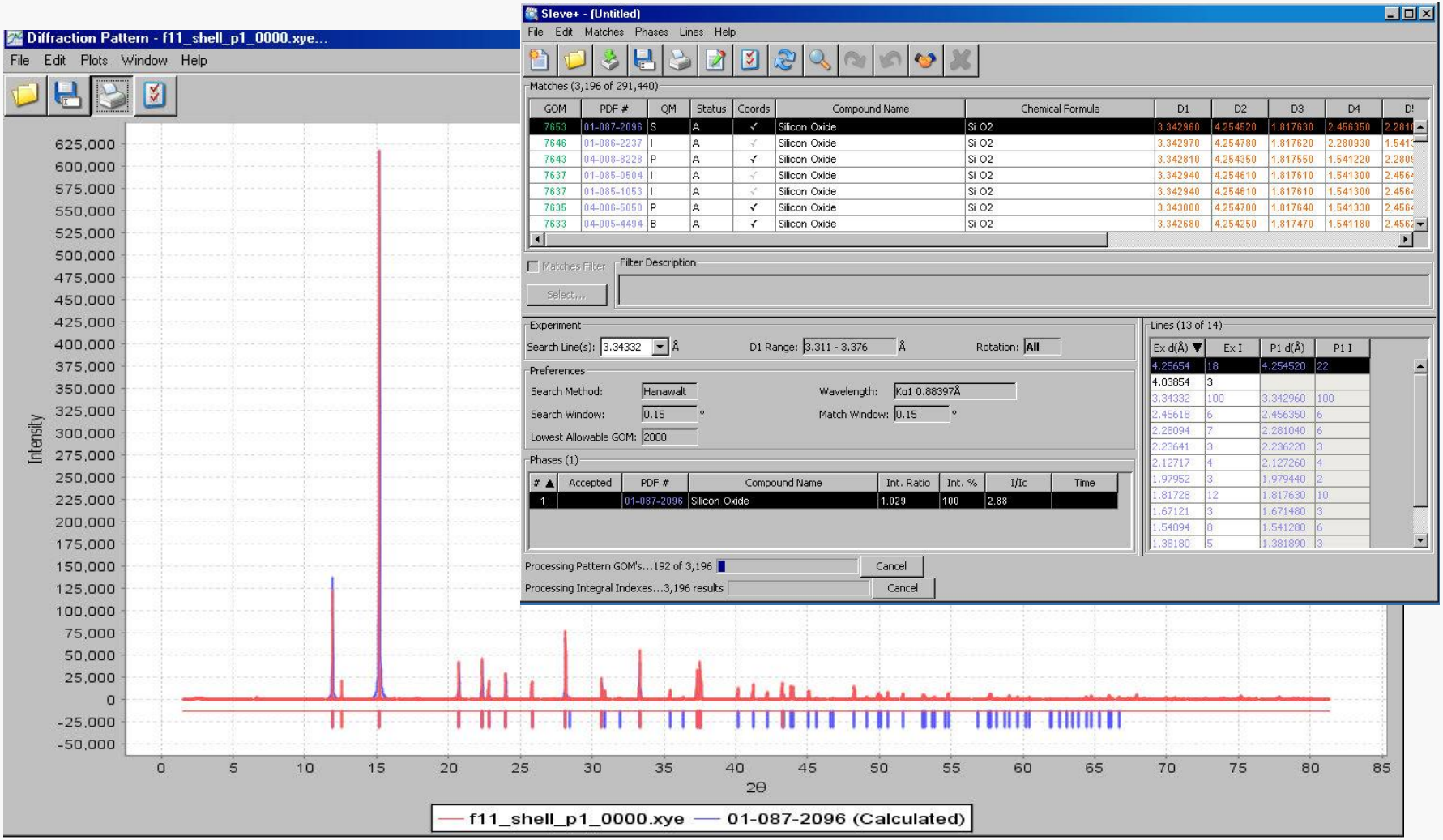


Gas/vacuum cell

Australian Synchrotron
Turning bright ideas into brilliant outcomes

Supported by
Australian Government
State Government Victoria AUSTRALIA

SEARCH / MATCH DATABASE

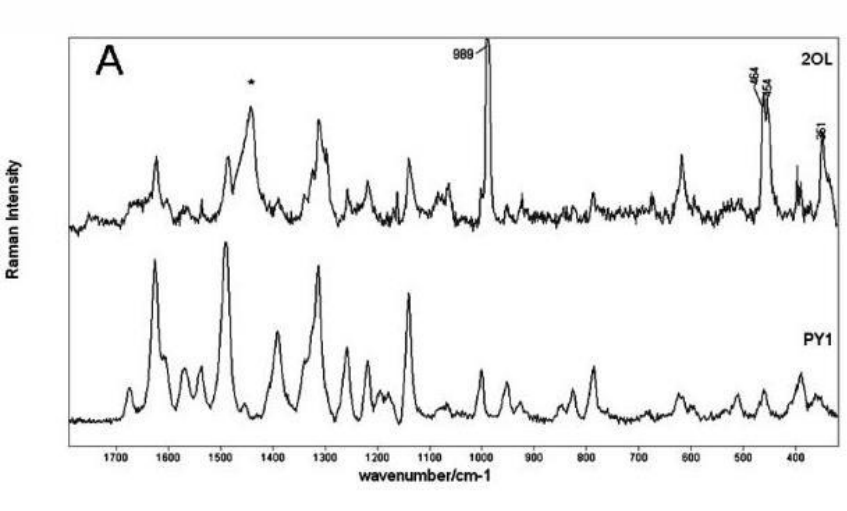


Combined X-ray Diffraction and Raman Identification of Synthetic Organic Pigments in Works of Art: From Powder Samples to Artists' Paints

