Powder diffraction

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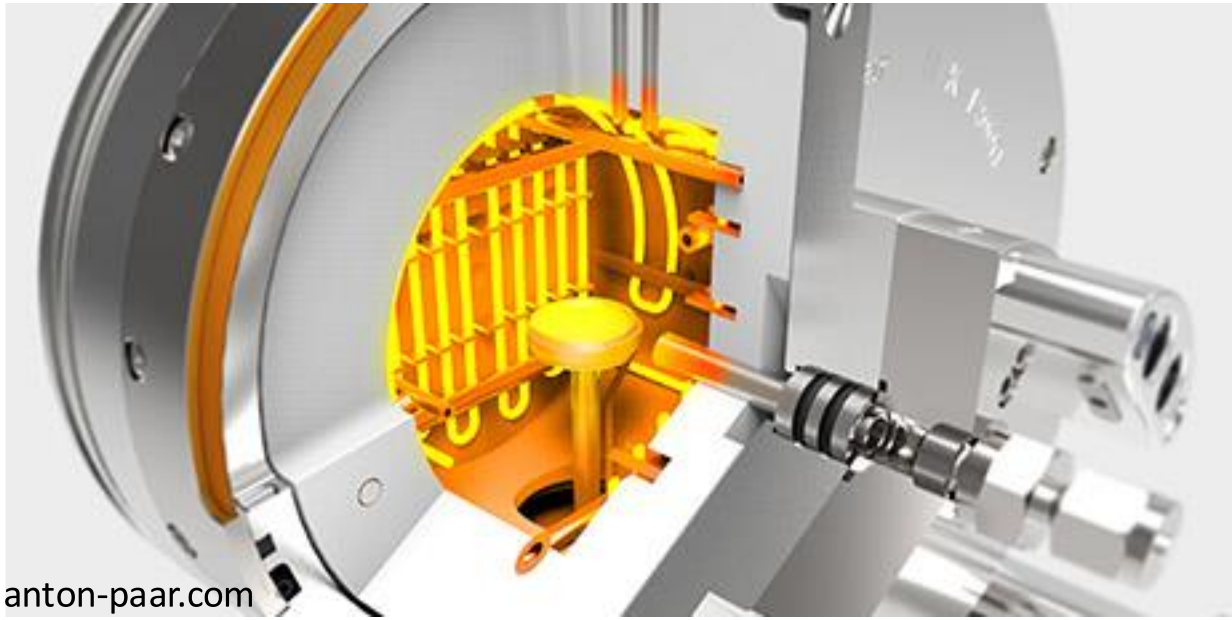
Outline

- Basic theory of Powder diffraction
- Instrumentation & facilities
- Highlight of case studies
- Beamline guide and access

Why powder diffraction?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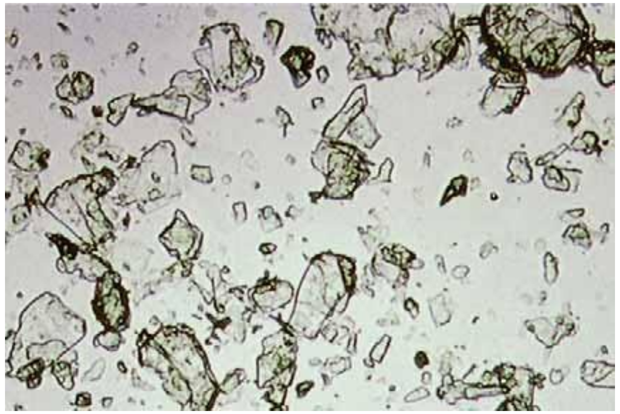
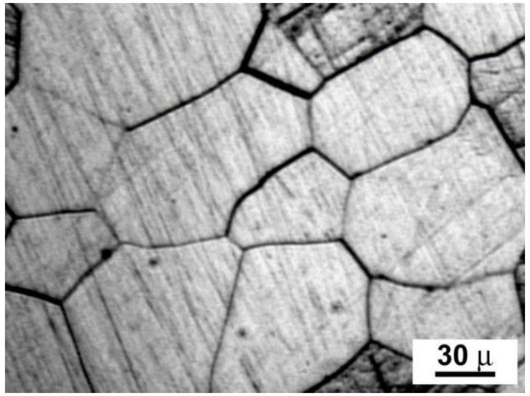
- Real materials are often powders
- Easy sample preparation
- No need to rotate the sample (simpler sample environments)
- Fast data collection – time-resolved experiments
- Multiple phases/fingerprinting

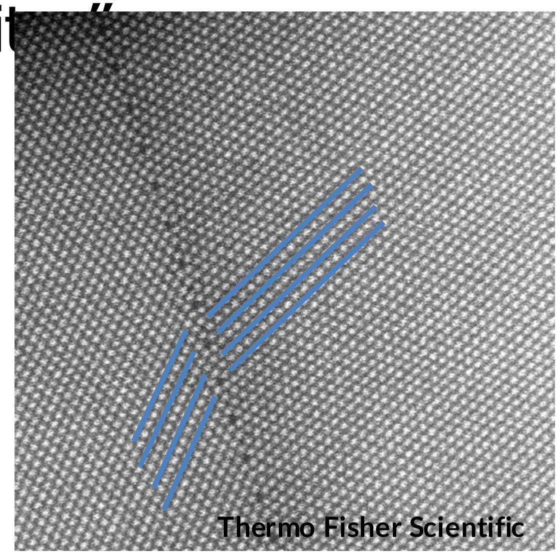
What is a powder?

- By “powder” we mean a polycrystalline material.
- Much of the world is crystalline but things rarely exist as large single crystals.
- Metals are typically polycrystalline and diffract like a powder.
- Small crystals are called “crystallit





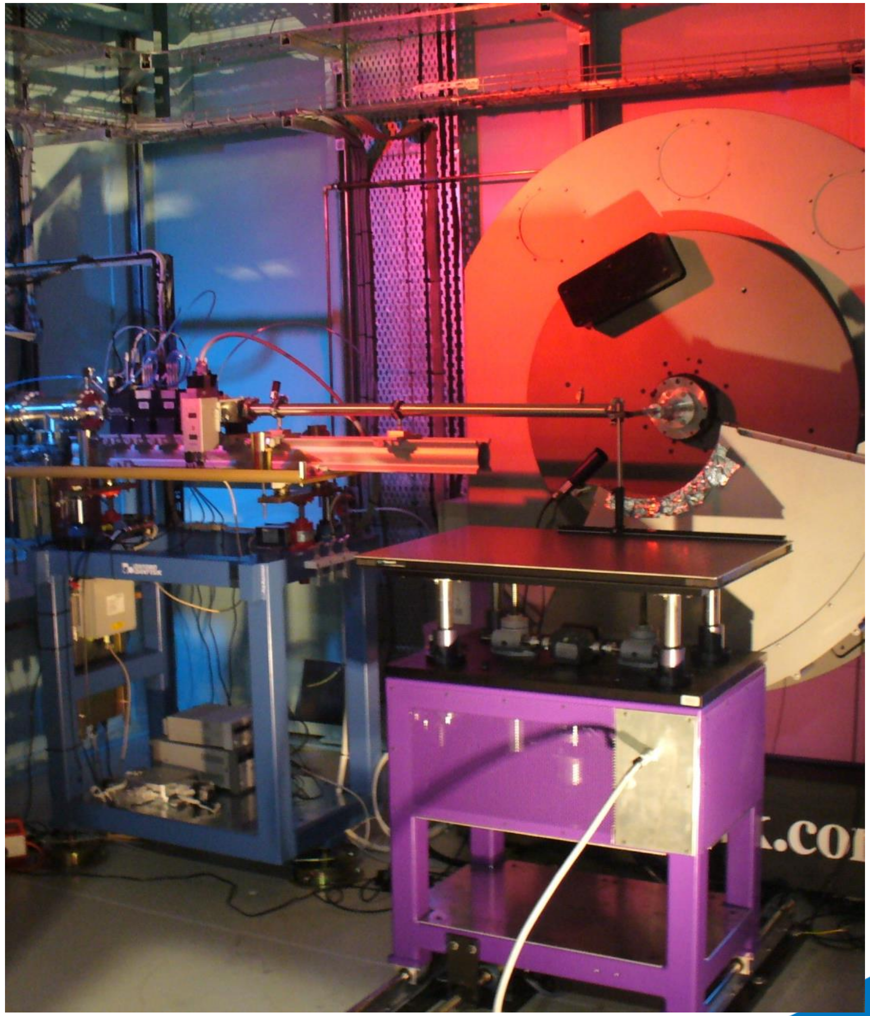
Metal grains are tiny crystals



Grain boundary in gold (STEM image)

Powder diffraction

- Unit cell parameters and space group determination
- Qualitative phase identification for single and multiple phases
- Quantitative phase identification
- Structure solution and refinement
- Crystallite size and strain from peak profiles
- Time-resolved and *in-situ* experiments
- Physical properties such as thermal expansion and compressibility



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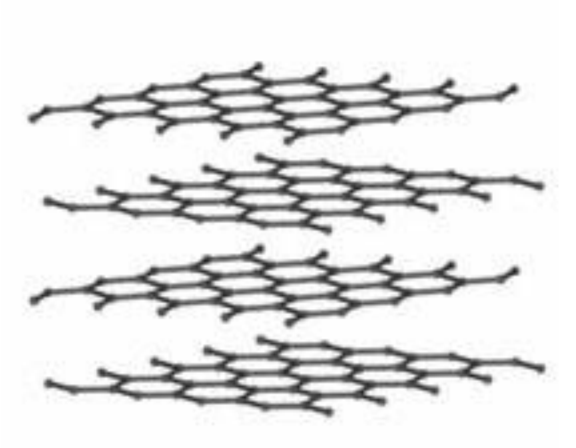
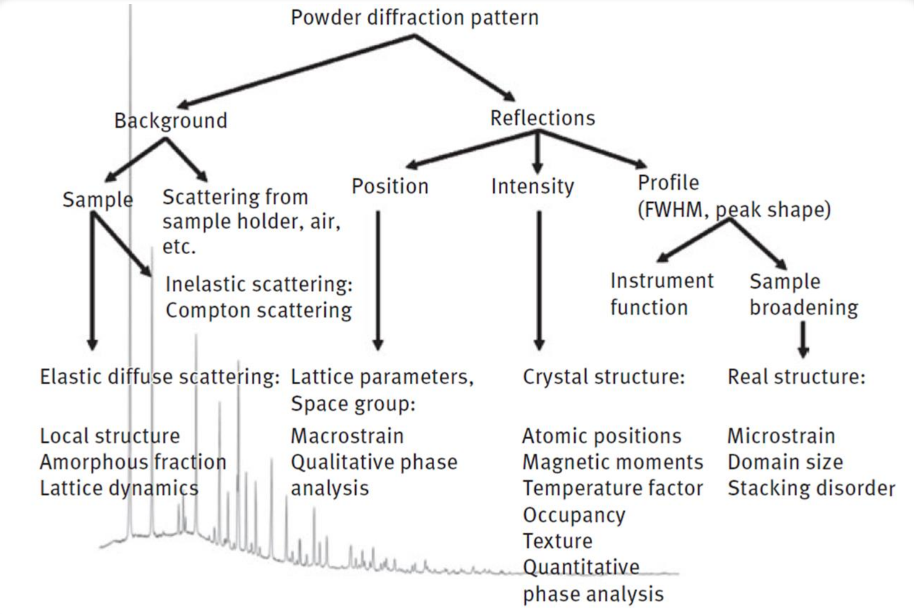
Powder diffraction

Graphite

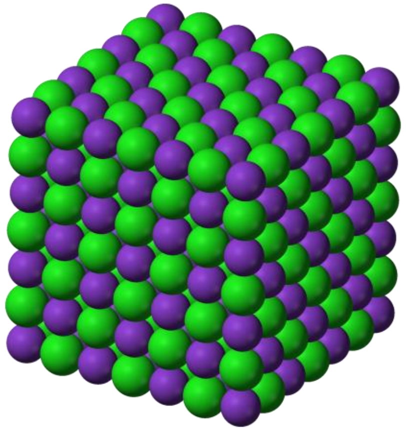


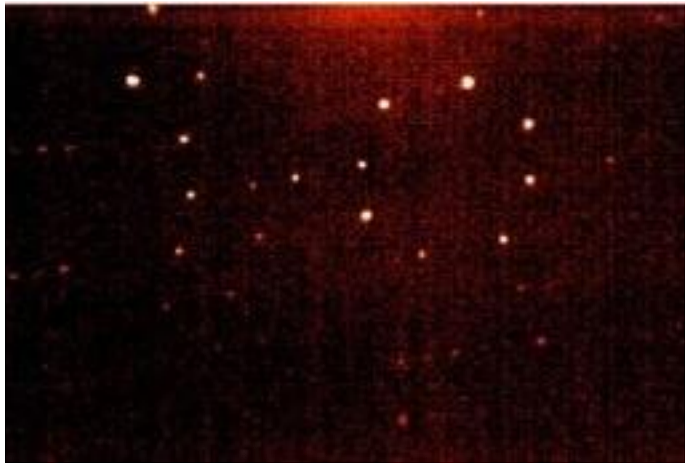
Diamond

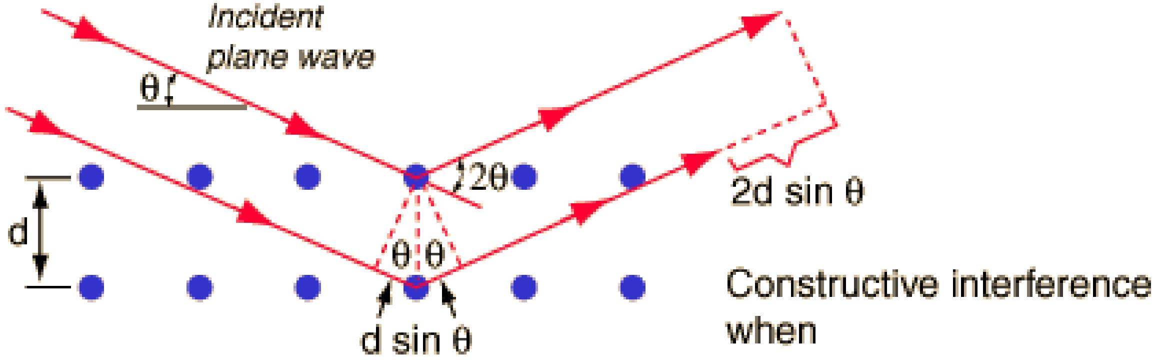


Diffraction - Theory

- Crystal lattices have regularly-spaced planes of atoms
- X-Rays (and some neutrons) have wavelengths close to these plane spacings ($\lambda \sim 1$ Å $= 10^{-10}$ m).

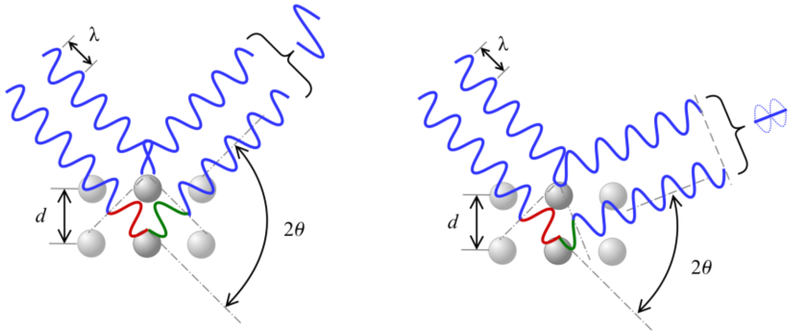






$$n\lambda = 2d \sin \theta$$

Bragg's Law



- We only observe diffracted X-rays when Bragg's law is satisfied.

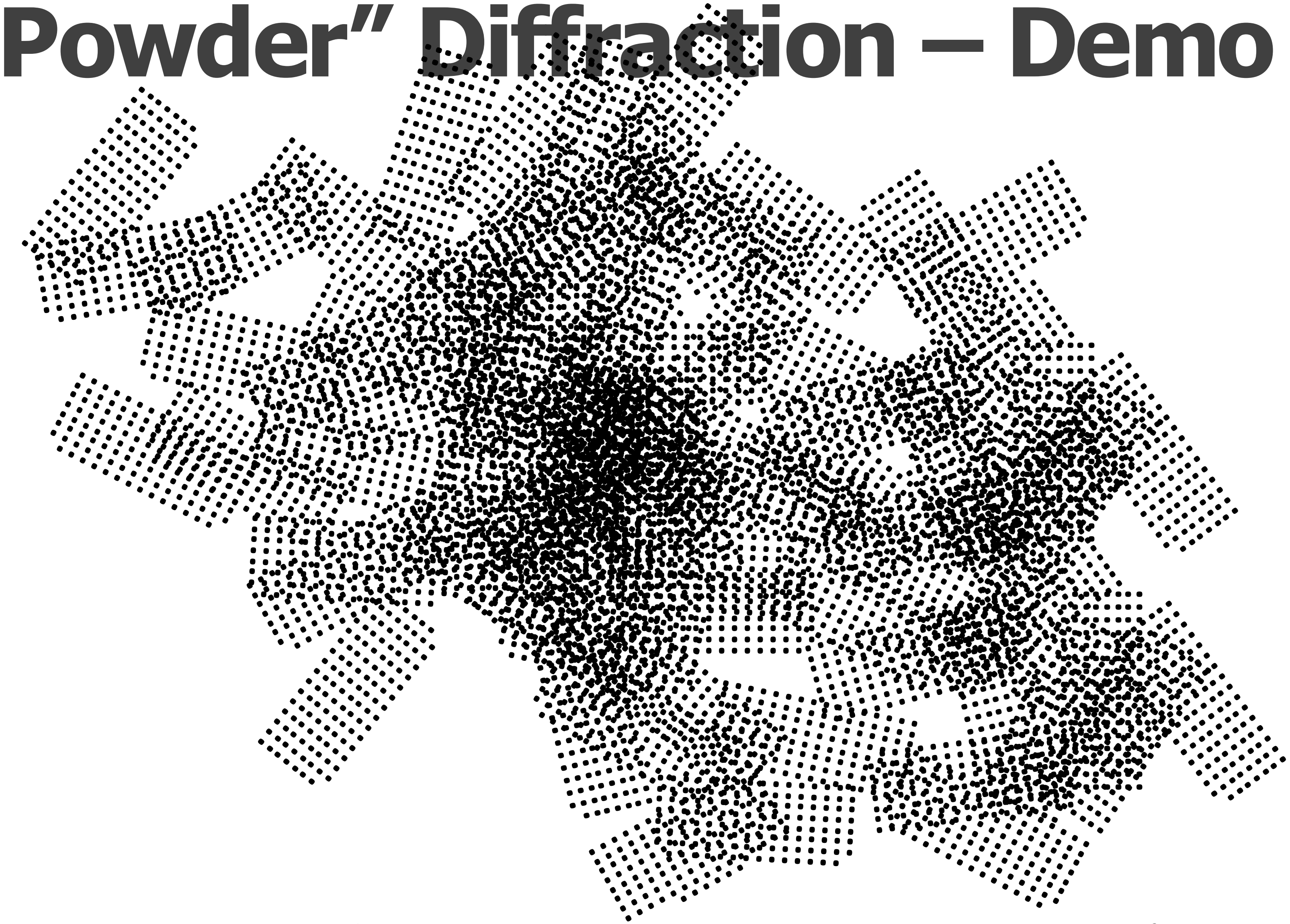
  n is an integer

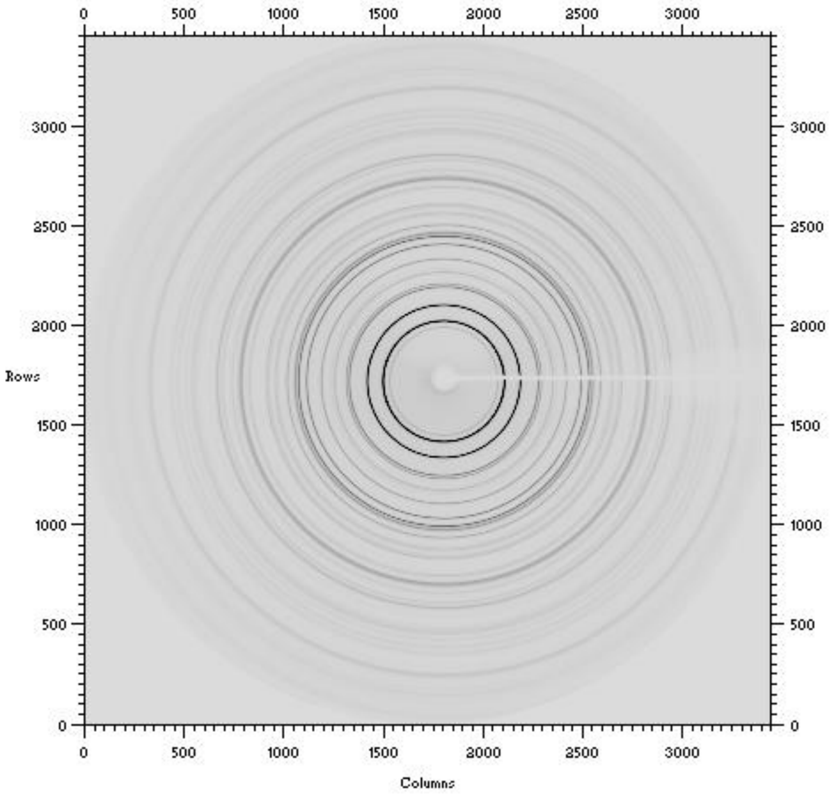
  λ is the X-ray wavelength

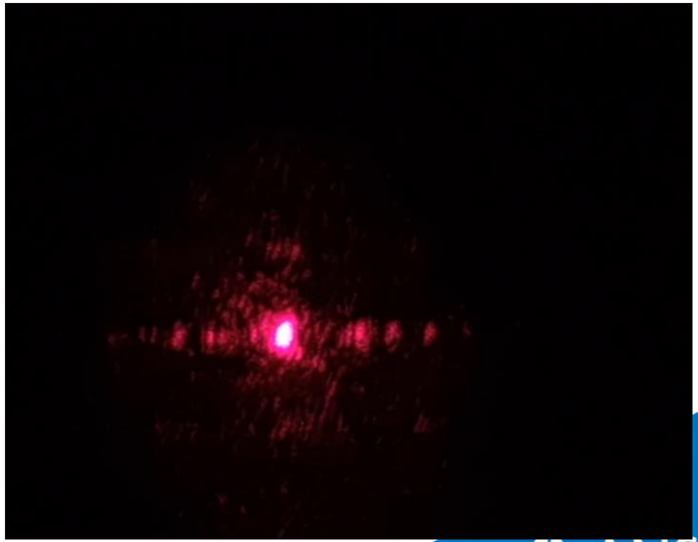
  d is the inter-planar spacing

  2θ is the diffraction angle

"Powder" Diffraction – Demo

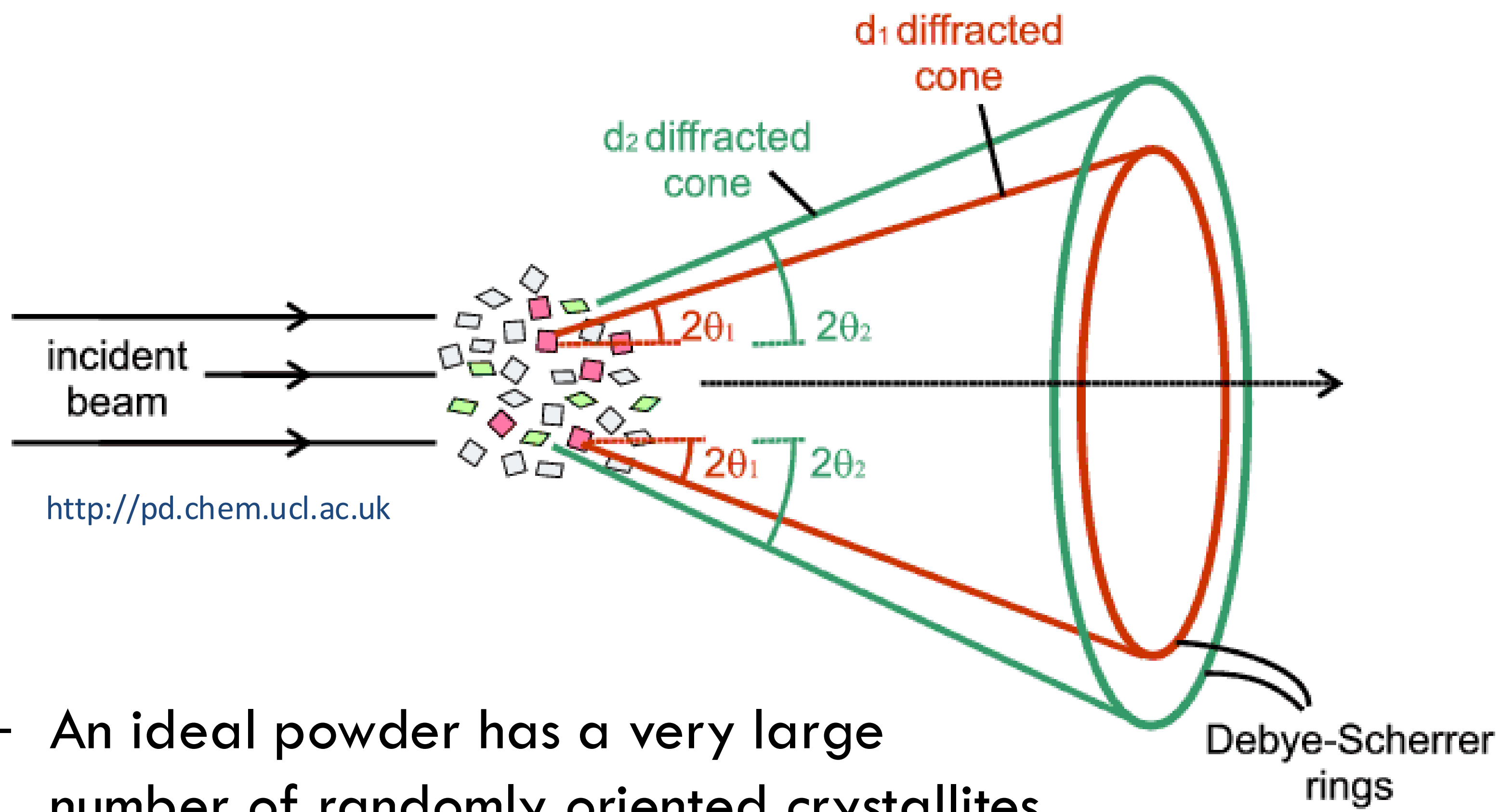




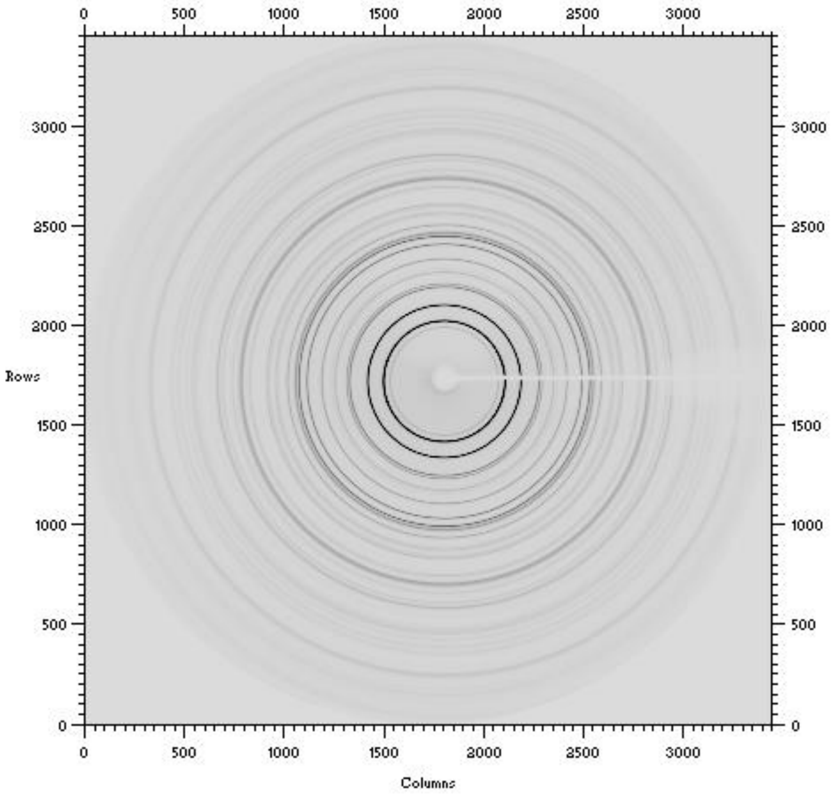


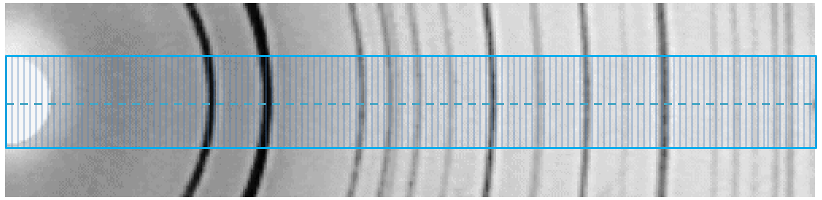
An ideal powder has a very large number of randomly oriented crystallites

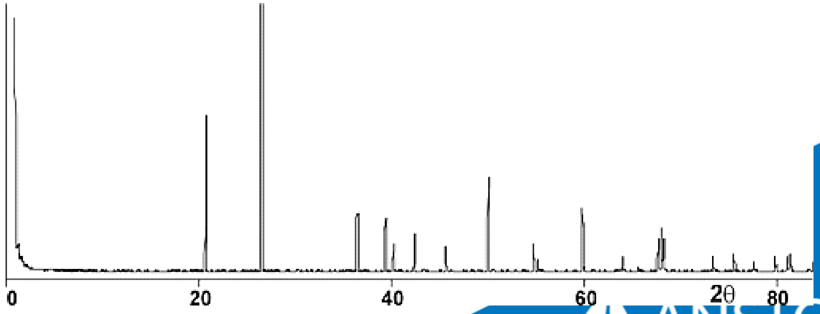
Powder Diffraction – Theory



http://pd.chem.ucl.ac.uk



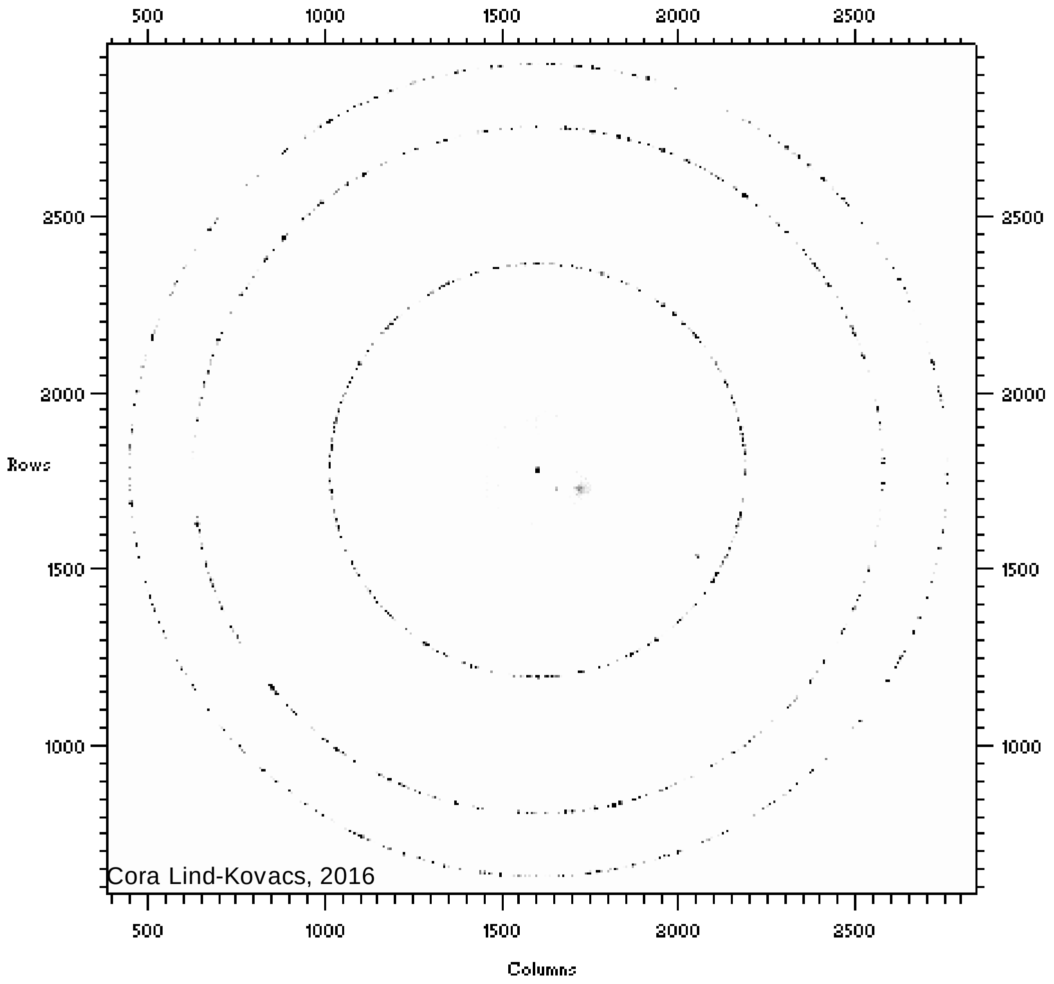




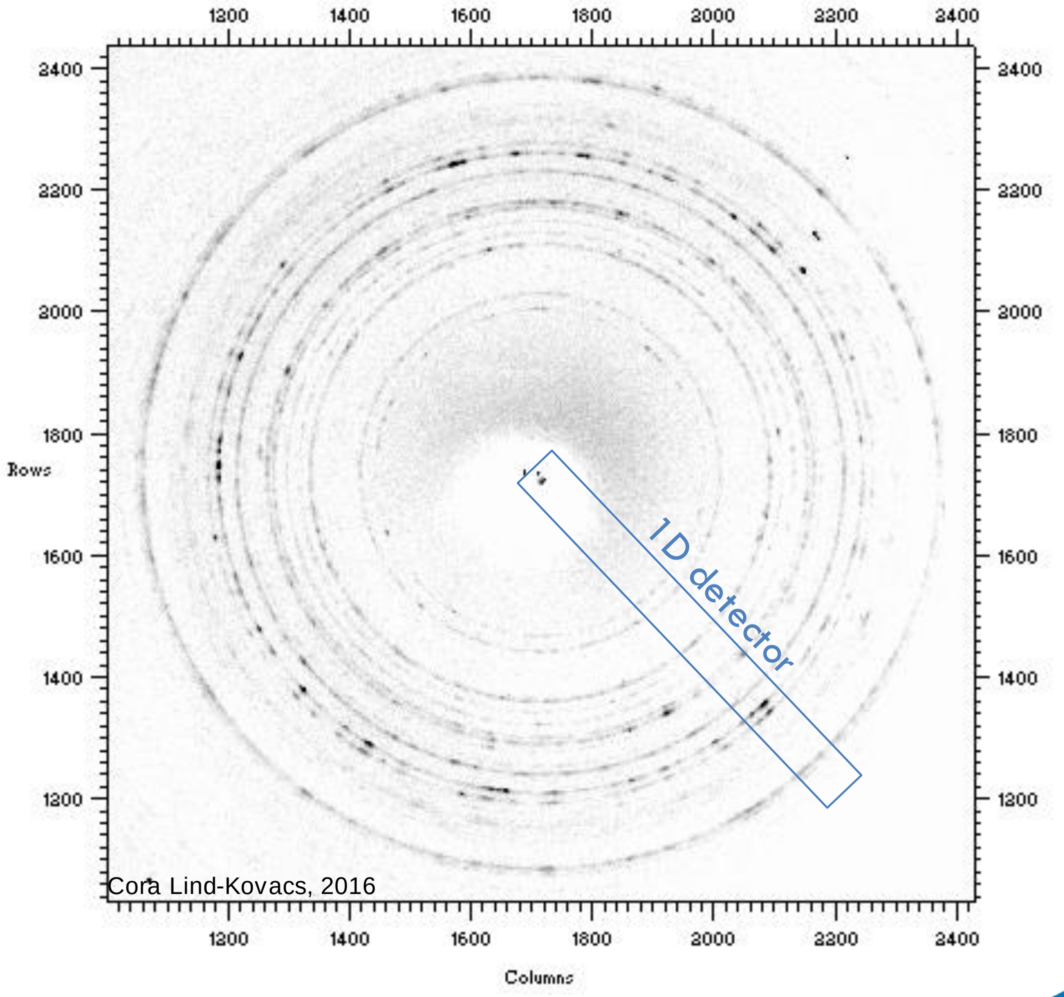
- An ideal powder has a very large number of randomly oriented crystallites.
- For each particular crystal plane spacing (hkl), there will be many crystallites with the correct orientation to satisfy the Bragg diffraction condition.

Imperfect powders

2D area detector images



Very grainy powder



Better, but not ideal

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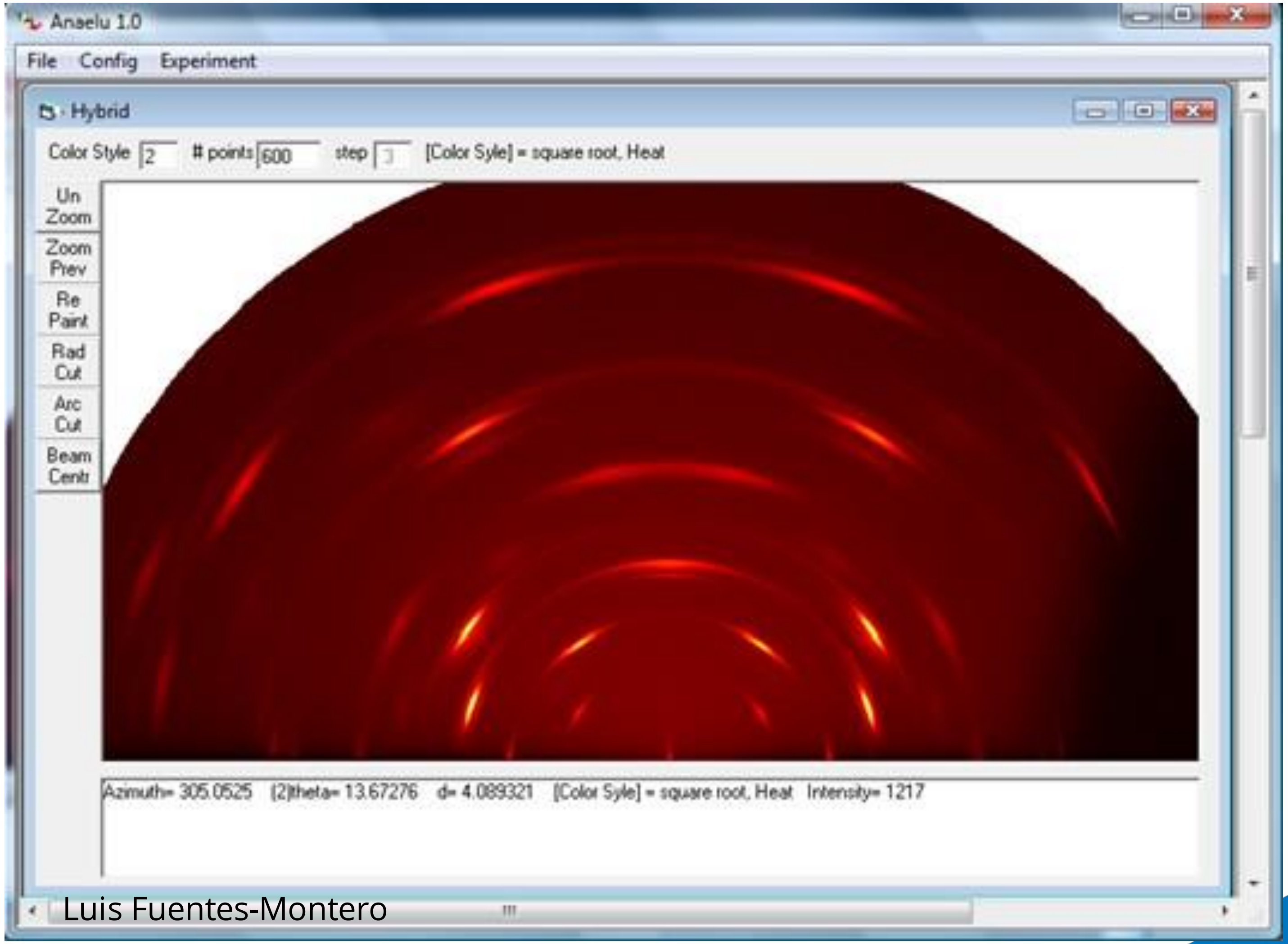
Texture

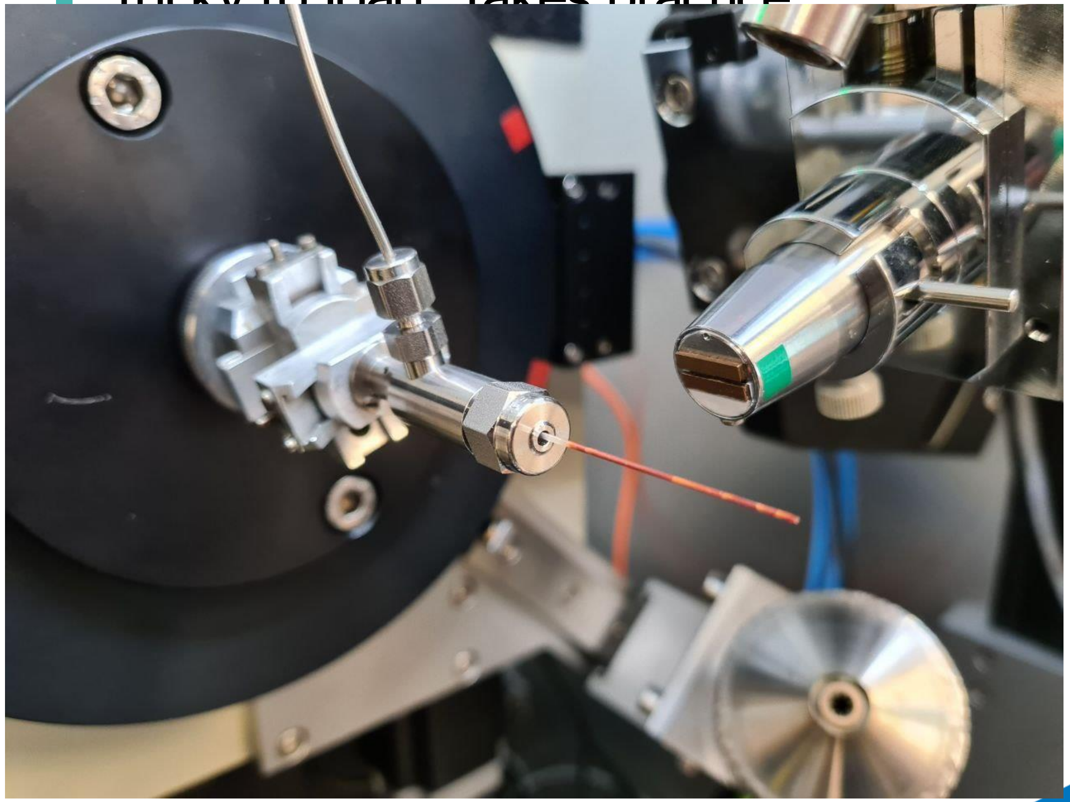
- On the other hand, “texture” can tell us about the non-random orientation of crystallites/grains in our materials.
- XRD is a powerful technique for investigating strain (residual stresses) in materials.
- Can provide information about the directionality of strain.



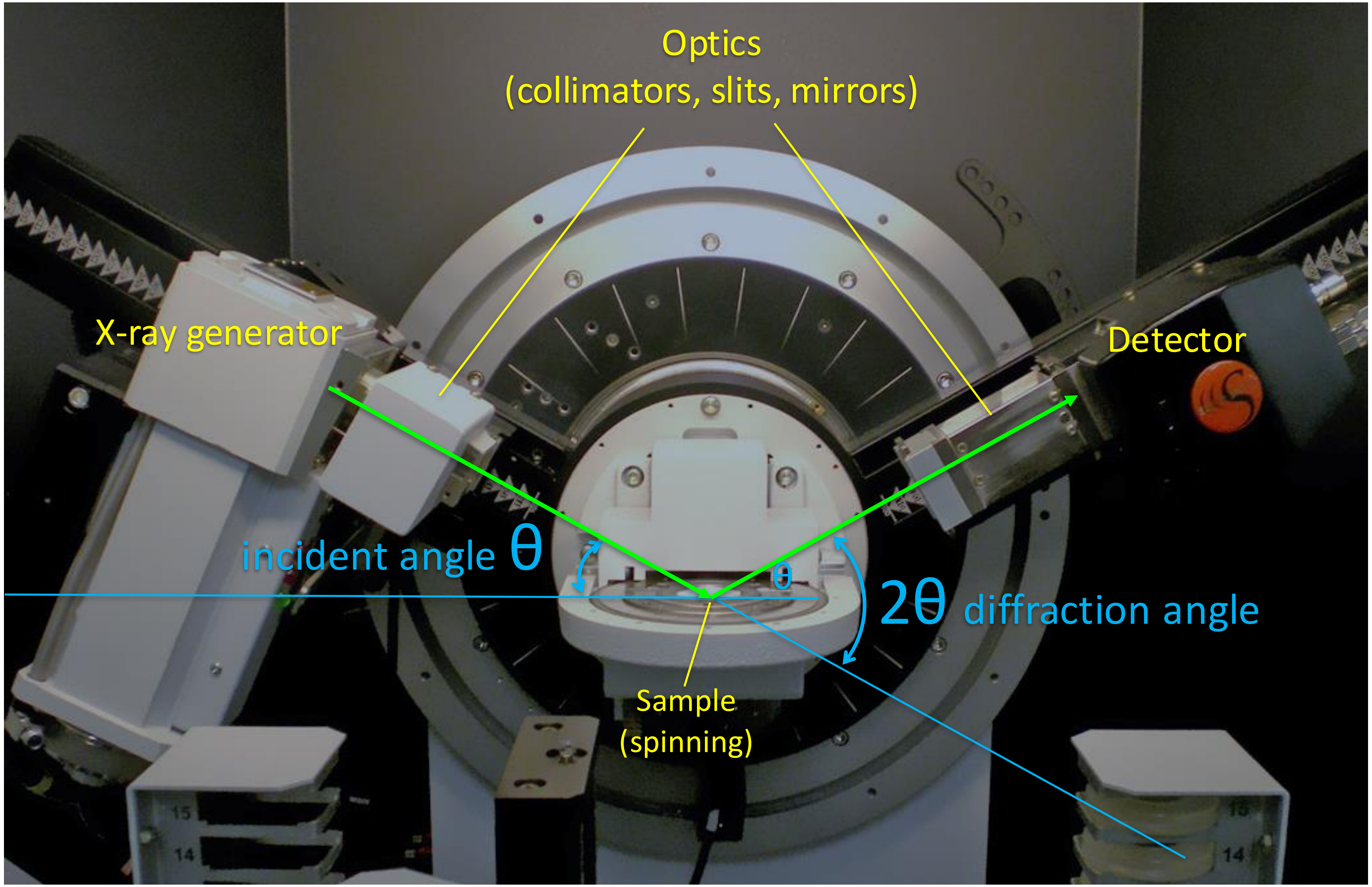
Luis Fuentes-Montero

Samples - capillary

- Glass or quartz capillaries. Sapphire for special uses.
- Diameter typically 0.2–1.0 mm, 0.01 mm wall thickness.
- Capillaries can be sealed to keep air/moisture out or solvents in.
- Tricky to load. Takes practice.



Instrumentation – Bragg–Brentano geometry



- "Flat plate" or "reflection" geometry.
- Large area of sample illuminated.
- Low beam absorption, higher peak intensity.
- Risk of preferred orientation.
- Sample spinning is commonly used.

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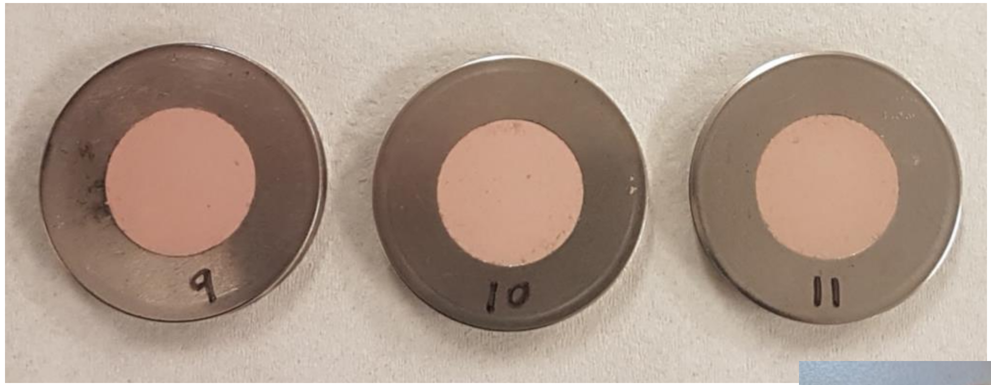
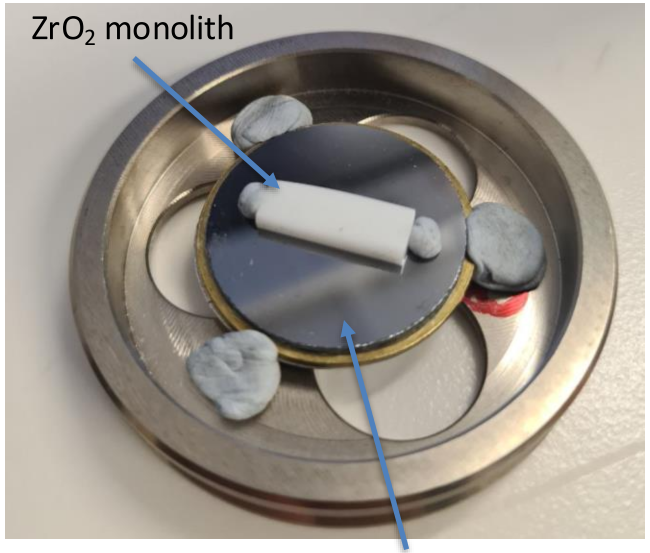
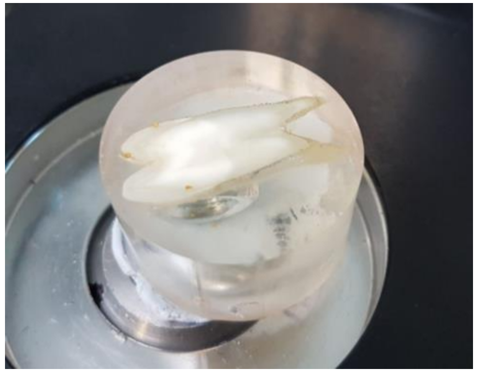
Samples – flat plate

- Ideal crystallite size is 1-10 µm.
- Smooth and flat surface, but polishing not required.
- Correct sample height
- Zero diffraction plates (ZDPs) for small samples.