L. B. Brostoff, S. A. Centeno, P. Ropret, P. Bythrow, and F. Pottier, *Anal. Chem*. 2009, **81**, 6096–6106

Raman

XRD



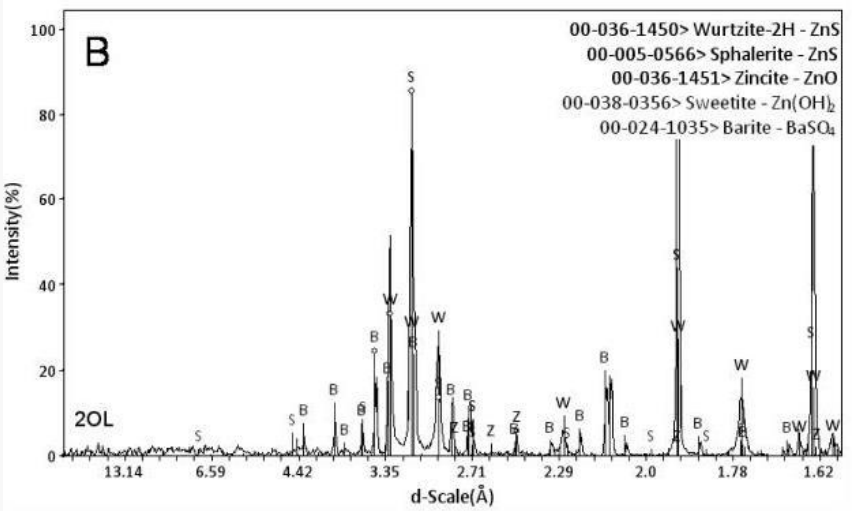
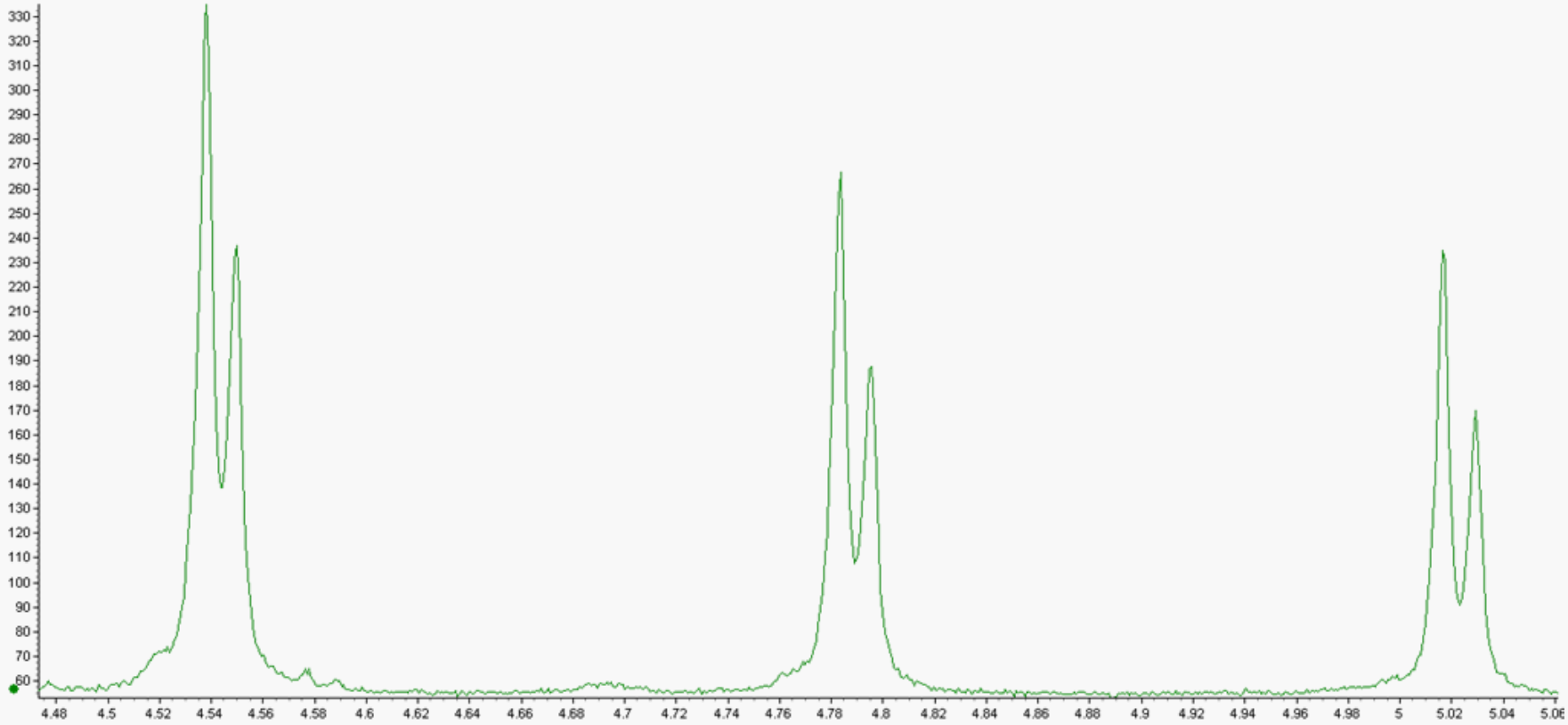


Figure 4. (A) Raman spectra of the oil bound sample 2OL (Lukas (Studio) Brillantgelb hell, top) and of a PY 1 reference sample (SunChemicals, bottom). Main bands are due to the extenders $BaSO_4$ (ca. 989 cm^{-1}, shown out of range, 464 and 454 cm^{-1}) and ZnS (ca. 351 cm^{-1}) indicated. The band at ca. 1440 cm^{-1}, marked by an asterisk, may arise from CH_2 deformations of the oil binder; $\lambda_0 = 785$ nm. (B) μXRD pattern detail (Cr Kα radiation) of paint sample 2OL, after background subtraction, with ICDD pattern matches shown in vertical bars (barite peaks marked with "B"; zincite peaks marked with "Z"; sweetite peaks marked with "S"; wurtzite peaks marked with "W"; sphalerite peaks marked with "S").