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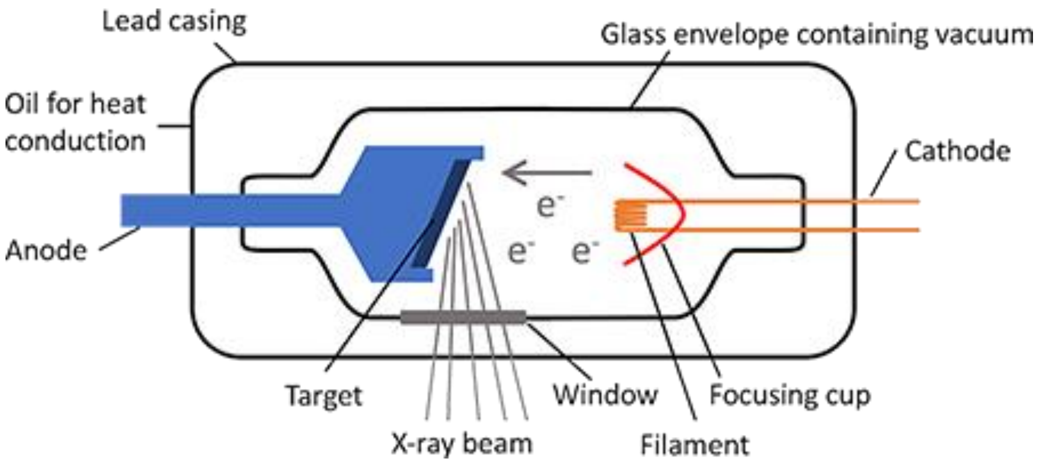
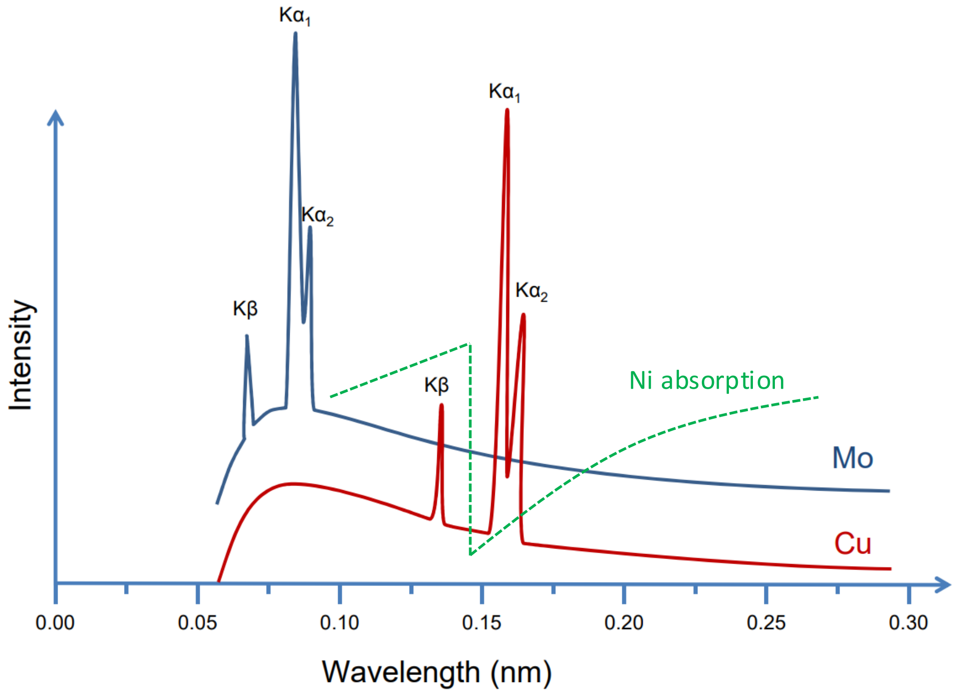
Instrumentation – X-ray generator



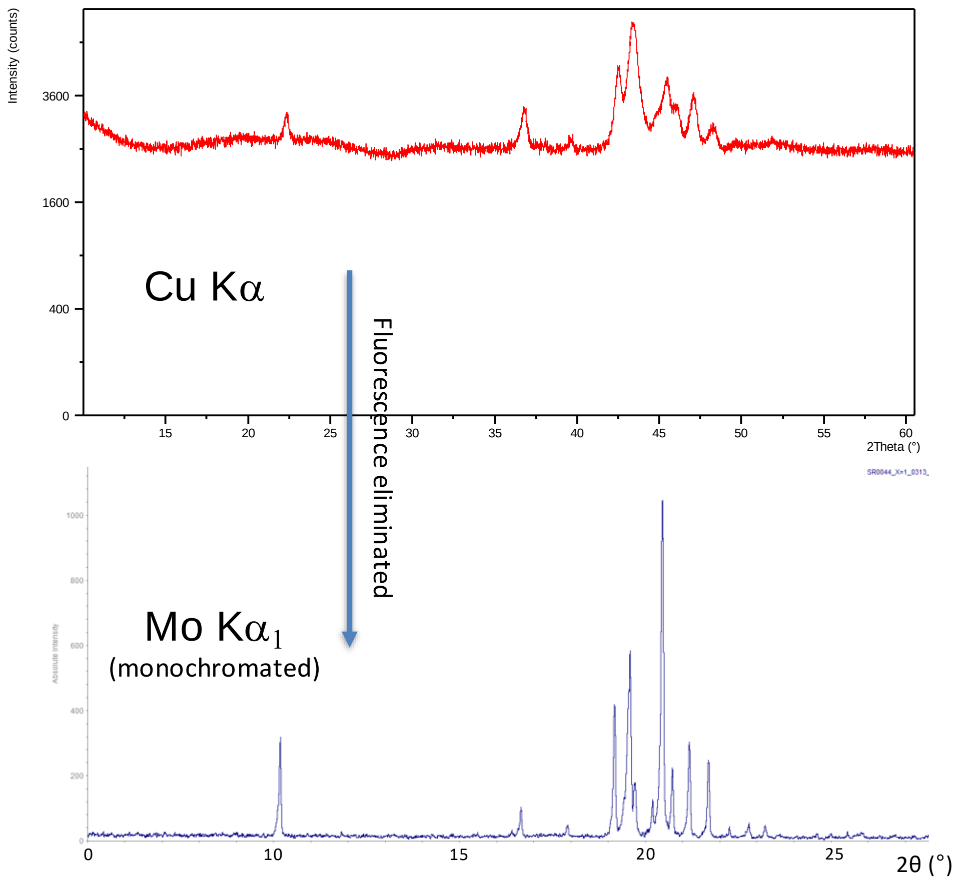


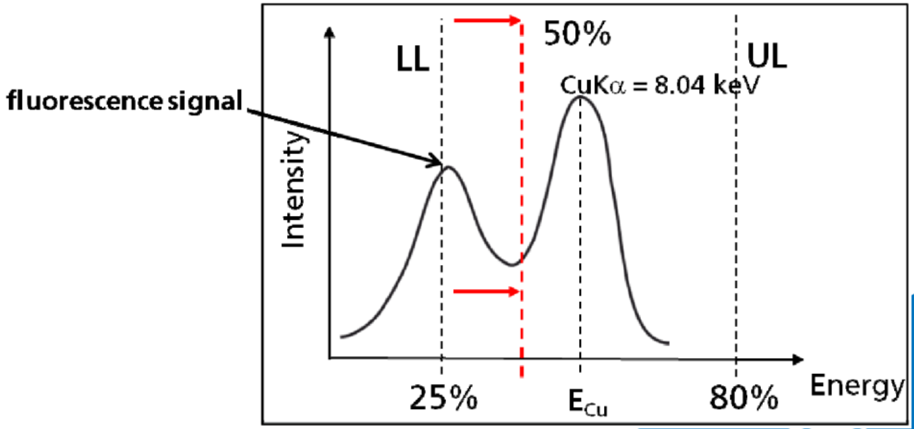
- A Cu target (yielding Cu Kα X-rays) is the most common radiation source for powder diffraction.
- Kβ wavelengths are excluded by a filter (e.g. Ni filters out Cu Kβ).
- Some instruments have a monochromator to select $K\alpha_1$ wavelength only.
- Shorter wavelengths have higher energy and are absorbed less but bring diffraction peaks closer together.

Element	Kα1 (Å)	Kα2 (Å)	Kβ (Å)
Cr	2.2898	2.2937	2.0849
Co	1.7890	1.7929	1.6208
Cu	1.5406	1.5444	1.3923
Mo	0.7093	0.7136	0.6323
Ag	0.5594	0.5638	0.4971

X-ray fluorescence

- Elements in the sample may emit fluorescent X-rays (e.g. Co, Fe fluoresce strongly when illuminated by Cu Kα X-rays).
- If the emitted X-rays are close in energy to the incident wavelength they will be detected. This manifests as an elevated background and gives a reduced peak-to-background ratio.
- Some solutions:
  - Use a different wavelength if available.
  - Change the detector energy threshold.





Detector pulse height distribution (PHD) window tuning

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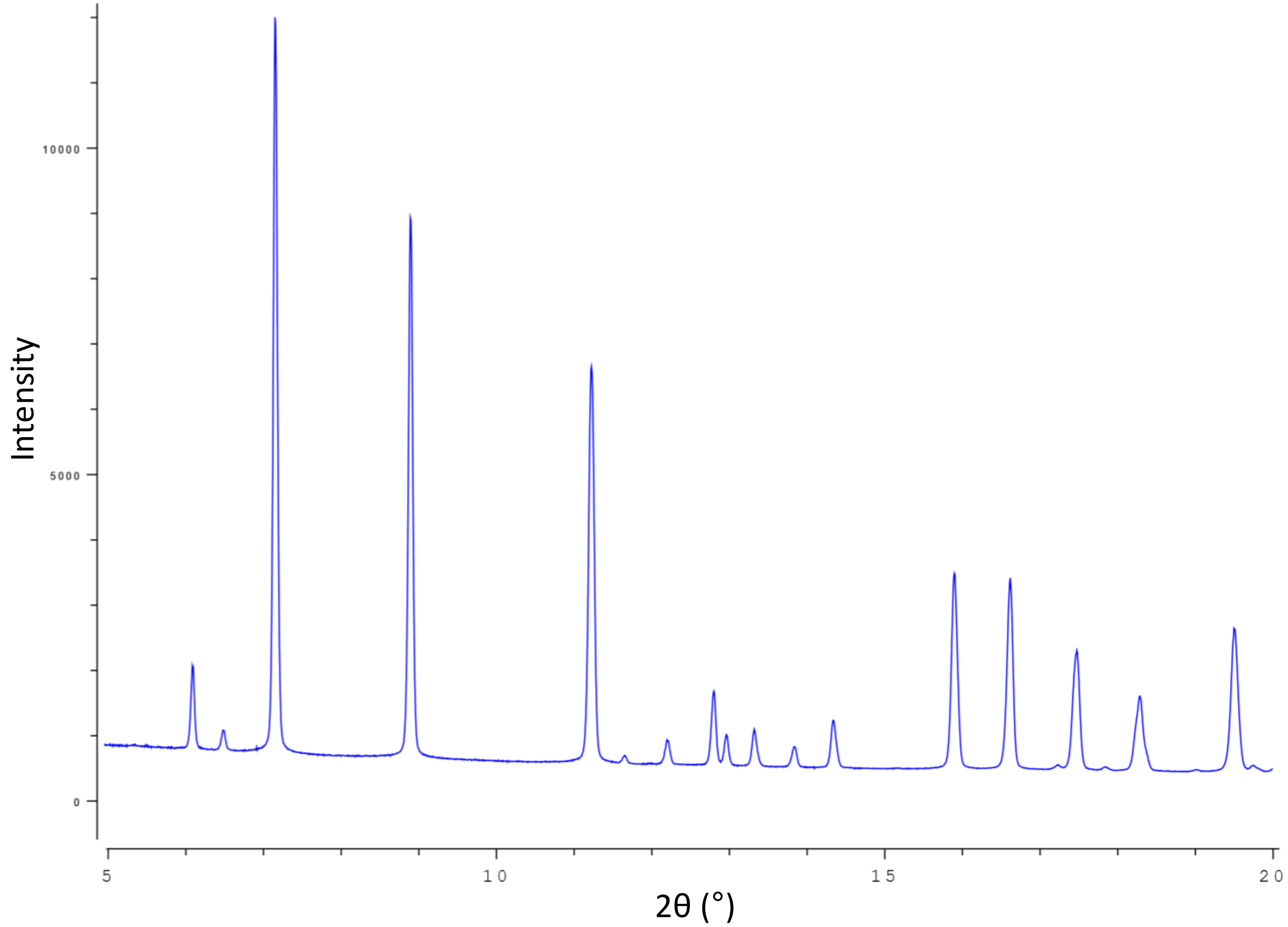
Diffraction Data – What do we get?



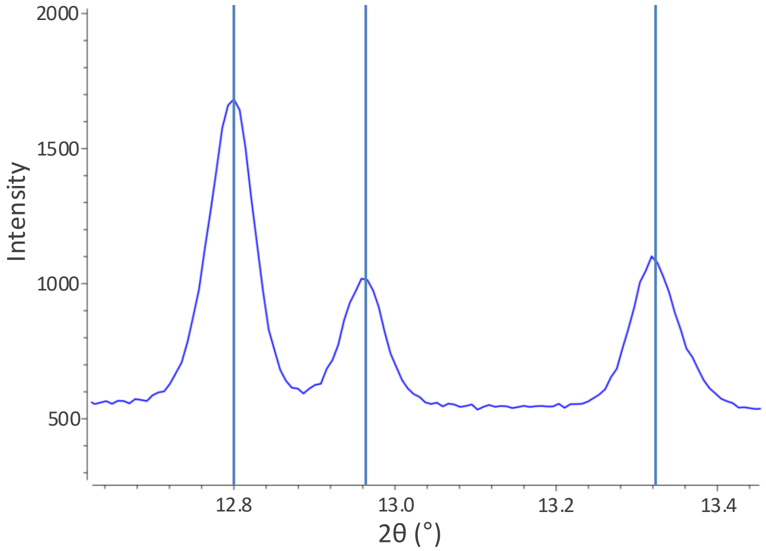
We call the measurement a diffraction pattern or diffractogram
(not a spectrum!)

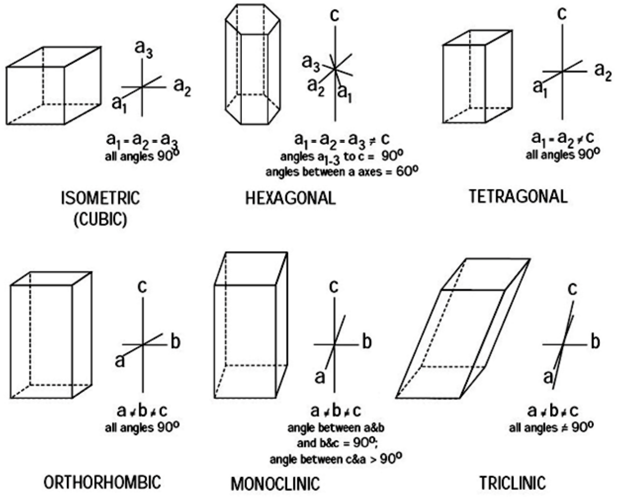
Each phase in the sample gives us a set of peaks
(a "fingerprint")

Important features:

- Background
- Peak positions
- Peak shapes
- Peak intensities

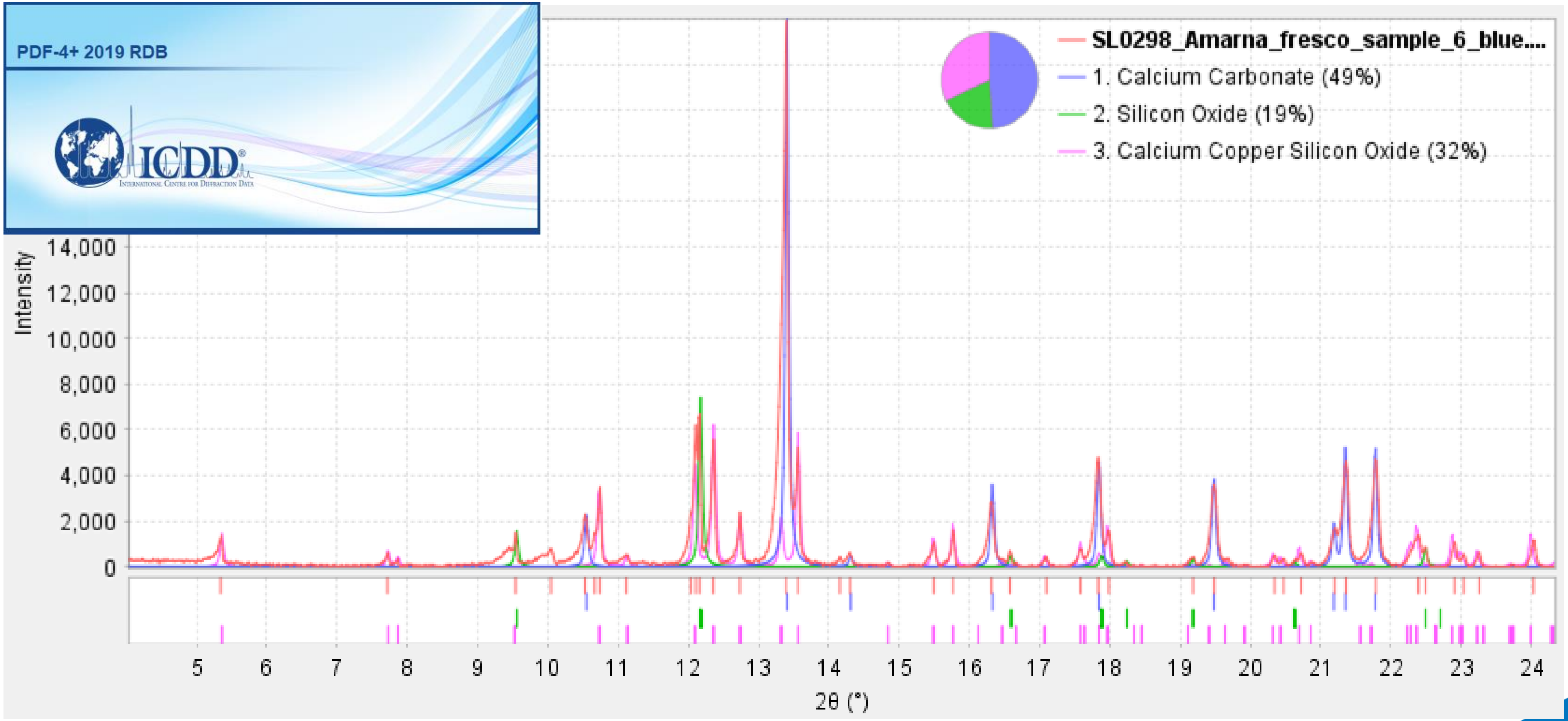
Diffraction Data – Peak positions





- Peak positions can be used to obtain:
  - d-spacings
  - Unit cell dimensions
  - Crystal class (cubic, hexagonal, etc.)
  - Possible space groups (from systematic absences)
  - Qualitative phase analysis (what's in the sample?)

XRD – Database search & match



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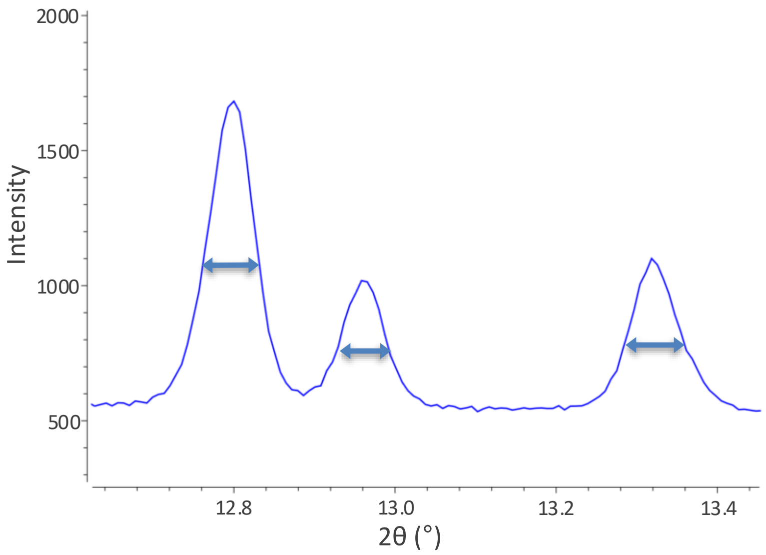
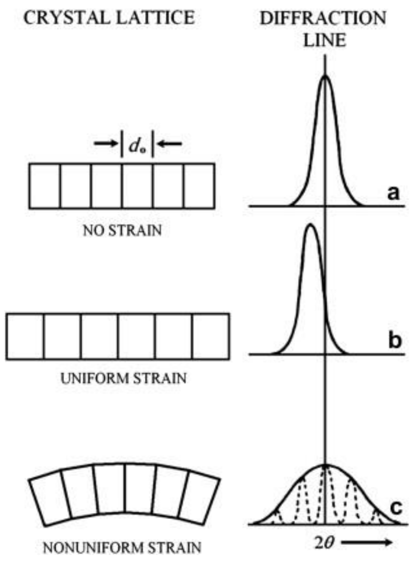
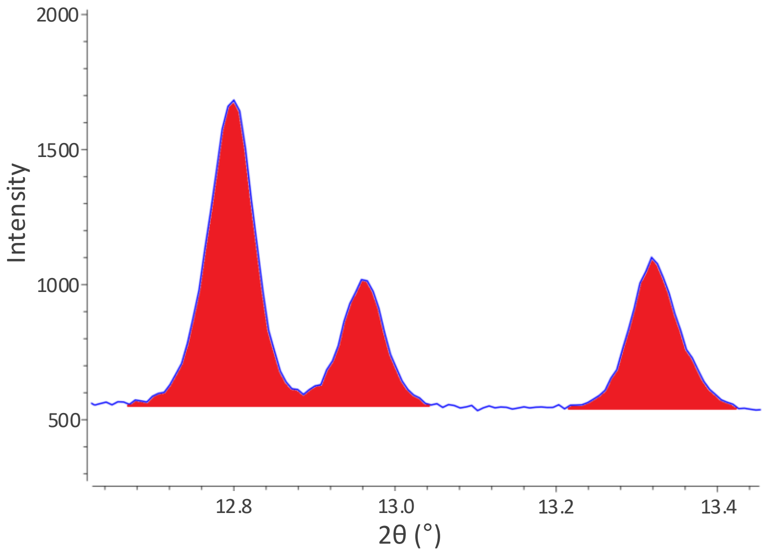
Diffraction Data – Peak Shapes

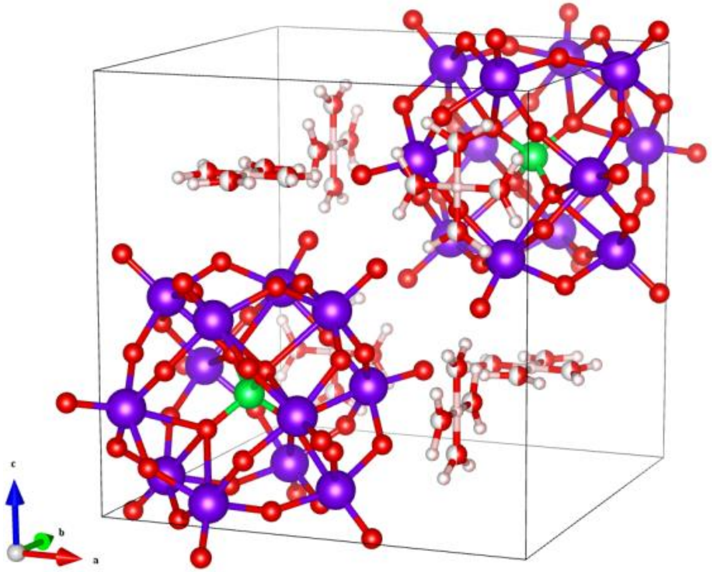




- Peak shapes are affected by:
  - Instrument – finite source size, axial divergence, slit dimensions, detector resolution.
  - Crystallite size below ~100 nm – can determine size from peak broadening (Scherrer equation).
  - Microstrain – a "range" of lattice parameters due to strain. Can be isotropic or anisotropic.
  - Ordered defects – Stacking faults, antiphase boundaries.
- For quantitative analysis, a standard with no strain or size broadening must be measured.
  e.g. Si, LaB_6, Al_2O_3, …

Diffraction Data – Peak Intensities





- Intensity of diffraction peaks (hkl) is proportional to the square of the structure factor amplitude

$$F(hkl) = \sum_{j=1}^{N} f_j \exp(2\pi i(hx_j + ky_j + lz_j))$$

- Intensities give information about:
  - Positions and types of atoms
  - Site occupancy of atoms
  - Atomic displacement parameters
  - Orientation of crystallites/grains
- Use **integrated** peak intensities (area under peak)
- Accurate intensities are required for quantitative phase analysis and structure refinement.

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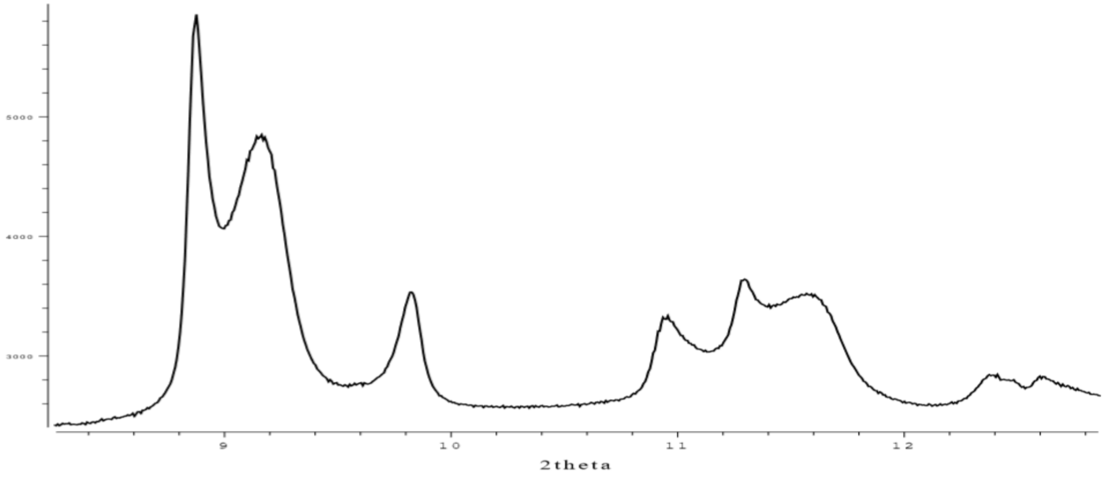
Analysis – How do we get the information out?