DATA QUALITY

Laboratory XRD Instrument

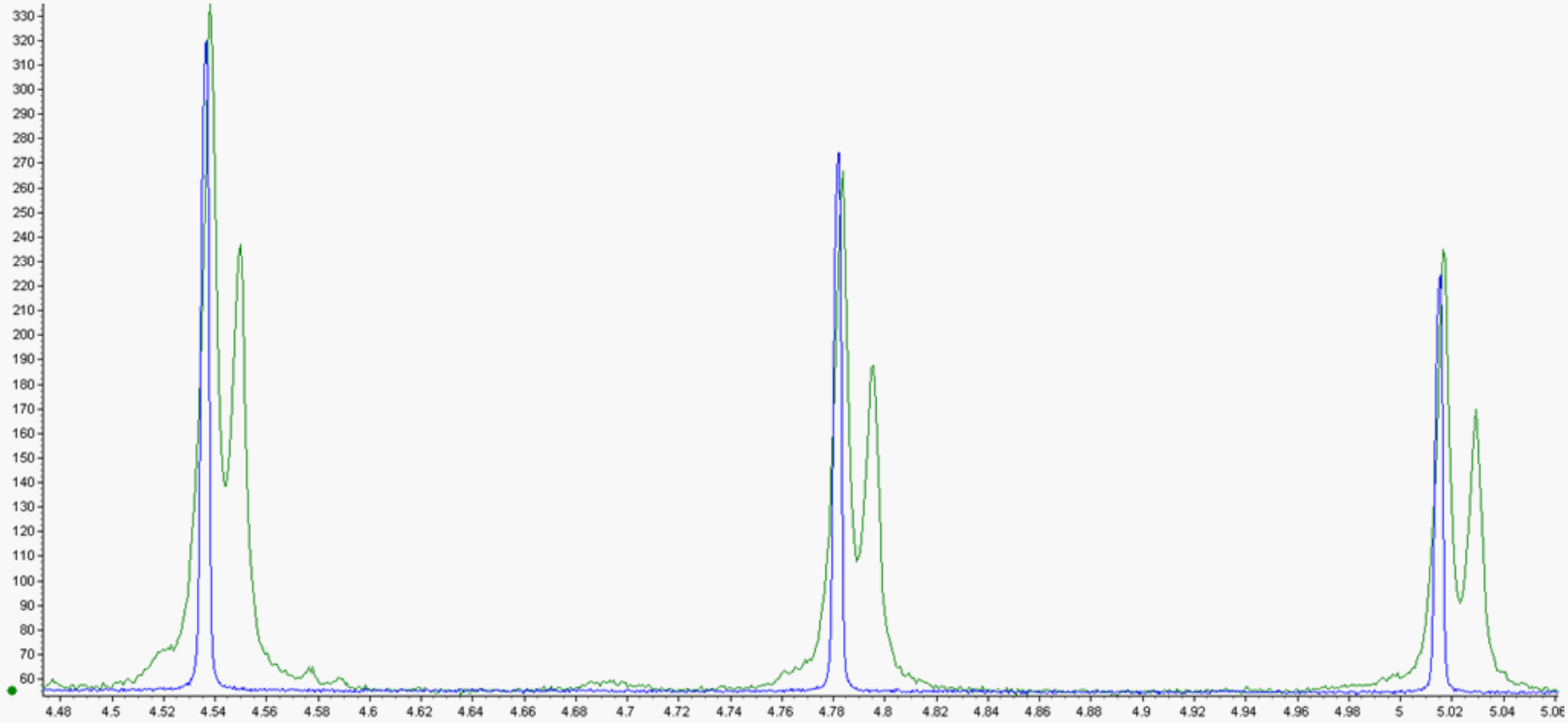
- Cu K$\alpha_{1,2}$
- Stationary sample – divergence in beam ensures good ‘powder average’