- Initially, powder methods were used mainly for phase identification – “fingerprinting”
- 1966-1969: Hugo Rietveld recognised the growing power of computers in crystallography and developed a “whole pattern” fitting approach to powder diffraction.
- This overcame the problems associated with overlapping reflections.

Hugo Rietveld



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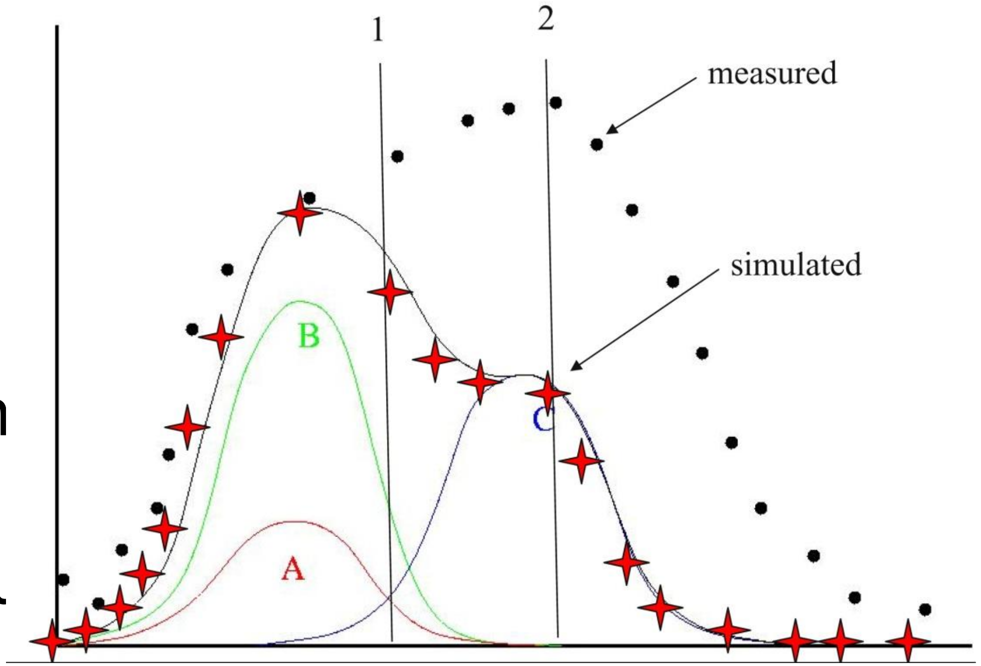
The Rietveld Method

- Each data point is an observation.
- Least-squares algorithm used to obtain the best fit of the structural model to the powder pattern.
- Refinement of multiple phases and even multiple datasets is possible.
- Requires an approximate starting structu and accurate intensities.
- Parameters can be highly correlated.
- Restraints/constraints may be required for limited data.



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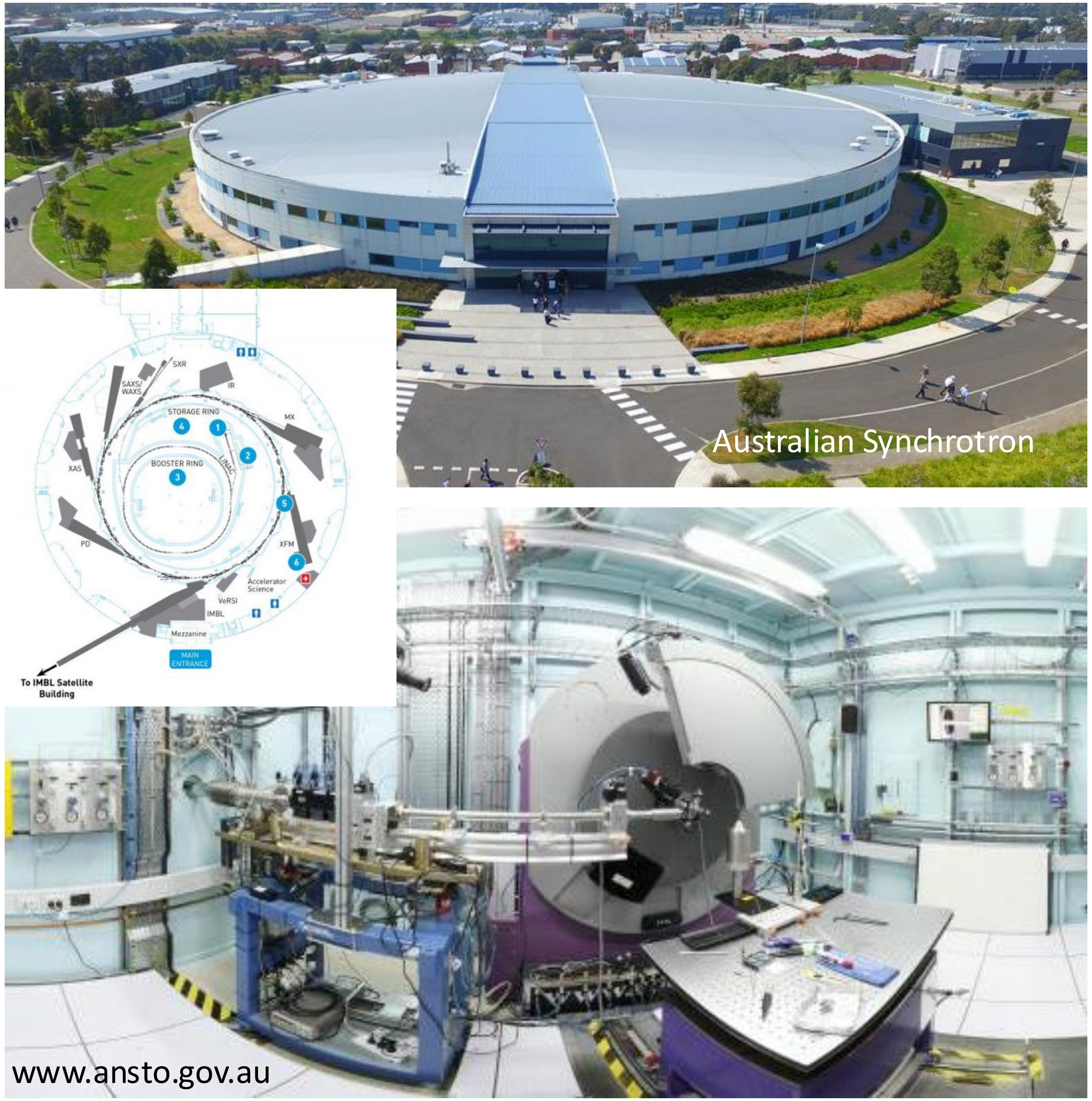
Parameters in Rietveld Refinement

- Structural variables
  - Atom positions, fractional occupancies, atomic displacement parameters (ADPs) (in most single crystal software, these are the only refinable parameters)
- Profile parameters
  - Background
  - Peak shape, including width and asymmetry
  - Unit cell constants
  - Wavelength
  - Diffractometer zero point
  - Sample height and displacement relative to instrument centre
- Correction terms
  - Absorption
  - Extinction
  - Surface roughness
  - Preferred orientation

Other Radiation Sources - Synchrotrons



- Synchrotrons are particle accelerators that use high speed electrons to produce X-rays.
- Synchrotron X-ray beams are:
  - Highly monochromatic, yielding narrow peaks
  - Tuneable in wavelength
  - Extremely intense and highly focused
- Special detectors
- Advanced sample environments
- Smaller samples possible
- Mapping of samples feasible

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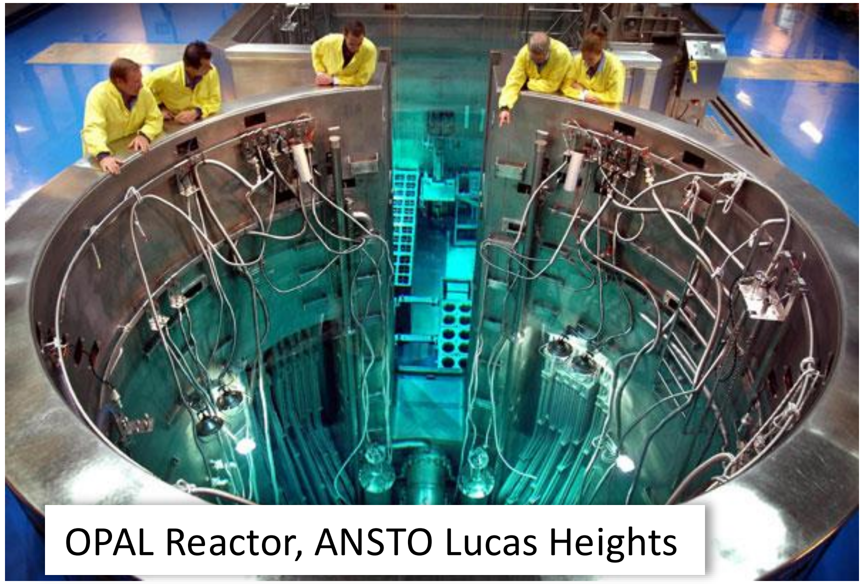
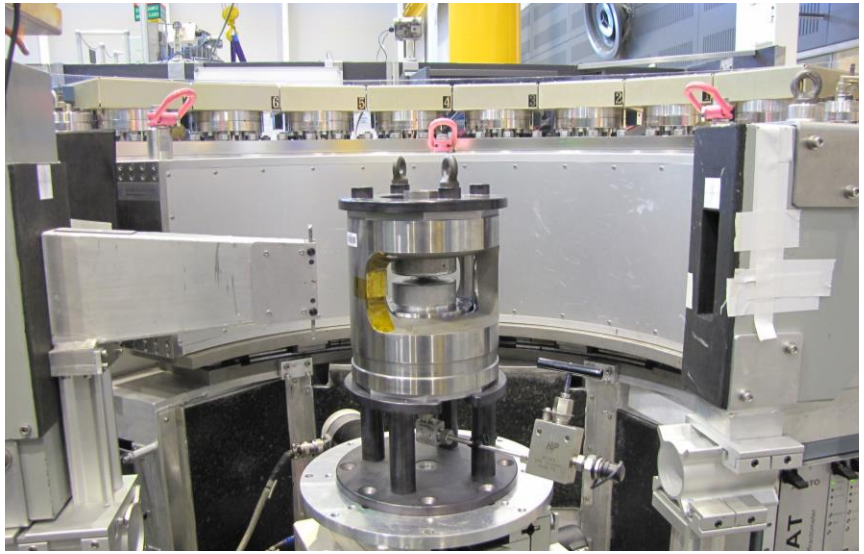
Other Radiation Sources - Neutrons



OPAL Reactor, ANSTO Lucas Heights



WOMBAT neutron powder diffractometer

- A neutron has a wavelength, dependent on its velocity $\lambda = h/mv$
  (λ is wavelength, h is Planck's constant, m is mass and v is velocity)
- Neutrons are produced by reactor or spallation (accelerator) sources.
- Neutrons interact with the nuclei of atoms and can penetrate bulk samples and sample environments.
- Determination of magnetic structure.
- Typically neutron sources have lower intensity. Measurements take longer.
- Time-of-flight experiments are possible.

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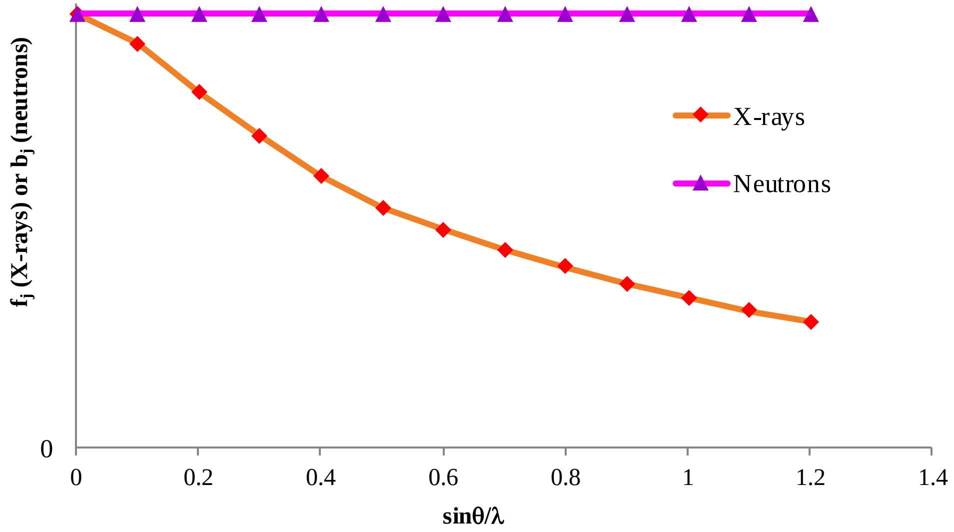
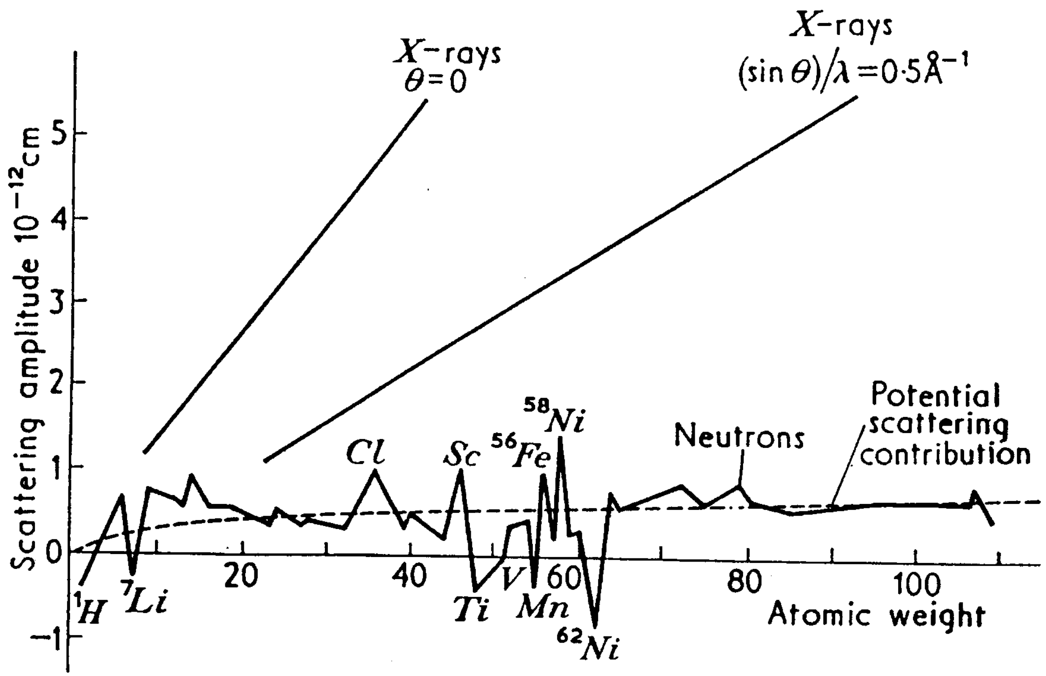
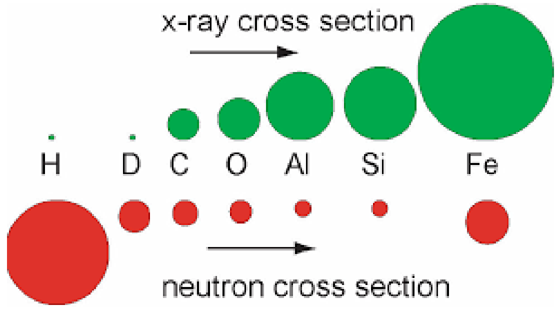
Other Radiation Sources - Neutrons





- The X-ray scattering power of atoms depends on No. of electrons in the atom and on $Q = 4\pi \sin\theta/\lambda$
- Scattering power for neutrons depends on isotope, independent of Q.



– For neutron scattering we need to consider incoherent scattering, which adds to the background.

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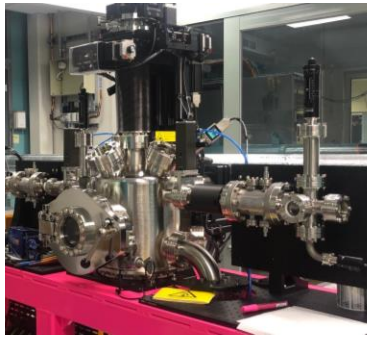
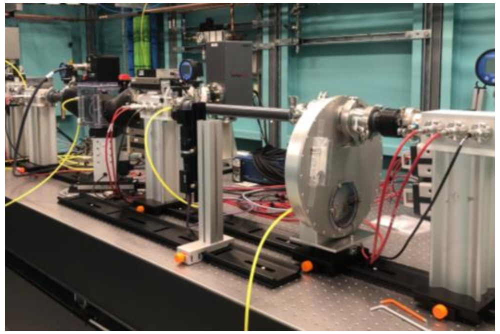
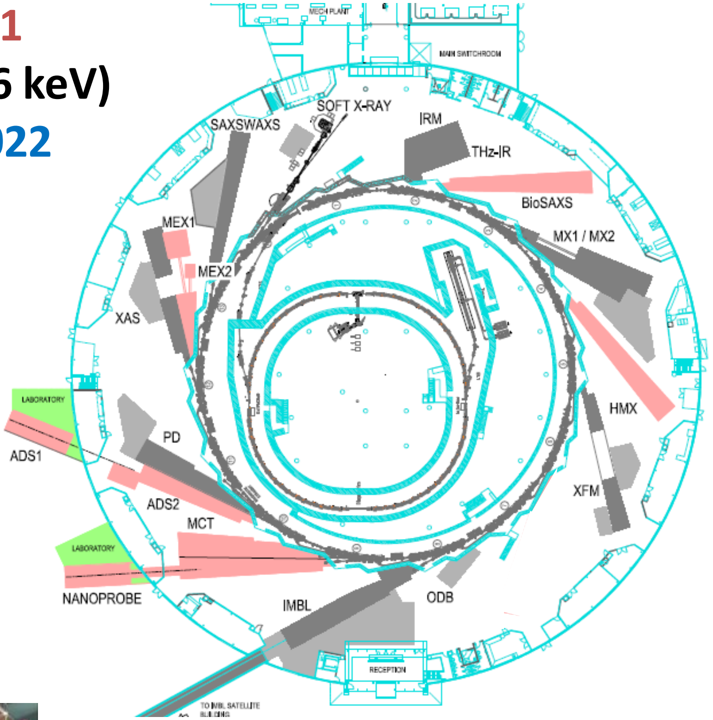
Why come to us?

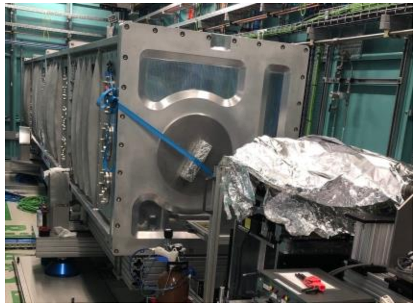
The BRIGHT Beamline Program: $100M (2022 – 2025)





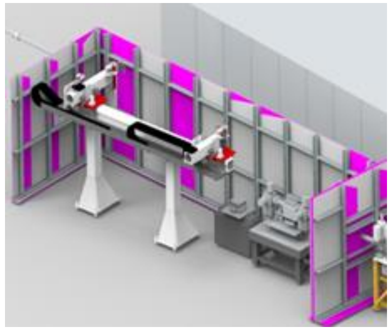
MEX-1
(3.5 – 13.6 keV)
Nov 2022



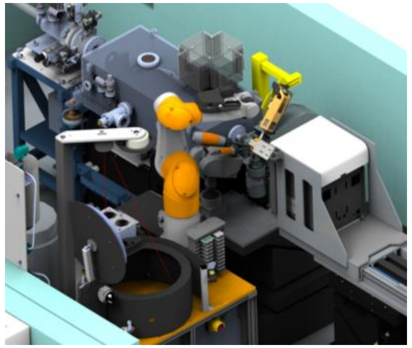


BioSAXS
(8-15 keV)
mid-2023

MEX-2
(1.3 – 3.5 keV)
mid-2023



ADS-2
(45 – 90 keV)
2025



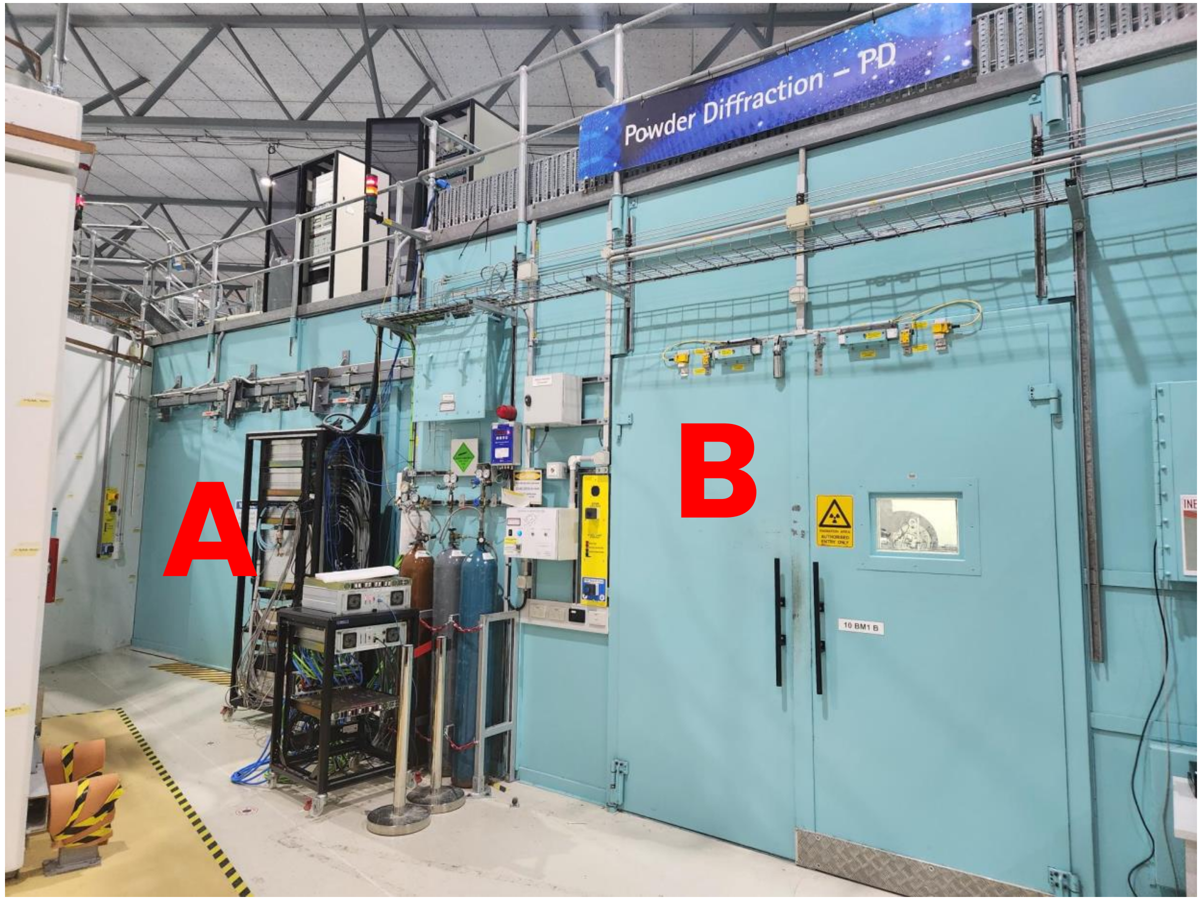
ADS-1
(50 – 150 keV)
2025

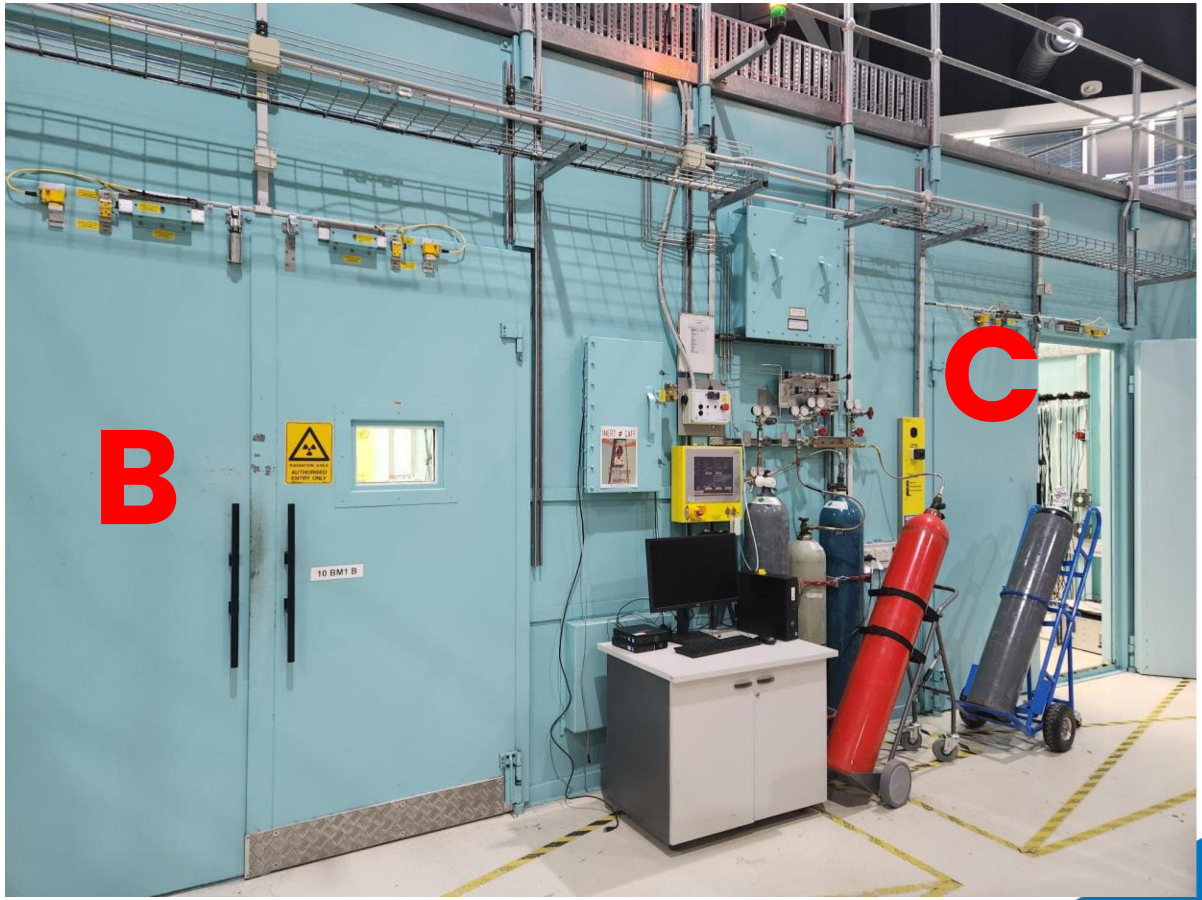
Micro-CT
(8 – 40 keV)
Sept 2022

MX3
(10-15 keV)
2024

NANO
(5–18 keV)
2025

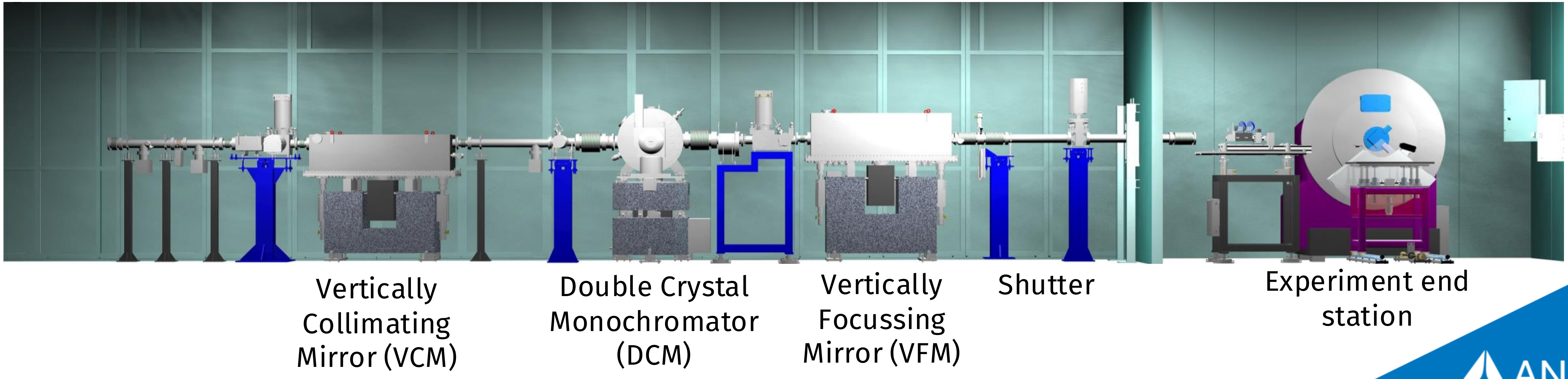
Hutches at PD



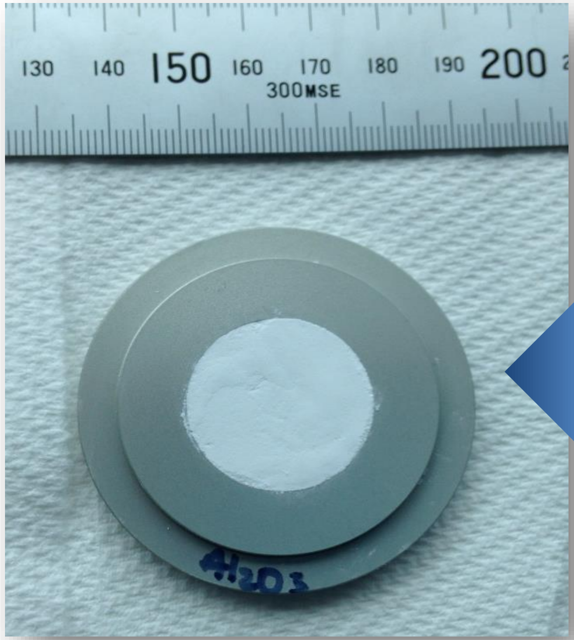


Technical details

Energy:	8 - 22 keV (0.56 - 1.54 Å)
VCM/VFM:	Modes for experiments in 1st or 2nd endstation
DCM:	Si (111) flat crystal pair
Beam size at sample:	0.8 mm (H) x 0.8 mm (V)



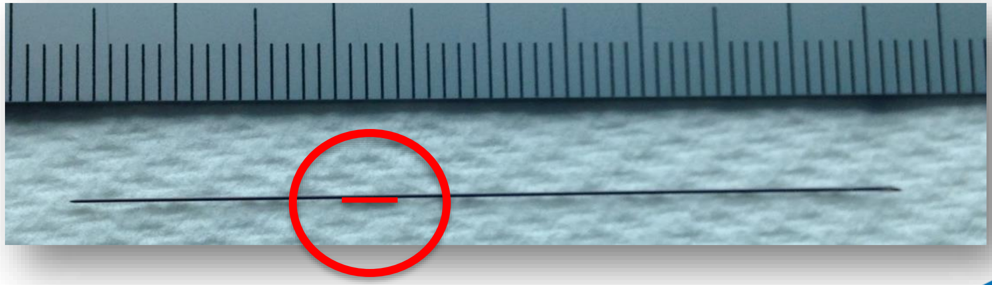
Tiny samples



Lab sample

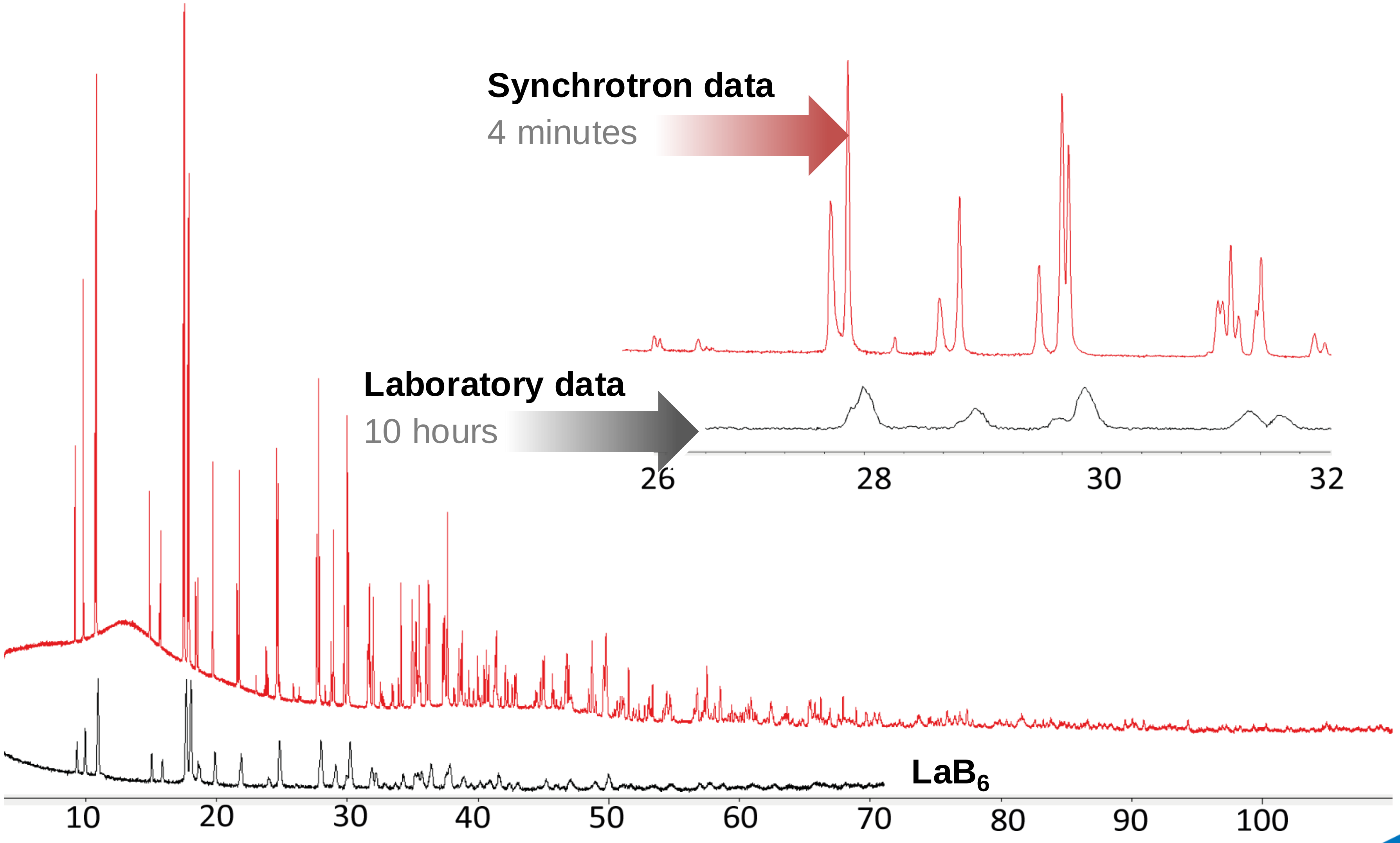
- Large amount of material required
- Sample loading can produce texture effects

Synchrotron sample

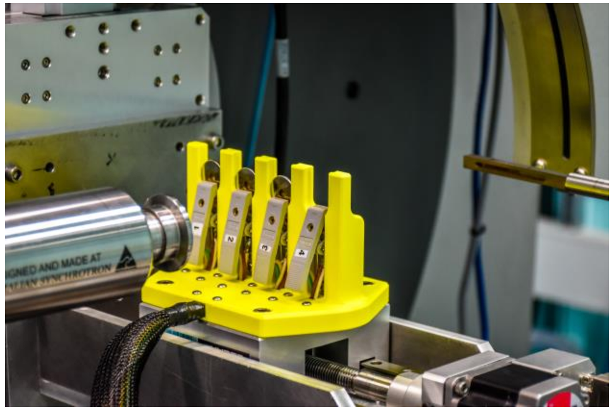


ANSTO

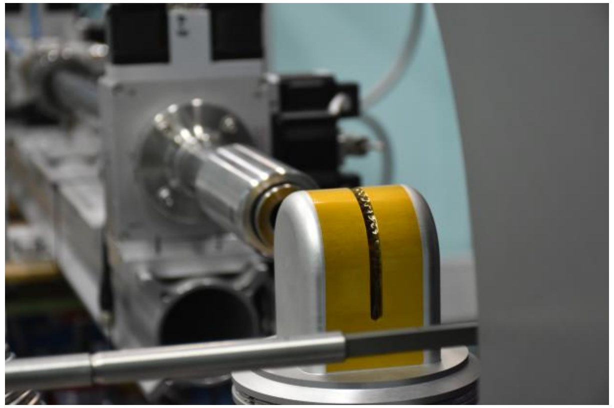
Speed + Resolution



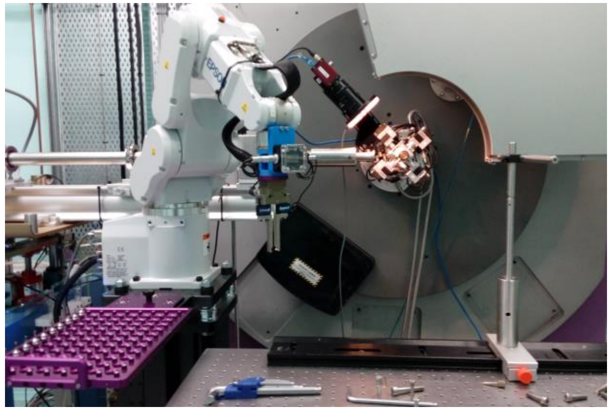
Sample environments



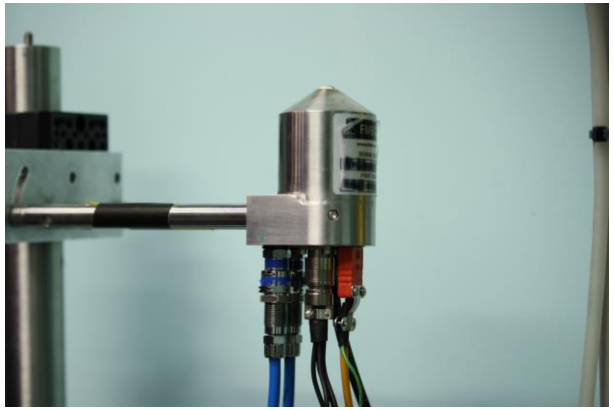
Battery tester



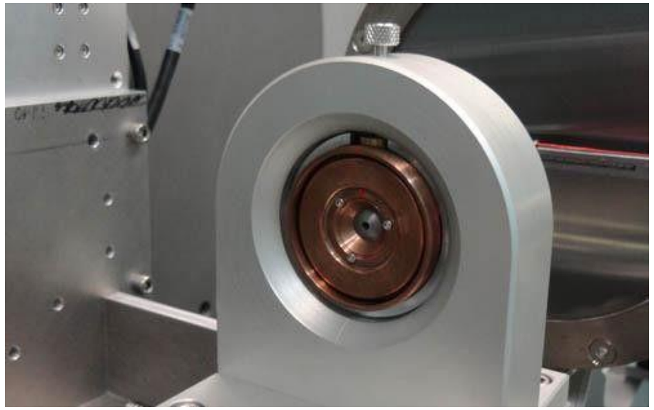
Cryostat (10 – 300 K) and



High throughput robot

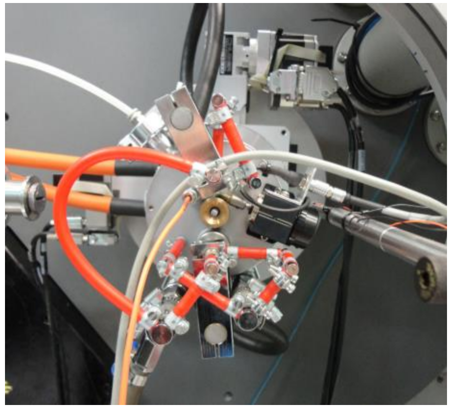


High Temperature
(RT – 950 °C)

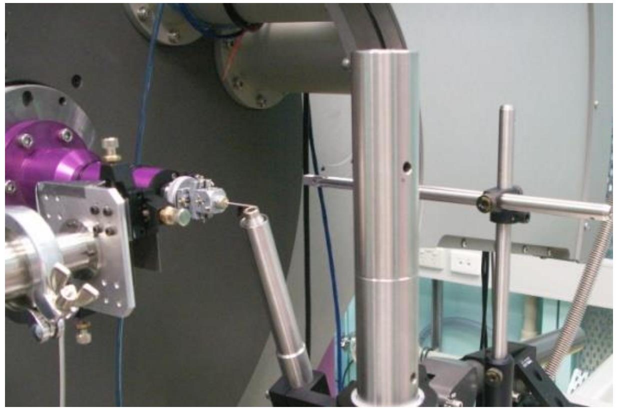


High Pressure - Diamond Anvil
Cells (0 – 10 Gpa)

Sample environments

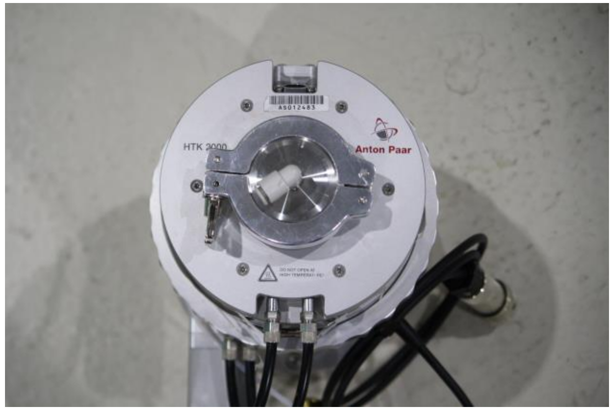


Capillary furnace

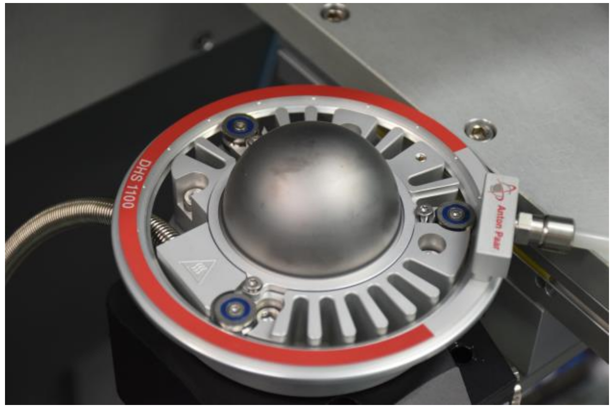


Cryostream (80 – 480 K)

Gas environments
Flowing or static

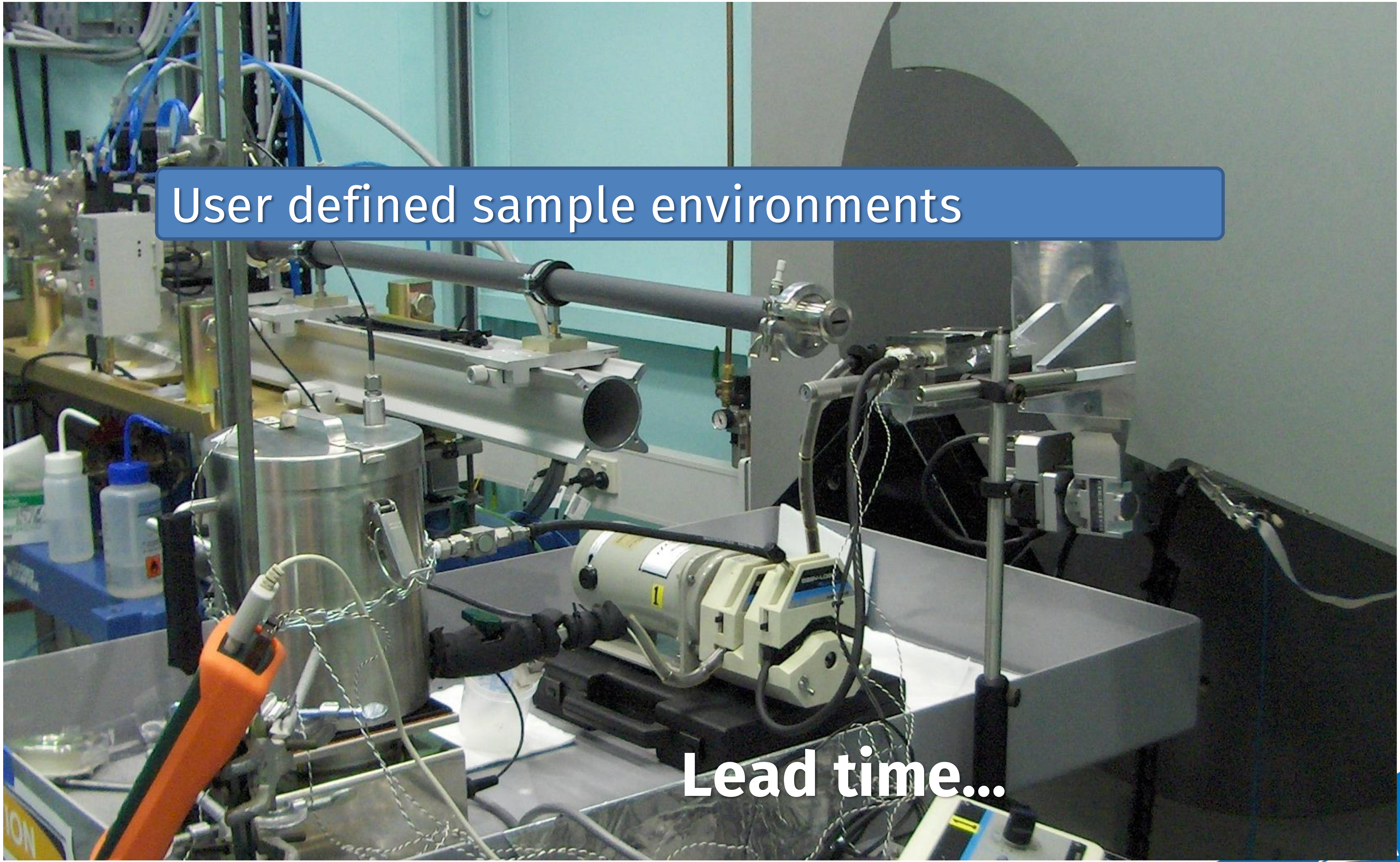


Strip furnace
(RT – 1600 °C)



DHS 1100
(RT – 1100 °C)

User defined sample environments



Lead time...

ANSTO

Australian Synchrotron

How climate change is erasing the world's oldest rock art

The Challenge

Australasia is home to some of the oldest rock art motifs in the world. In tropical latitudes, due to climate change, the rock art deterioration is accelerating. **This study asked why and how can we preserve them?**

The Solution

The PD Beamline was able to identify both the rock art pigments and the salts that form destroying the art. This helped determine the mechanisms of decay

The Impact

Minimising the impacts of climate change will help preserve the incredible artworks Australasia's earliest people left to us

The effects of climate change on the Pleistocene rock art of Sulawesi
J Huntley, M Aubert, AA Oktaviana, R Lebe, B Hakim, B Burhan, LM Aksa, ...
Scientific Reports 11 (1), 9833

ANSTO

Australian Synchrotron

Probing the depths of the sea

The Challenge

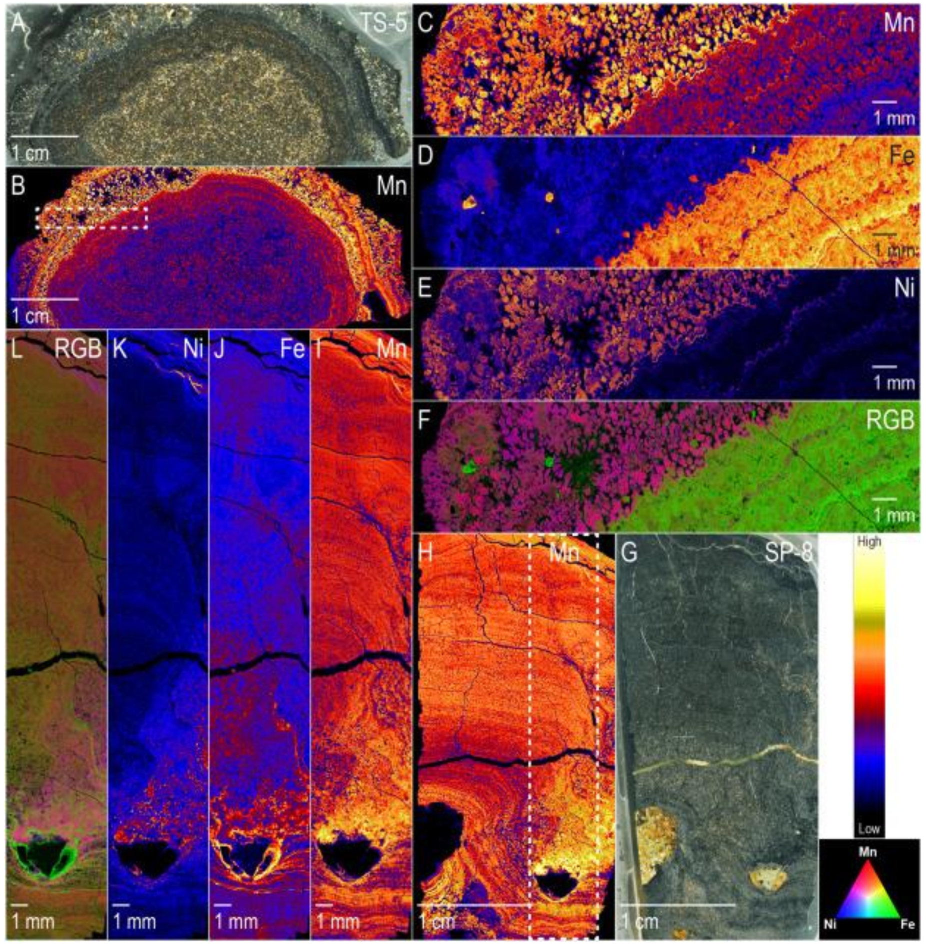
To understand how Ni^{2+} behaves during (bio)geochemical redox cycling. This is important to gain vital information about changes in ocean circulation and ocean productivity.

The Solution

The ***Powder Diffraction Beamline*** is able to identify and quantify trace amounts of phases in complex mineral mixtures

The Impact

- Ni^{2+} exchanges in the presence and absence of Mn^{2+}, making metal concentrations in seawater determined from deposits unreliable
- Gain insight into ocean and climatic evolution
- Understand biogeochemical manganese cycle



Hens, T. *et al. Chem. Geol.* **2019,** *528*, 119276.

ANSTO

Australian Synchrotron

Dolomite for low cost thermochemical energy storage

The Challenge

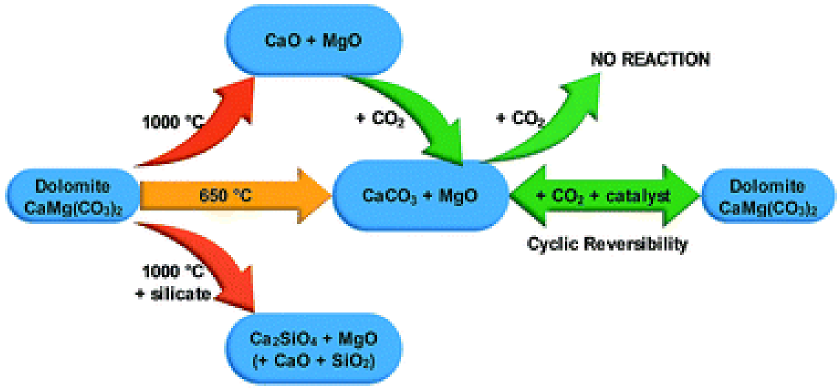
Increase the energy density and reduce the amount of storage material required in concentrating solar thermal power plants. This is important to lower the cost of solar energy production.