DATA QUALITY

Synchrotron PD data overlaying laboratory data

- 0.3mm capillary
- Spinning sample – rotating sample ensures good ‘powder average’



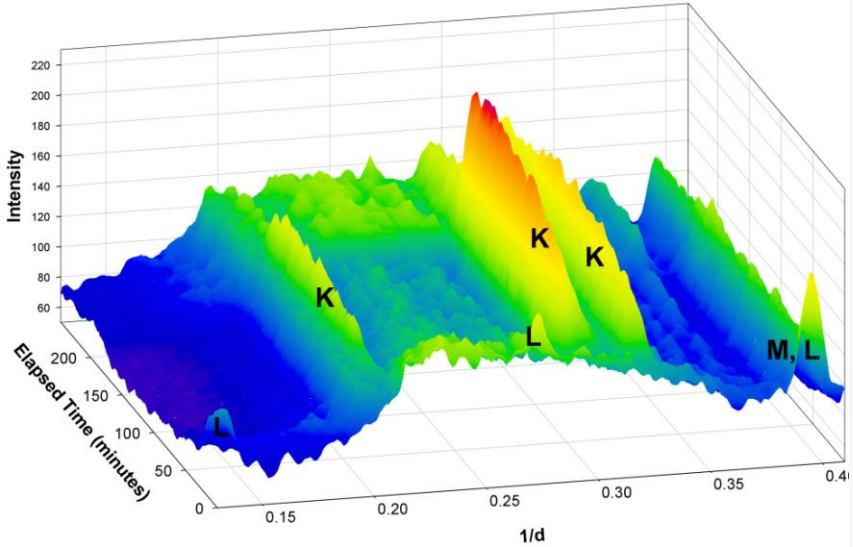
Australian Synchrotron
Turning bright ideas into brilliant outcomes