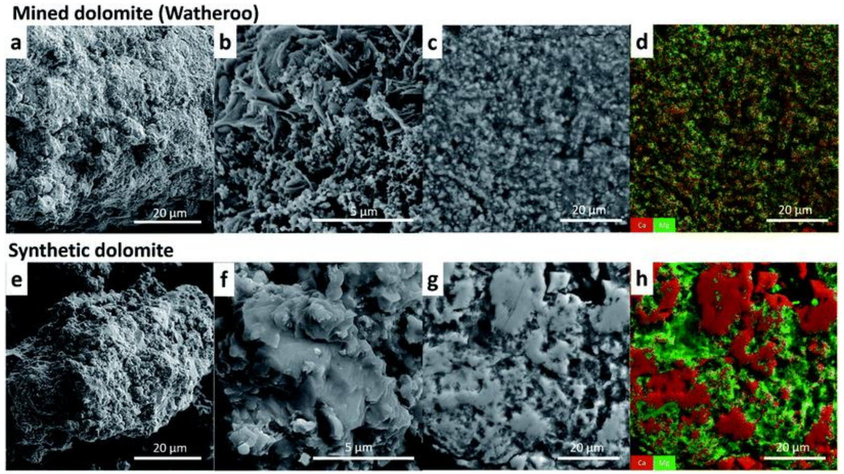
The Solution

The ***Powder Diffraction*** beamline is able to carry out in situ experiments to establish the phase changes and presence of trace phases that may form during reversible CO_2 absorption.

The Impact

- Lower cost
- Less material is needed
- Greater uptake in solar energy by the industrial and commercial sectors

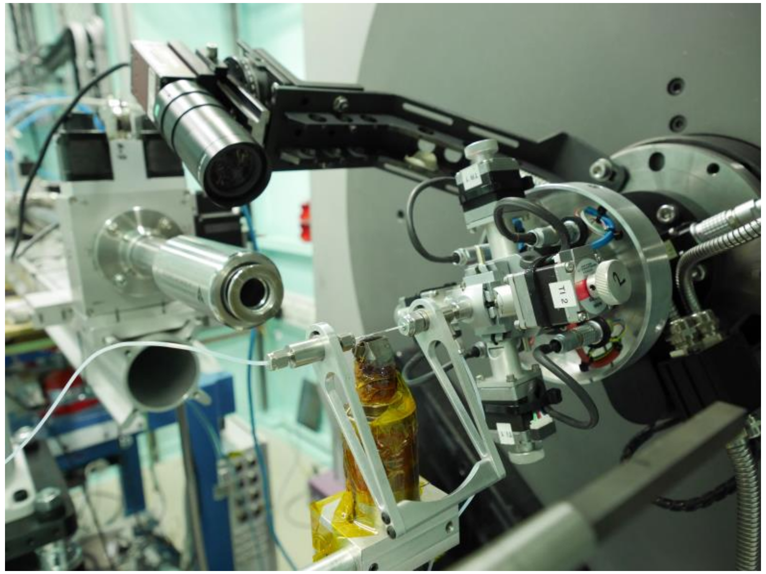


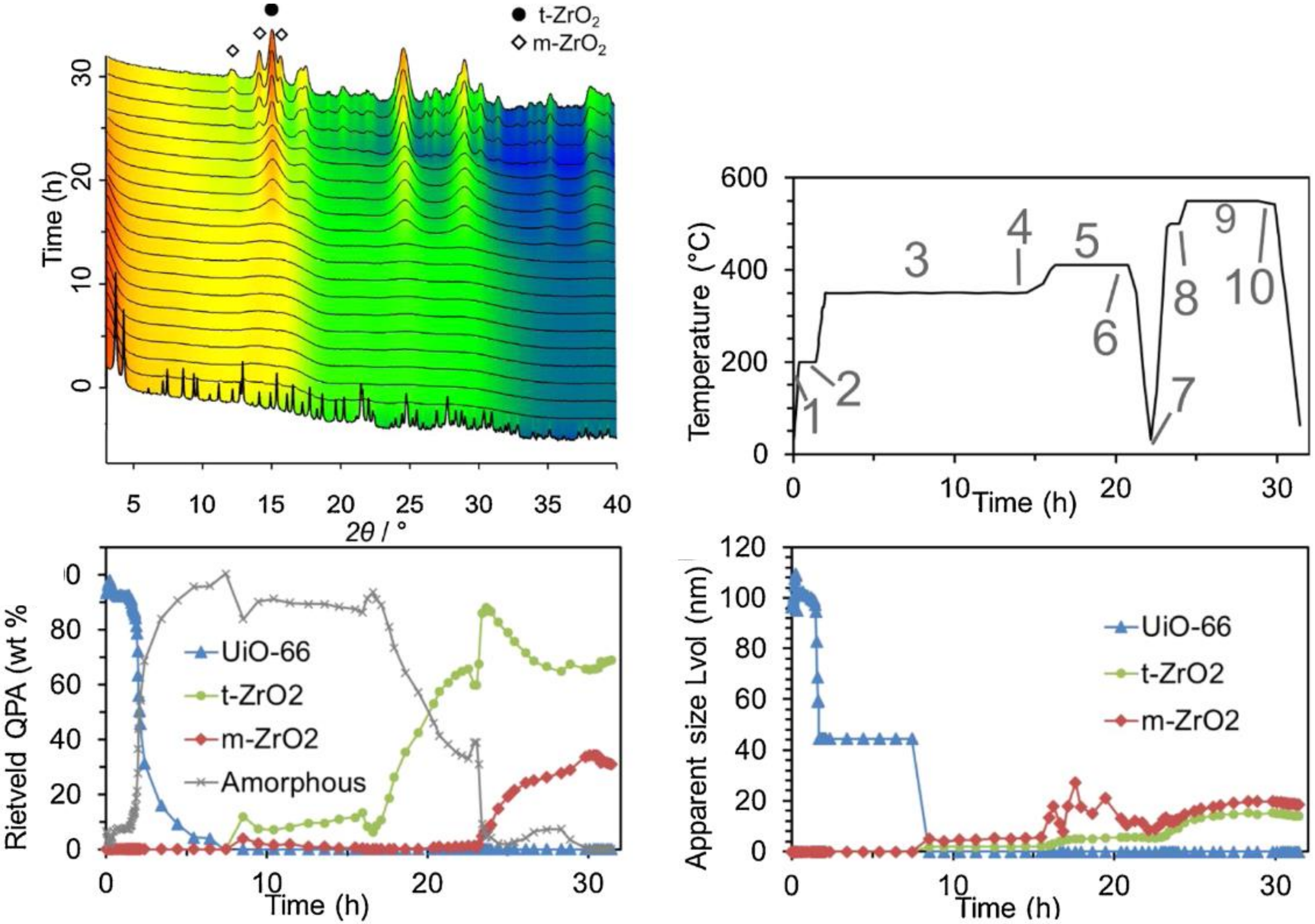


Humphries, T. D, *et al.*, *Journal of Materials Chemistry A* **2019,** *7* (3), 1206-1215.

CO_2 methanation

MOF-derived
Ru^0/ZrO_2 catalysts



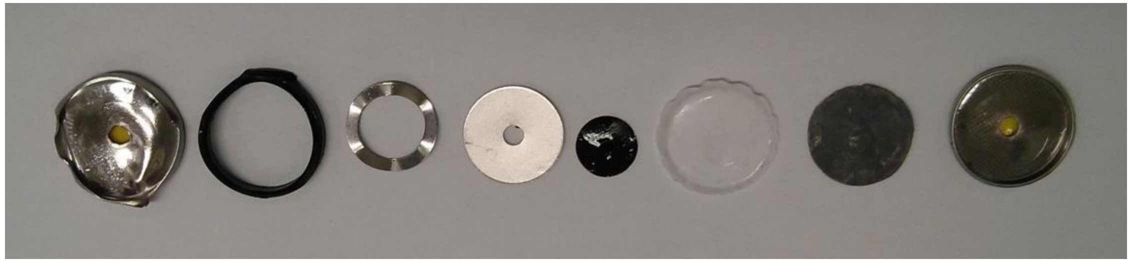


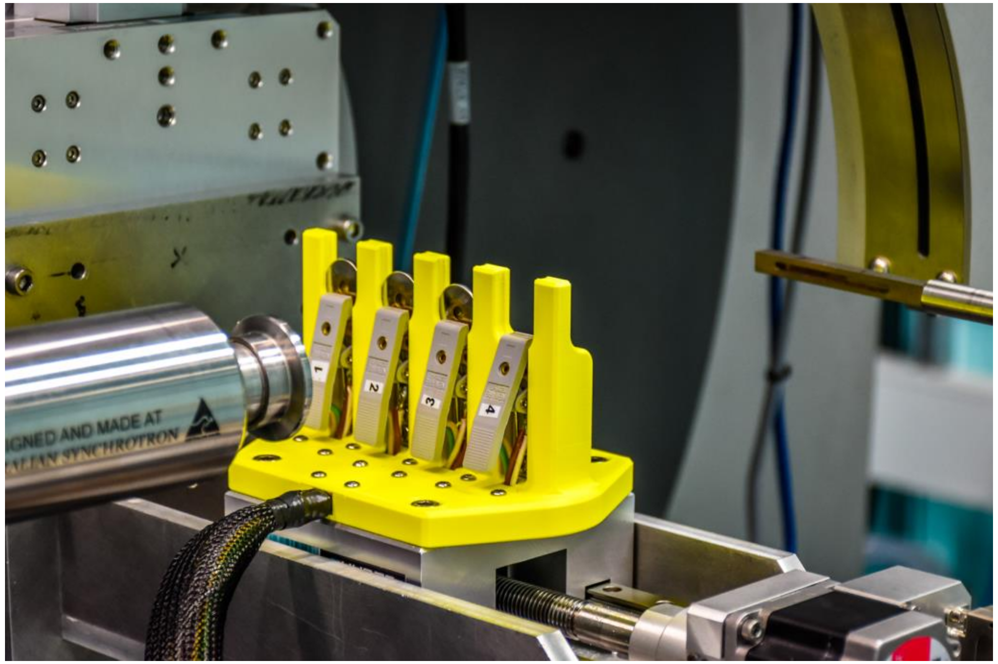
R. Lippi, A. M. D'Angelo, C. Li et al Catalysis Today 368 (2021) 66–77

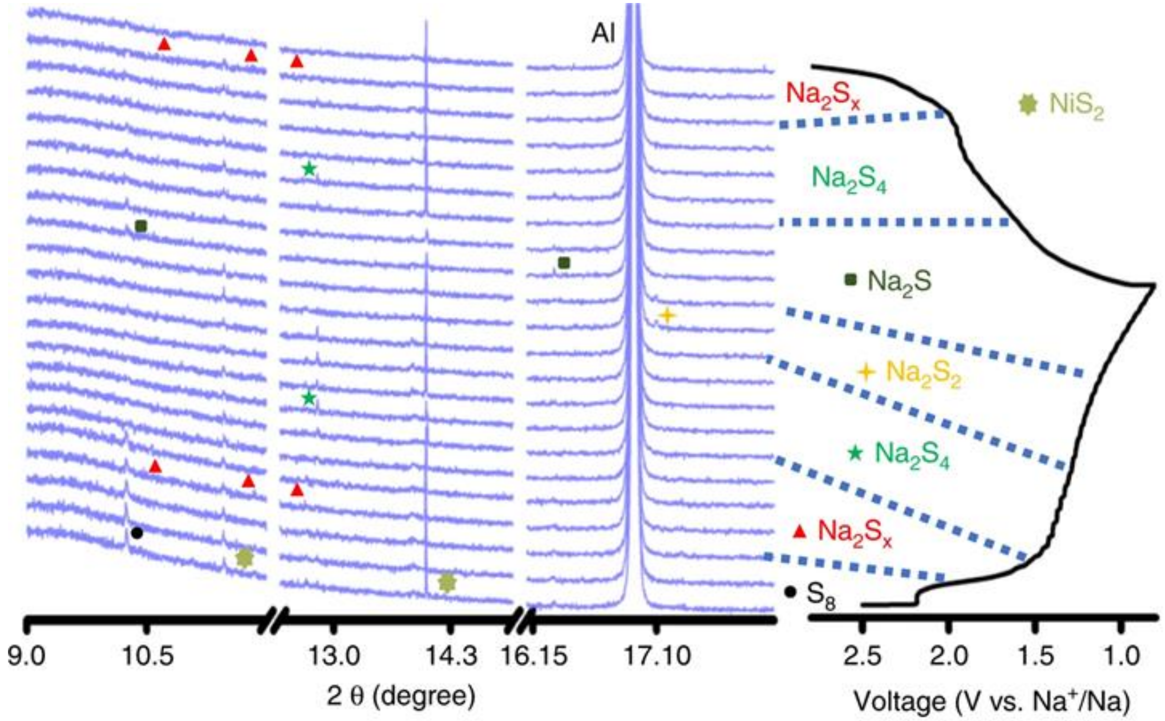
ANSTO

Batteries

Sodium-sulfur batteries

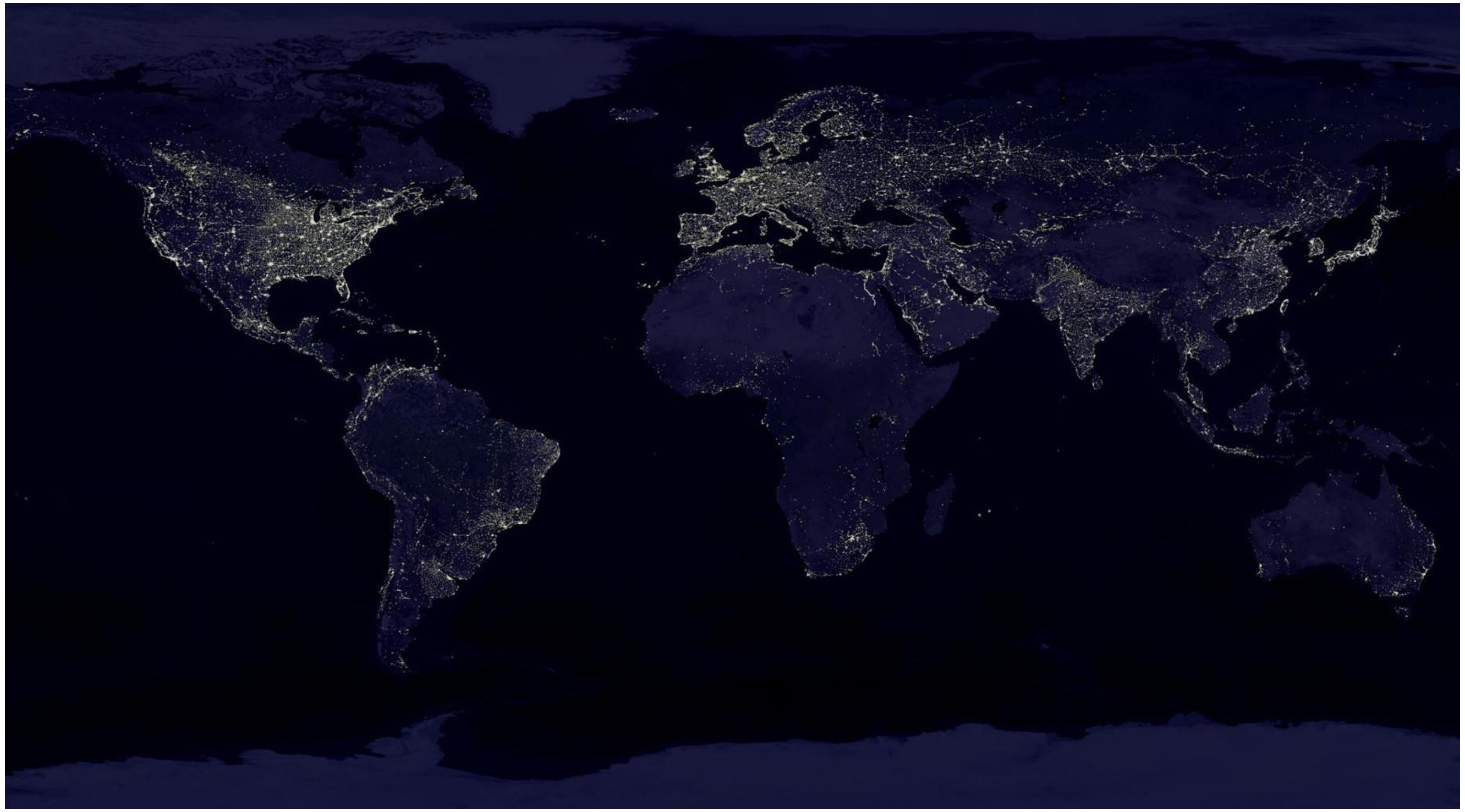


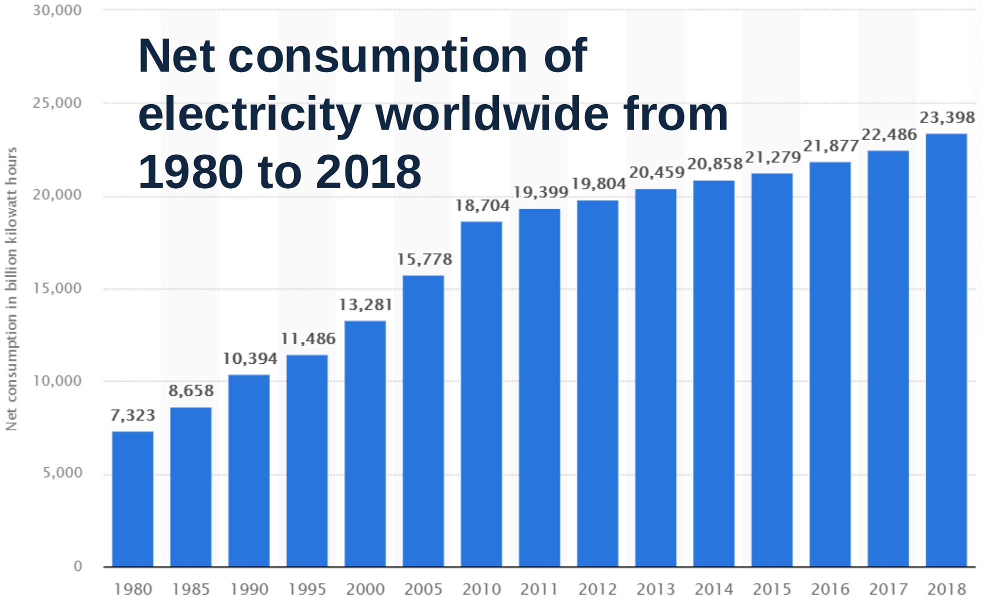


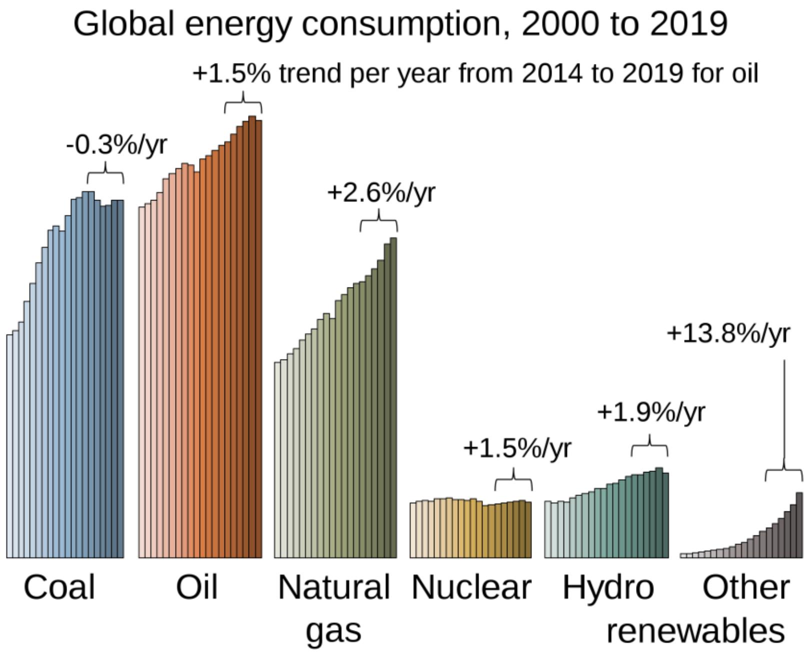


Z. Yan, J. Xiao, W. Lai et al Nature Communications 10 (2019) 4793

❖ **The limited resources that are used to power our society are diminishing as the demand rises.**







Environmental Research Letters(14), 2019.

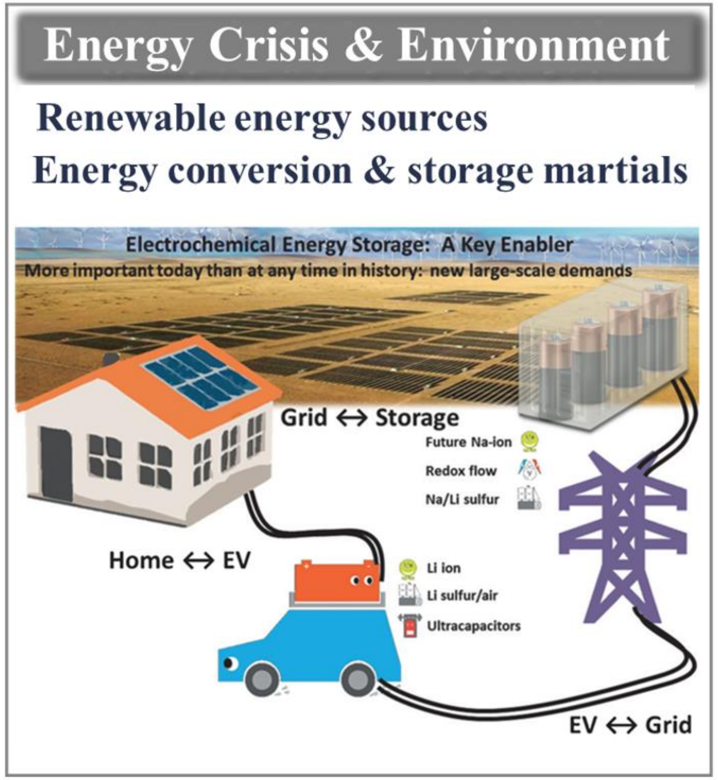
We live in the real world:

- ✓ Carbon neutrality and renewable energy
- ✓ Efficient energy storage solutions
- ✓ Portable tech and electric vehicles (EVs)
- ➢ User community growth of ~ 500% over past 5 years of beamline operations

G20 countries' 2030 emissions targets

NDCs with enhanced 2030 emissions reduction targets compared to previous NDCs · Have not yet submitted new or updated NDCs · NDCs that are the same or weaker compared to previous NDCs

G20 Members	Updated 2030 reduction targets	Net-zero targets in law, policy, or pledge
Argentina	Not exceeding 359 metric tons of carbon dioxide by 2030	Net zero by 2050
Canada	40-45% below 2005 levels by 2030	Net zero by 2050
EU (incl. Italy, France and Germany)	55% below 1990 levels by 2030	Net zero by 2050
Japan	46-50% below 2013 levels by 2030	Net zero by 2050
Russia	70% of 1990 levels by 2030	No target
South Africa	Not exceeding 350-420 metric tons of carbon dioxide by 2030	No target
UK	68% below 1990 levels by 2030	Net zero by 2050
US	50-52% below 2005 levels by 2030	Net zero by 2050
Brazil	43% below 2005 levels by 2030	Net zero by 2050
Mexico	Reduce its emissions by 22% below a business-as-usual scenario by 2030, but a conditional 36% target below BAU is on the cards	No target
Australia	26-28% below 2005 level by 2030	Net zero by 2050
Indonesia	29% reduction and conditional reduction up to 41% by 2030	No target
South Korea	30% below 2017 level by 2030	Net zero by 2050
China	Previous target: 60-65% carbon intensity reduction below 2005 level by 2030	Net zero by 2060
India	Previous target: 33-35% emissions intensity reduction below 2005 by 2030	No target
Saudi Arabia	Previous target: Actions only	Net zero by 2060 (excluding exports)
Turkey	Previous target: 21% by 2030	Net zero by 2053



Angew. Chem., Int. Ed., 2015, 54(11)
Adv. Energy Mater., 2015, 5(19)

ANSTO

✓ Hydrogen storage solution

McPhy station hydrogène SMT AG Lens Bethune Hauts de France

McPhy energy

TOSHIBA
Leading Innovation >>>

Solar-Powered Hydrogen Station Can Produce Enough Hydrogen to Fill 560 Fuel Cell Vehicles a Day

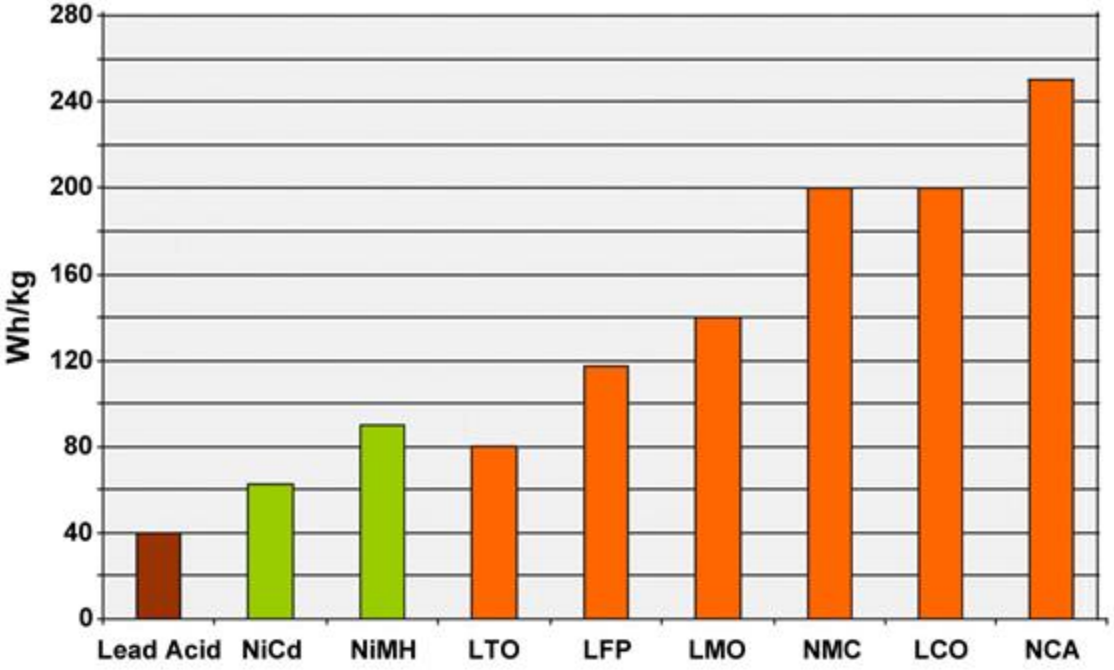
Kazuhiro Nogita as a co-funder of the company

hydrexia

ANSTO

Lithium is the basis for batteries we use every day (LIBs)

- LIBs energy density increased **300%**, and cost reduced **80%** over last 10 years.
- The most popular LIBs materials are Lithium Nickel Cobalt Aluminum Oxide ($LiNiCoAlO_2$) — **NCA** and Lithium Nickel Manganese Cobalt Oxide ($LiNiMnCoO_2$) — **NMC**
- High cost, limited safety, slow charge/discharge, limited cycle life



Sodium technology towards stationary applications (SIBs)

ANSTO

iPhone 13 (a **3227mAh battery @ 3.7V = 0.01194 kWh)**

Tesla model 3 (a **50-75 kWh battery)**

Li-ion

❖ **4000-5000 times more battery requirement**

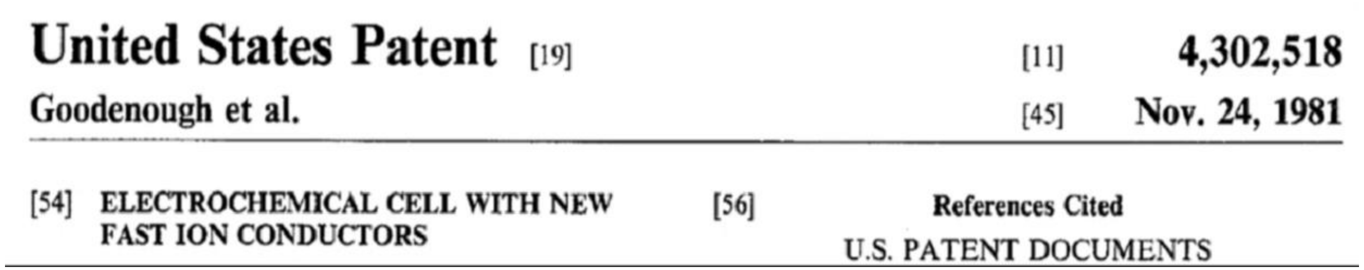
Powerwall Energy Capacity **13.5 kWh**

Size and Weight L x W x D

1150 mm x 753 mm x 147 mm

114 kg

ANSTO



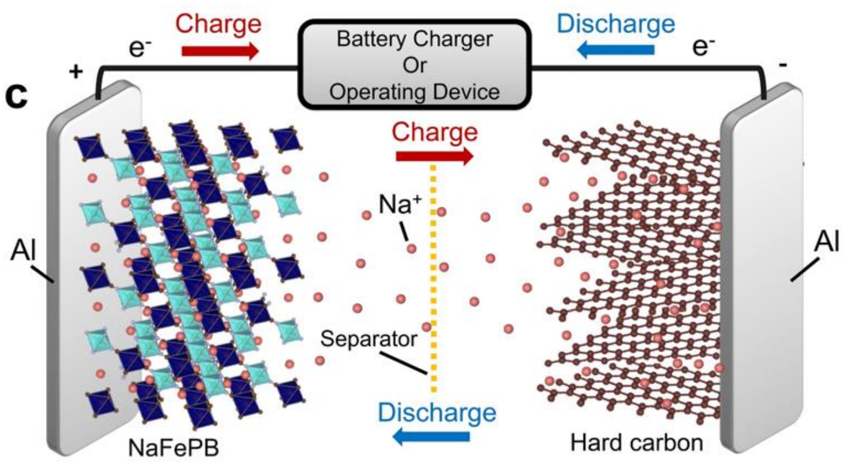
United States Patent [19] | [11] 4,302,518

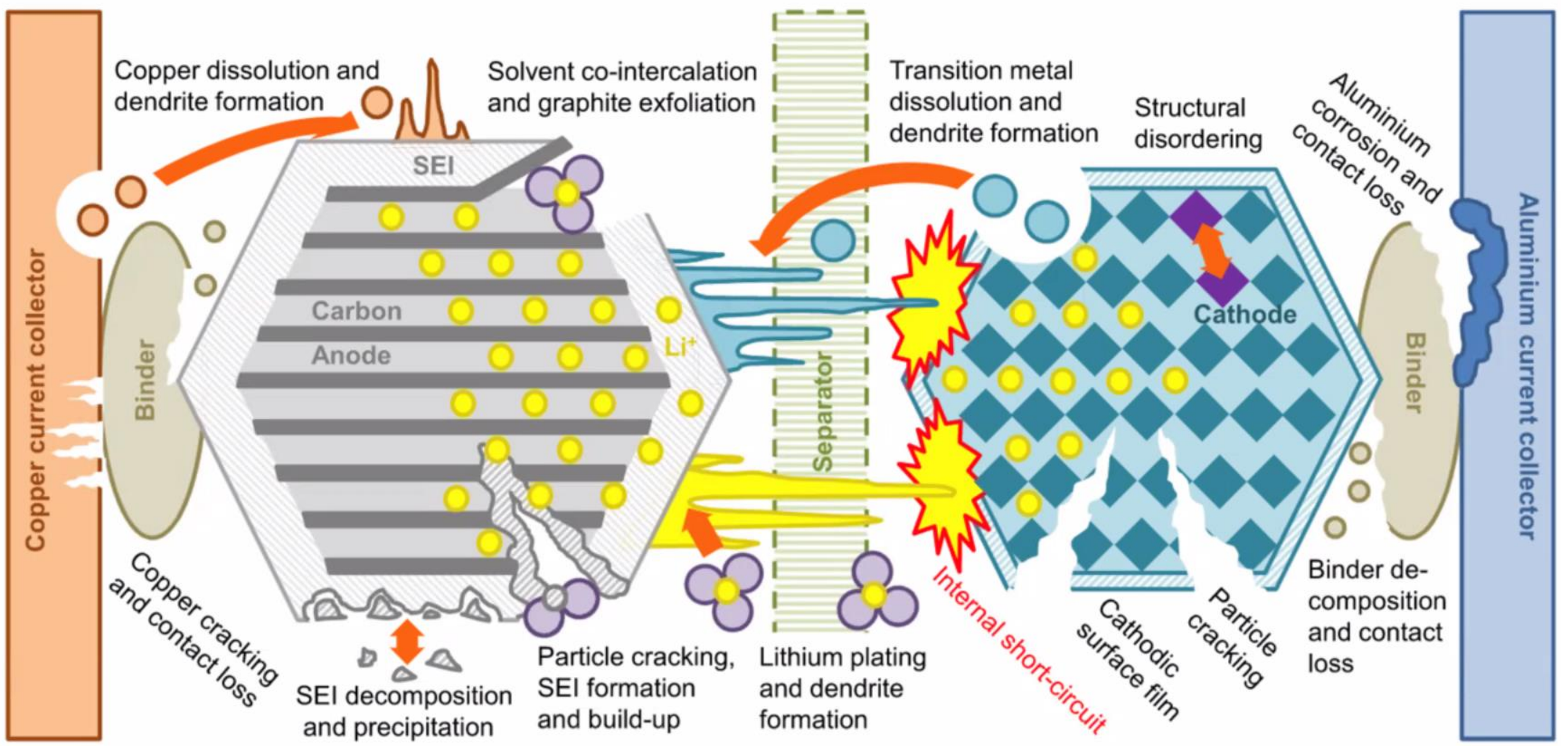
Goodenough et al. | [45] Nov. 24, 1981

[54] ELECTROCHEMICAL CELL WITH NEW FAST ION CONDUCTORS

[56] References Cited

U.S. PATENT DOCUMENTS





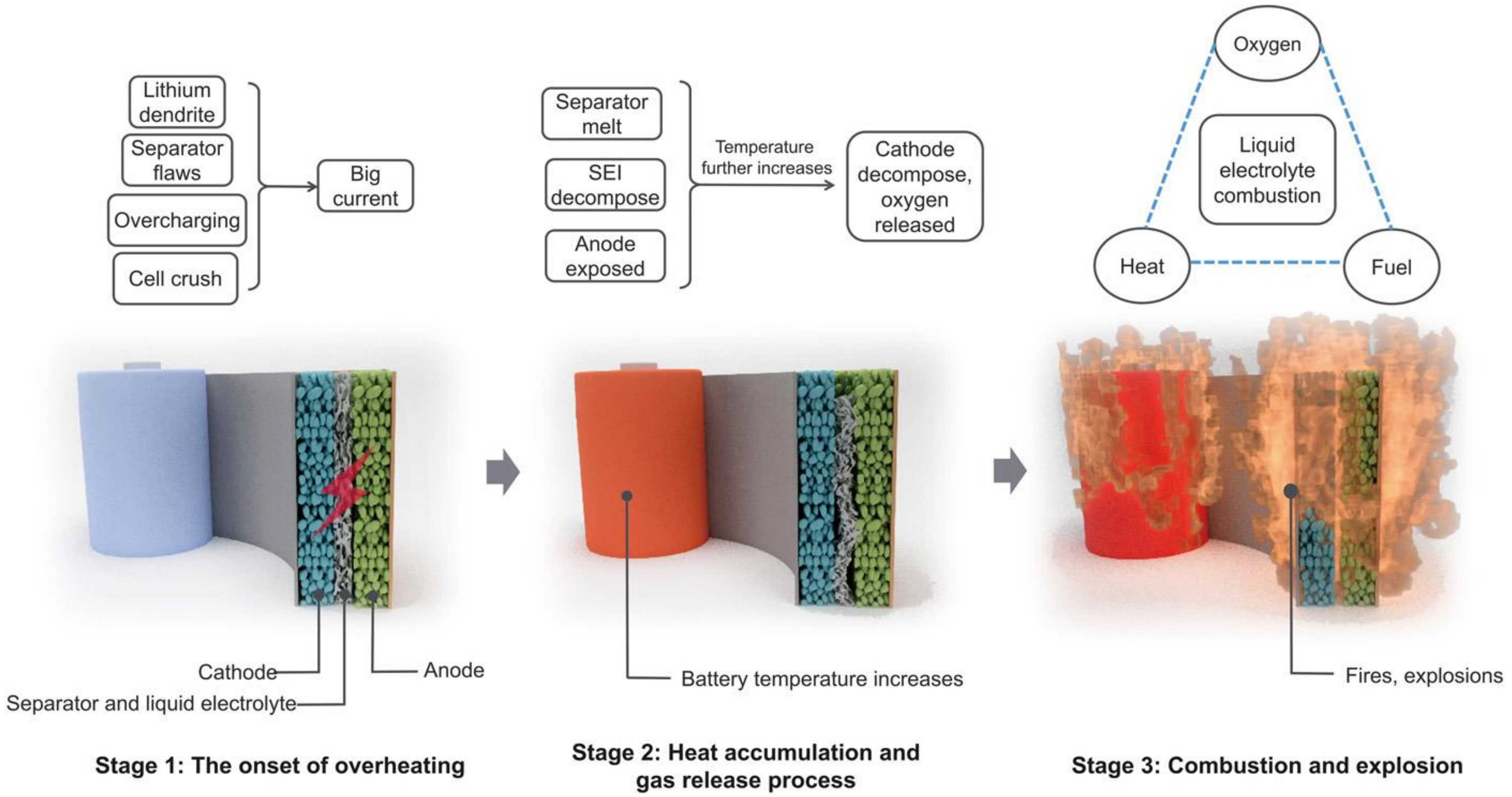
Research Approach to Multi-faceted System

1. **Multi-scale**
   (cm to nm)
2. **Multi-modality**
   (electrochemistry, structures, lattice, bonds, transient states)
3. **Multi Contrast**
   (soft to hard materials)
4. **Non destructive**
   (3D, In situ, In operando)

Birkl et al., Journal of Power Sources 341, 373-386 (2017)

ANSTO

- ✓ **Thermal runaway process is the key problem for Li ion batteries.**
  - Mechanical, thermal, and electrical
- ✓ **Many different failure pathways that lead to thermal runaway and consequent fires/explosions.**



Stage 1: The onset of overheating

Stage 2: Heat accumulation and gas release process

Stage 3: Combustion and explosion

Tesla fire that damaged two Megapacks in Victoria (Aug 2021)

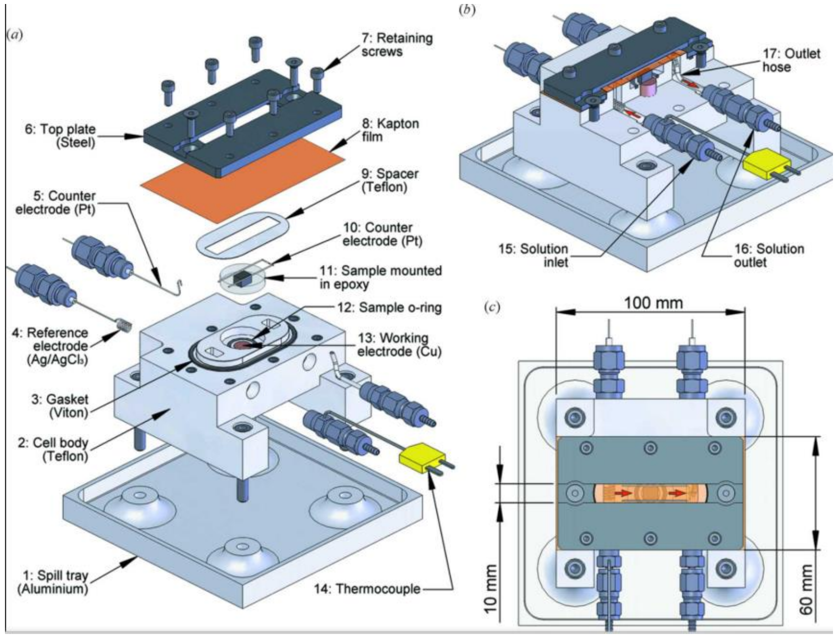
❖ **Three stages for the thermal runaway process.**

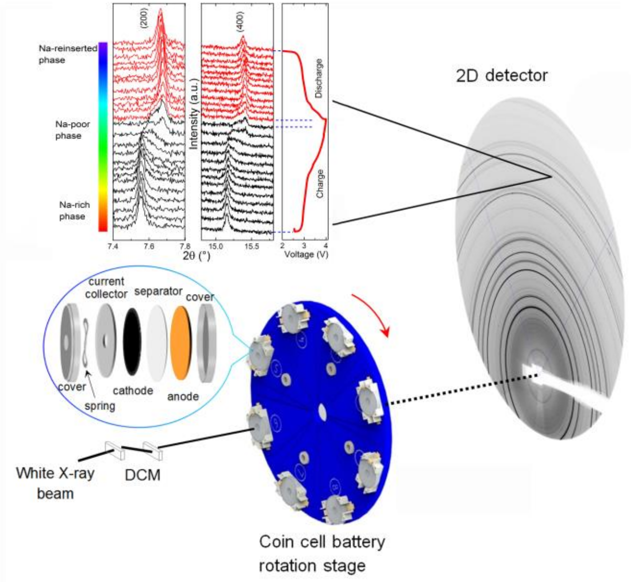
SCIENCE ADVANCES Vol 4, Issue 6 (2018)

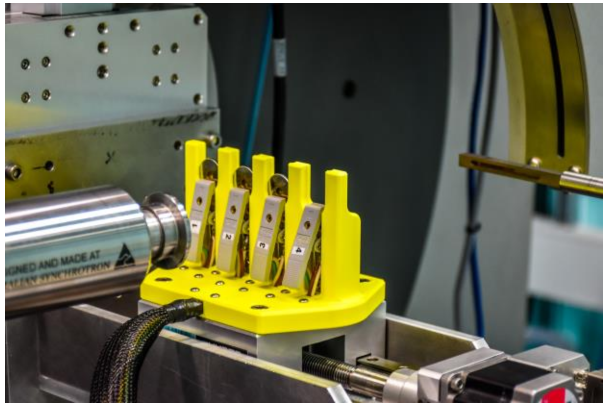
TOOL CHEST	XAS	MEX (new in 2022)	PD	ADS (ca. 2024)
Capability	✓ Valence states ✓ Atomic structure		✓ Crystalline structure ✓ Phase composition	✓ Add imaging
Energy range	5 – 31 keV	1.8 – 14 keV	5.5 – 22 keV	50 – 150 keV
Resolution	0.2 eV	< 0.1 eV (HERFD)	Monochromatic	Monochromatic, pink, and white
Beam size	0.25 x 5 mm	2 x 10 mm, or µm	0.8 x 1 mm to 1 x 5 mm	10x50µm - 15x15mm
Experimental stations	Both beamlines have two stations; incl standard XAS station suited for batteries		Two stations; incl high throughput station for batteries	Three stations; incl station suited to fast battery work (msec)
Detection limit	< 100 ppm (XANES)		0.2 wt.%	
Scan speed	20 min (XANES) 3 min (slew scan, 2022)		Few seconds	
Automation	Batch scanning ex-situ samples		Robot automation for capillary samples, batch scanning coin cells	

ANSTO

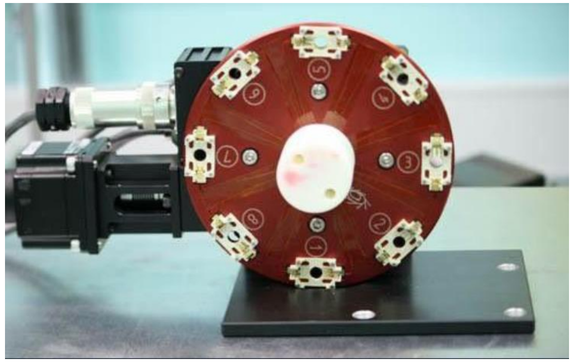
Powder Diffraction (PD)

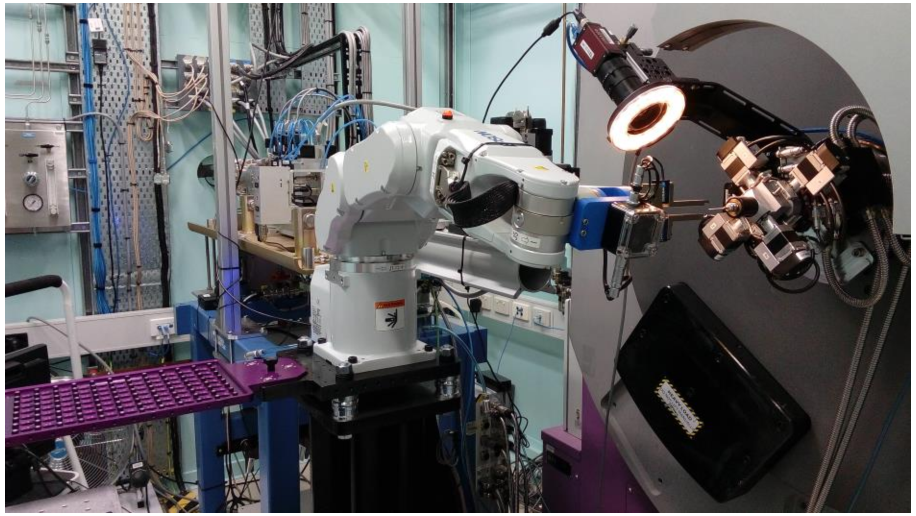






✓ Multiple *in-situ* battery sample holders





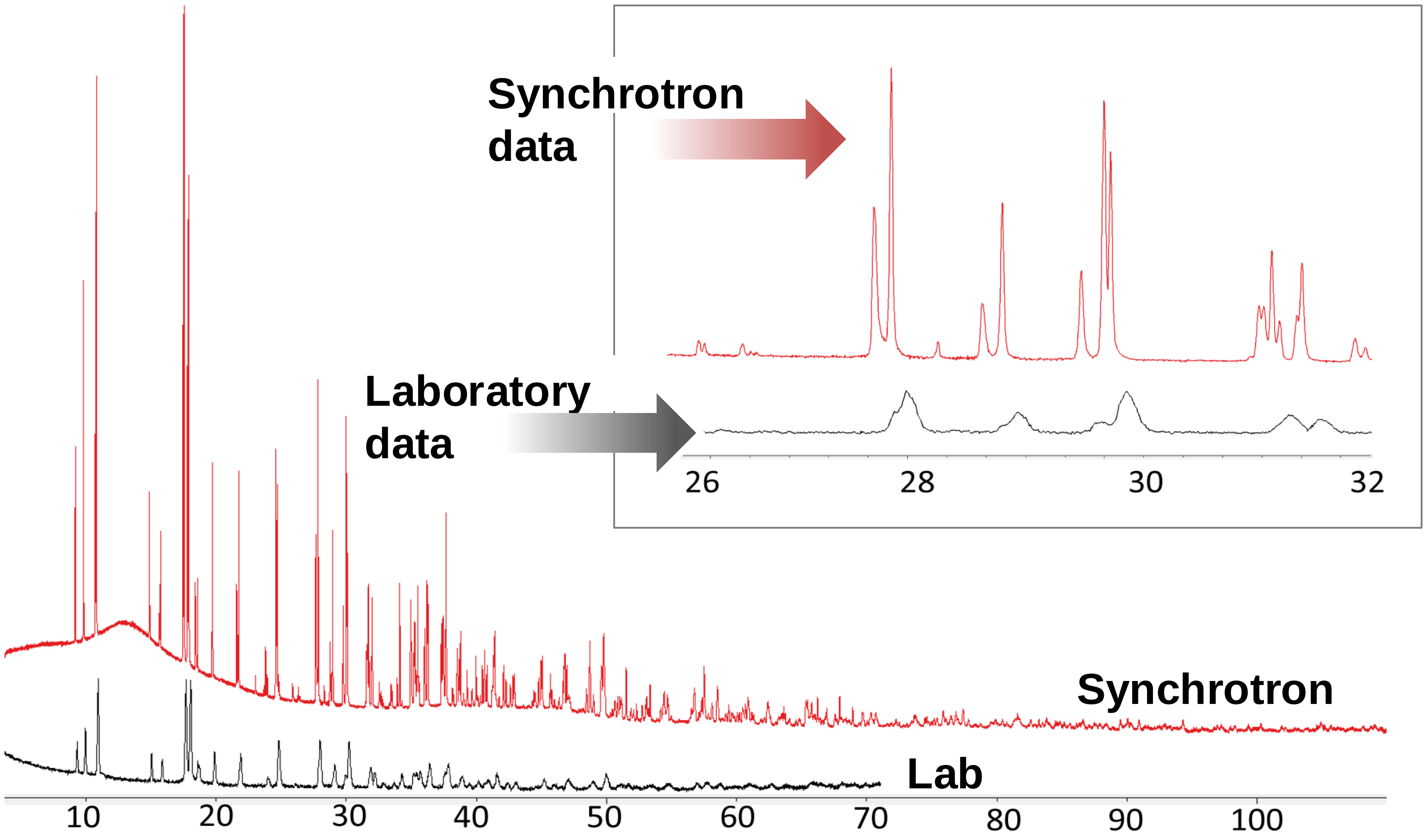
✓ Automatic *ex-situ* robotic sample change over

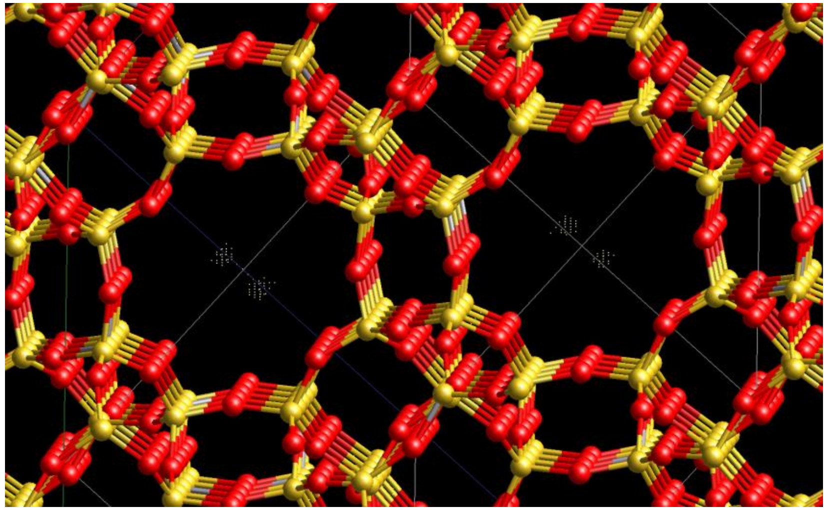
Use PD to determine:

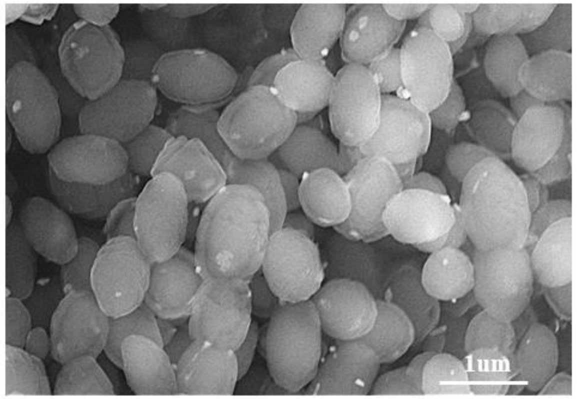
- ➢ **Crystalline structure**
- ➢ **Phase composition**

✓ **Determine long and short range order of structures in solid state materials**