Supported by
Australian Government
State Government Victoria AUSTRALIA

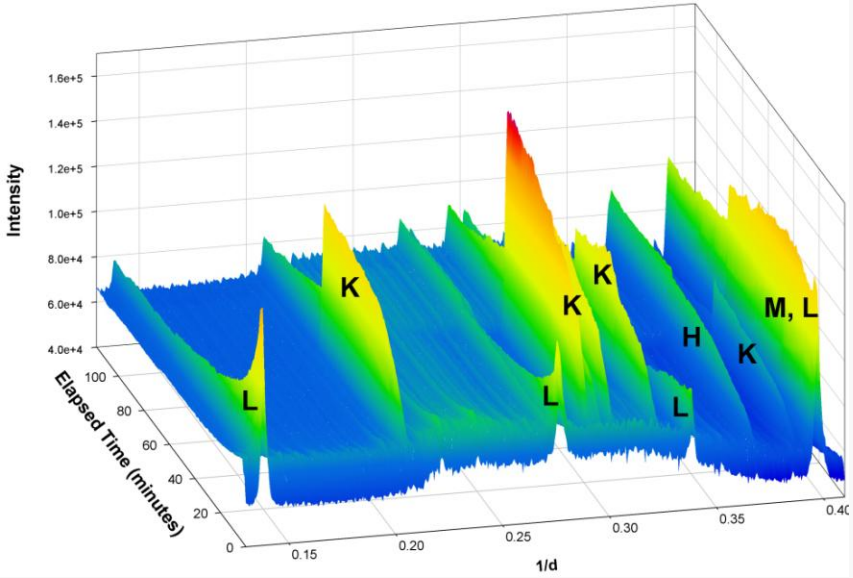
Data and Slide by Ian Madsen, CSIRO

SIGNAL TO NOISE

Laboratory XRD



Synchrotron XRD



Data from Nicola Scarlett, CSIRO

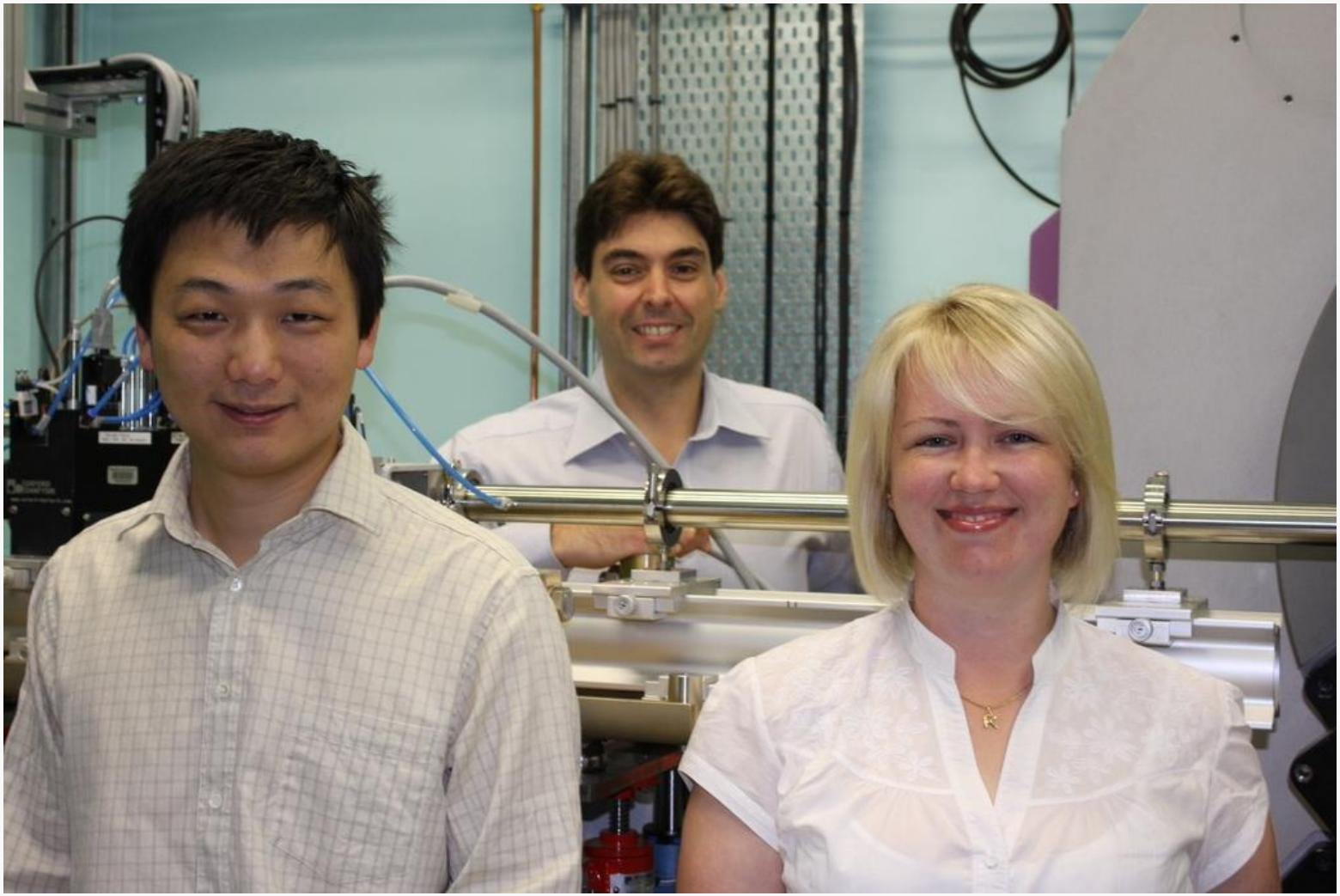
TAKE-HOME MESSAGE

Synchrotron powder diffraction

- Important tool for materials characterisation
- Non-destructive
- Flexible and tailored sample mounting options
- High resolution data
- High intensity X-rays
- Tunable, high energy X-rays

Supported by
Australian Government
State Government Victoria AUSTRALIA

THANK YOU



Australian Synchrotron
Turning bright ideas into brilliant outcomes

Supported by
Australian Government
State Government Victoria AUSTRALIA