ANSTO







- ✓ Synchrotron PD provides the most sensitive detection of crystalline phases
- ✓ High throughput and fast measurement time allows more specimens
- ✓ Synchrotron PD provides a gold standard for this well established technique

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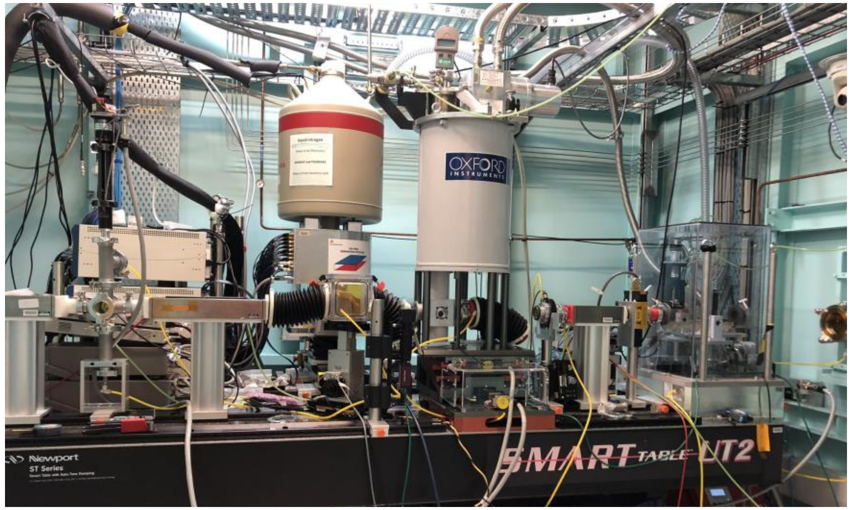
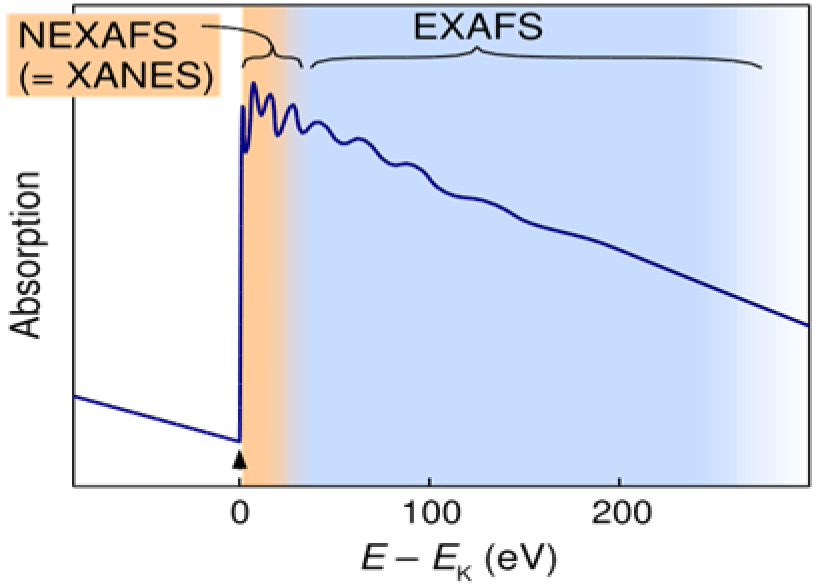
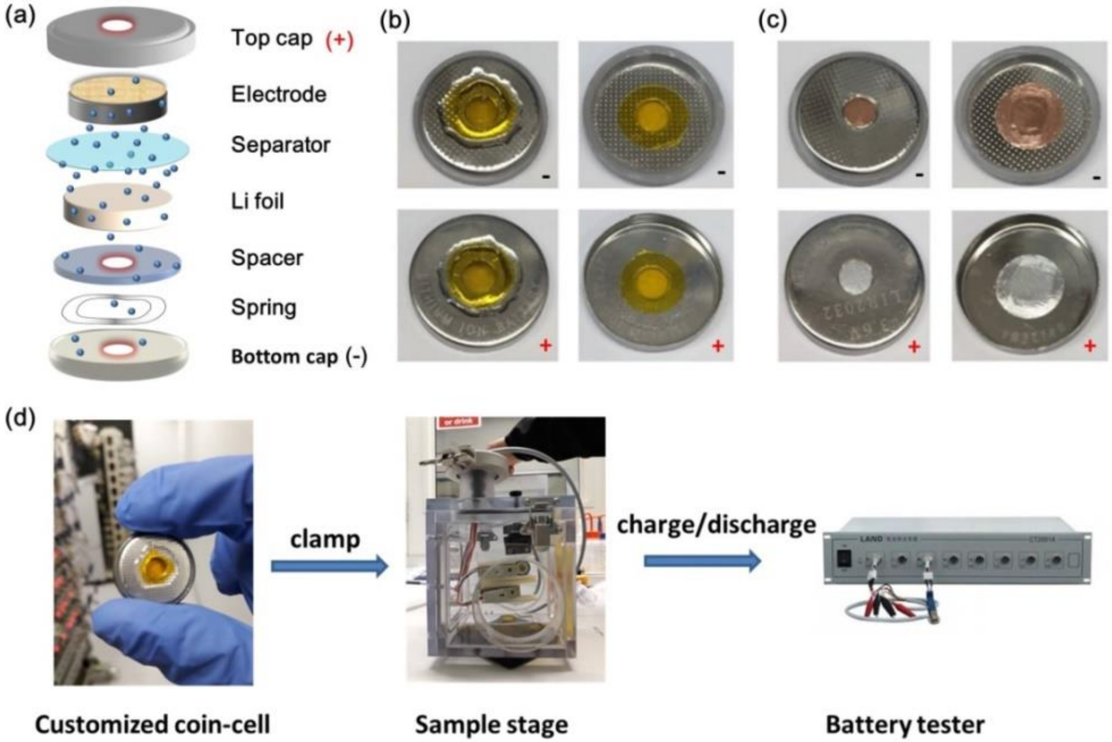
X-ray Absorption Spectroscopy (XAS)



- ✓ Efficient *ex-situ* loading and batch scanning
- ✓ *In-situ* battery sample holder available





Use XAS to determine:

- ➢ **Valence states via XANES**
- ➢ **Local atomic structure via EXAFS**

✓ **Independent of structure and size: nanoparticles, single atoms, and amorphous materials are fine.**

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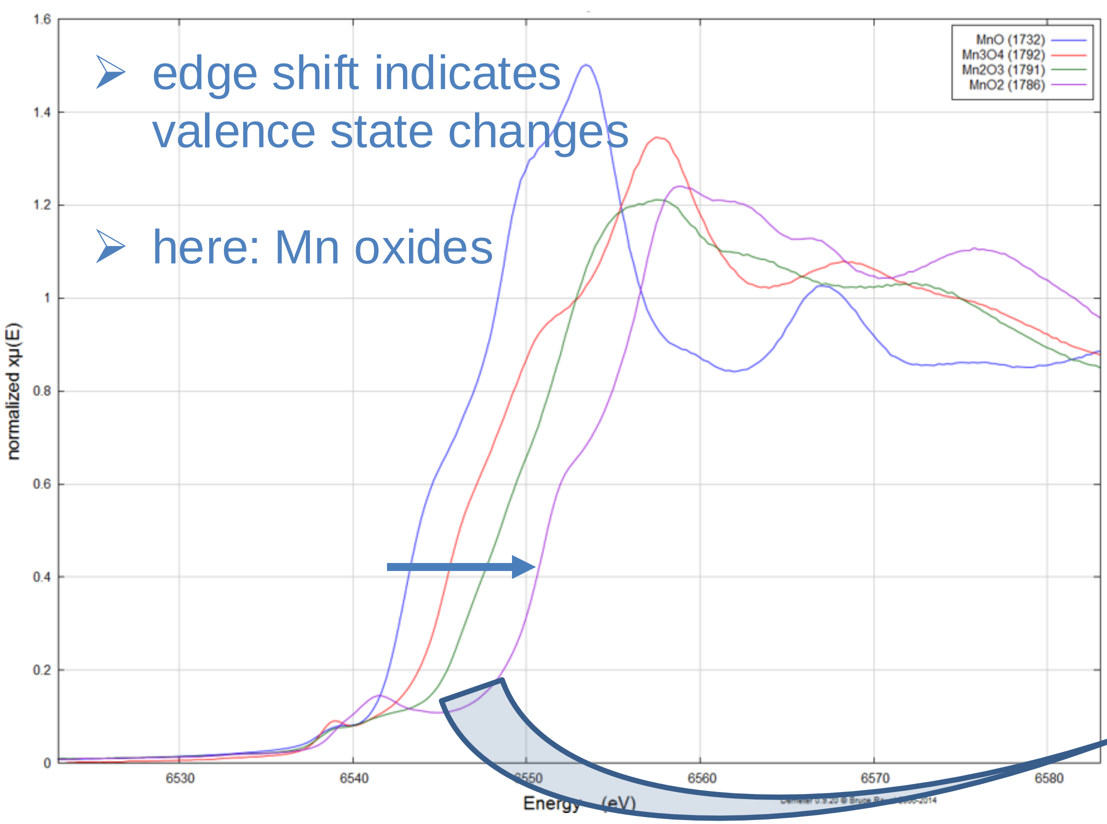
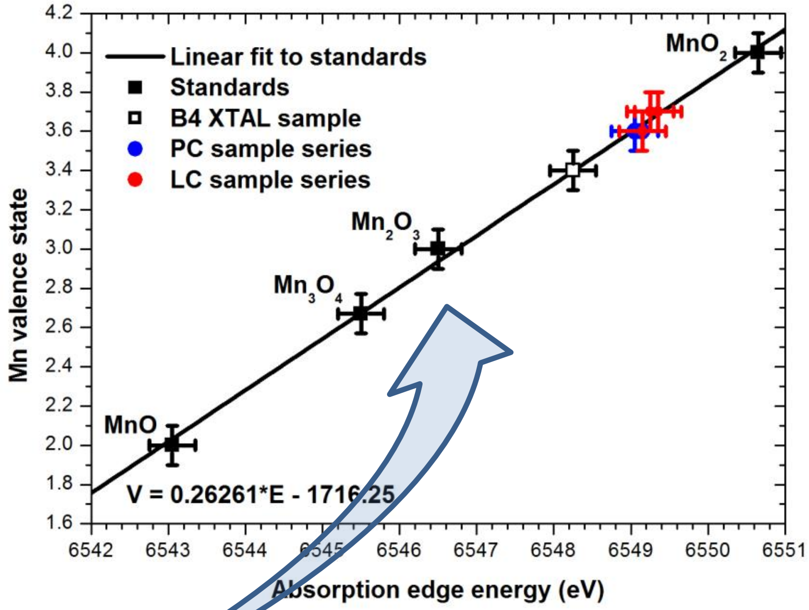
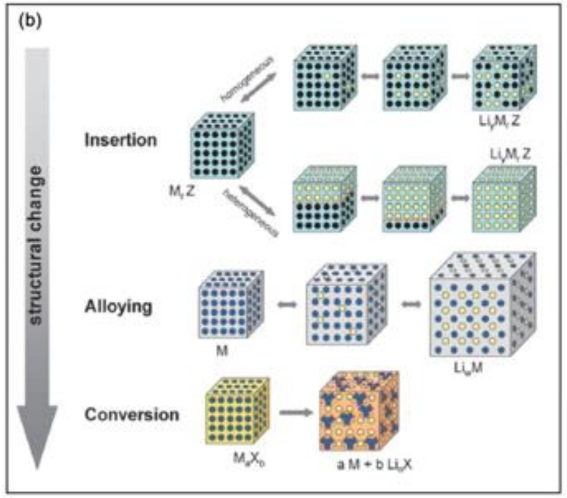
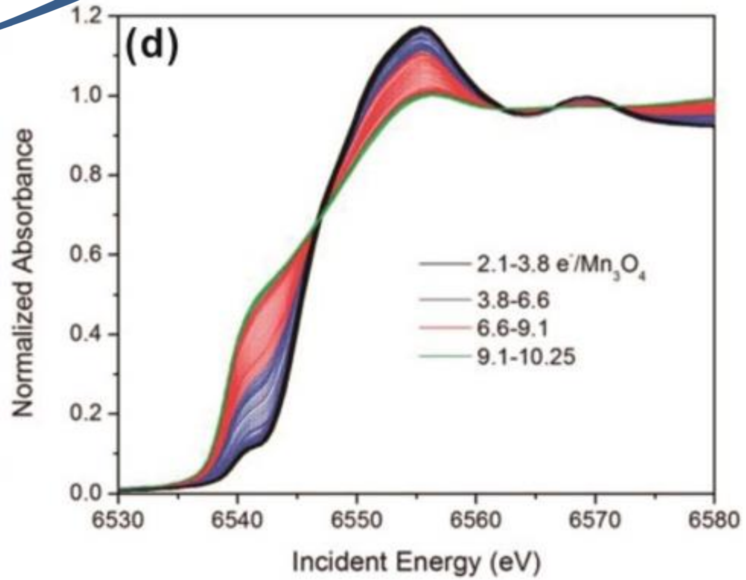
- edge shift indicates valence state changes
- here: Mn oxides





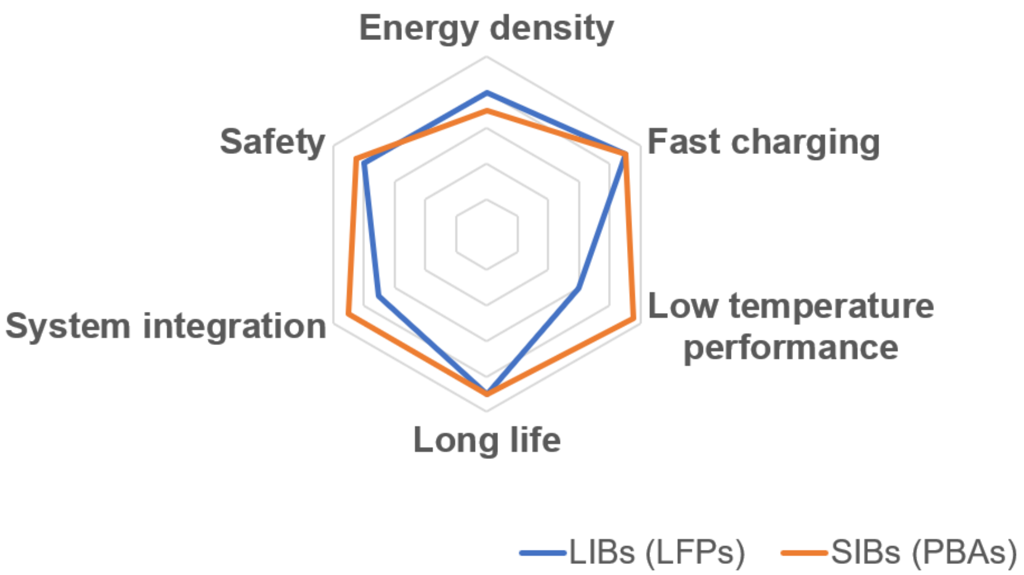




RHS: *Mn_3O_4 anode during (dis)charge*

Bat. Sup. 2021 (https://doi.org/10.1002/batt.202100006)

Battery radar chart



Li Vs Na		
Lithium	Sodium	Category
0.76	1.06	Cation radium (Å)
6.9	23	Atomic weight ($g.mol^{-1}$)
0	0.3 V	E^0 (vs. Li/Li^+)
5000	150	Cost, carbonates (USD/ton)
3829	1165	Specific Capacity ($mAh.g^{-1}$)

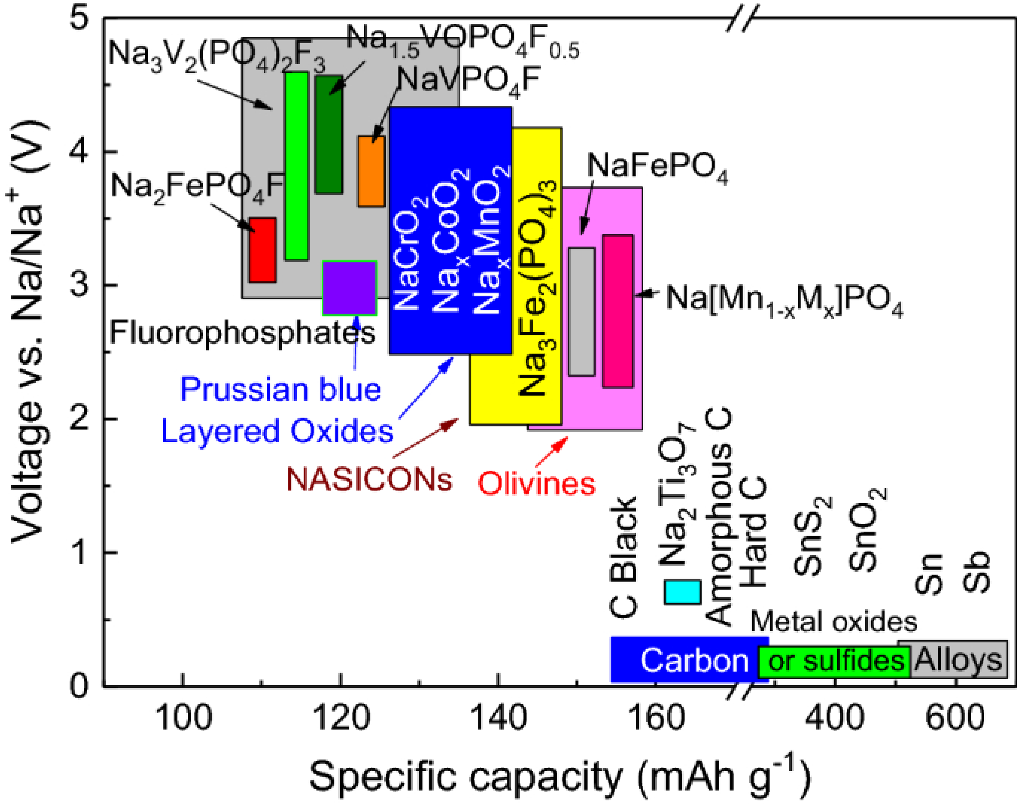
- ✓ SIBs and LIBs show similar working principles, with different charge carriers: **Na+** vs **Li+**.
- ✓ Need develop suitable cathode, anode and electrolyte for SIBs.

Cathode	Not limited to expensive metals, e.g. Co, Ni, Mn	**R&D for new electrode materials**
Anode	Can not use graphite (Na > Li ion)	
Separator	Can use the existing separator systems	**Adjust the existing system**
Electrolyte	Similar as LIBs (sodium based electrolytes)	

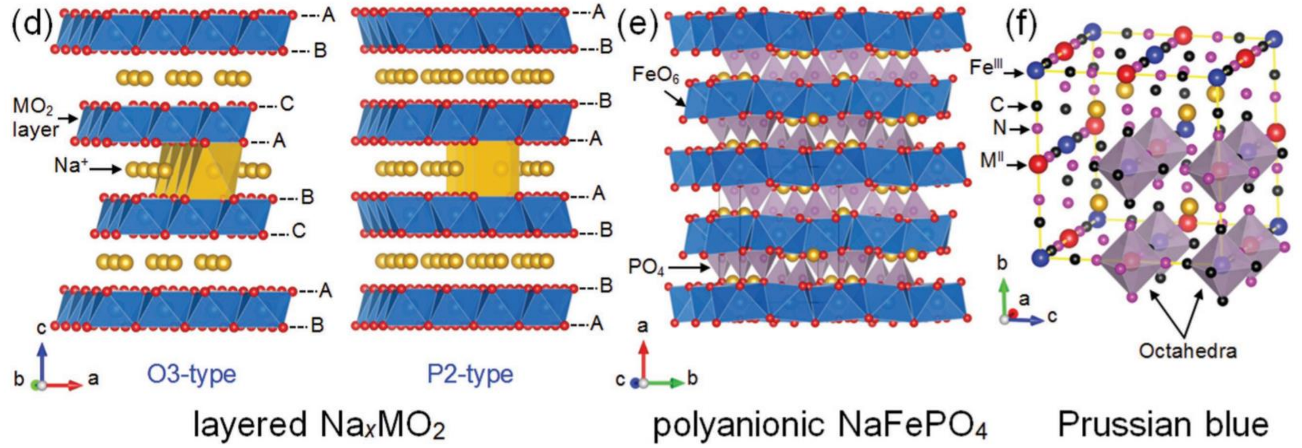
ANSTO

Sodium ion batteries cathodes

Adv. Energy Mater. **2017**, 1602831



Electrochemical Energy Reviews volume 1, pages 200–237 (2018)



❖ Different cathodes for different applications

	3D polyanion compounds	layered transition metal oxides	Prussian blue analogues
Examples	$Na_3V_2(PO_4)_3$ $Na_3V_2O_2(PO_4)_2F_{3-2x}$	O3-$NaNi_{0.5}Mn_{0.5}O_2$ P2-$Na_{2/3}Mn_{0.8}Fe_{0.2}O_2$	$Na_2FeFe(CN)_6$ $Na_xMnFe(CN)_6\cdot yH_2O$
Pros	✓ High voltage ✓ Good cycling	✓ Easy to synthesize ✓ Higher Specific capacity	✓ Good stability in air ✓ Easy to synthesize
Cons	✓ Higher molecular mass ✓ Lower Specific capacity	✓ Poor cycling ✓ Lower stability	✓ Lower energy density

Prussian Blue pigment (1706)

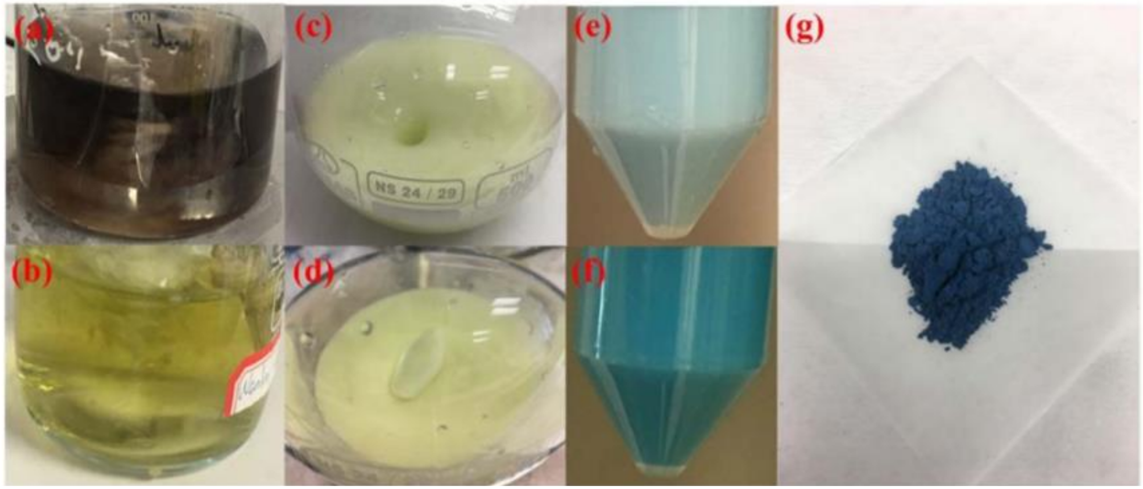
Landscape with Diogenes by Poussin (1647)

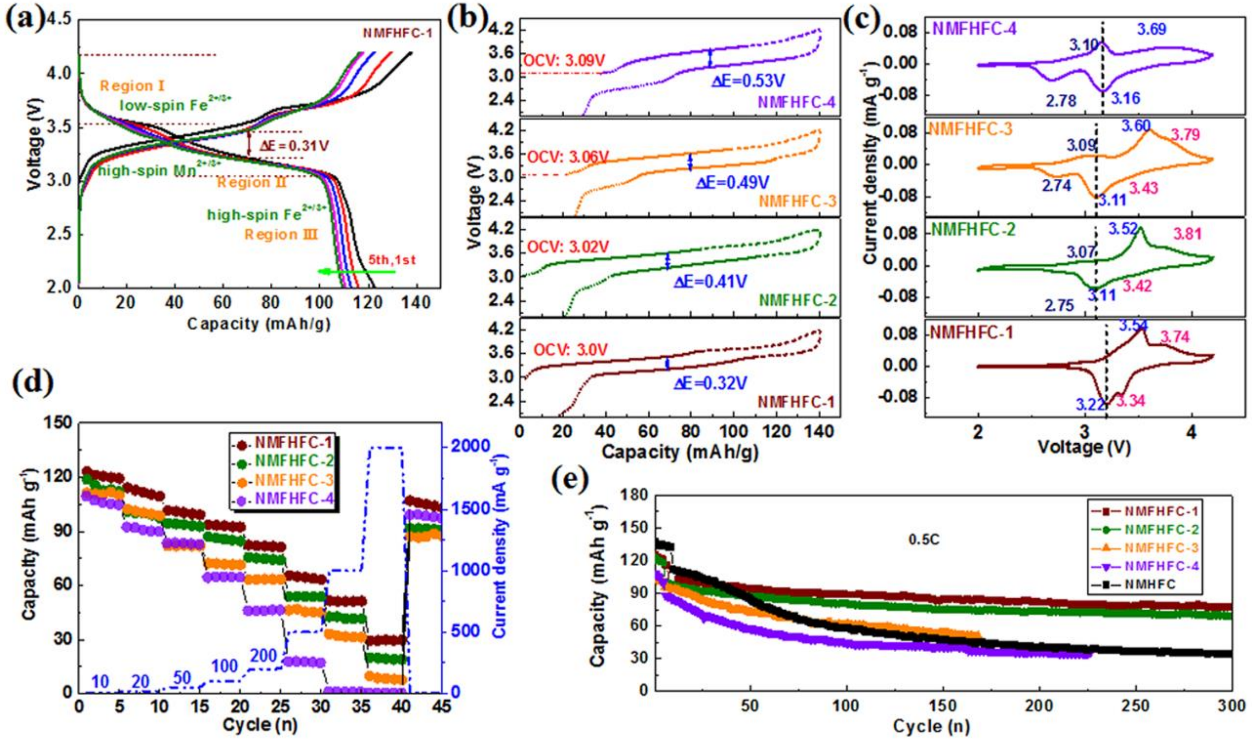
A cheap blue pigment was invented by accident in Berlin and subsequently traded across the world

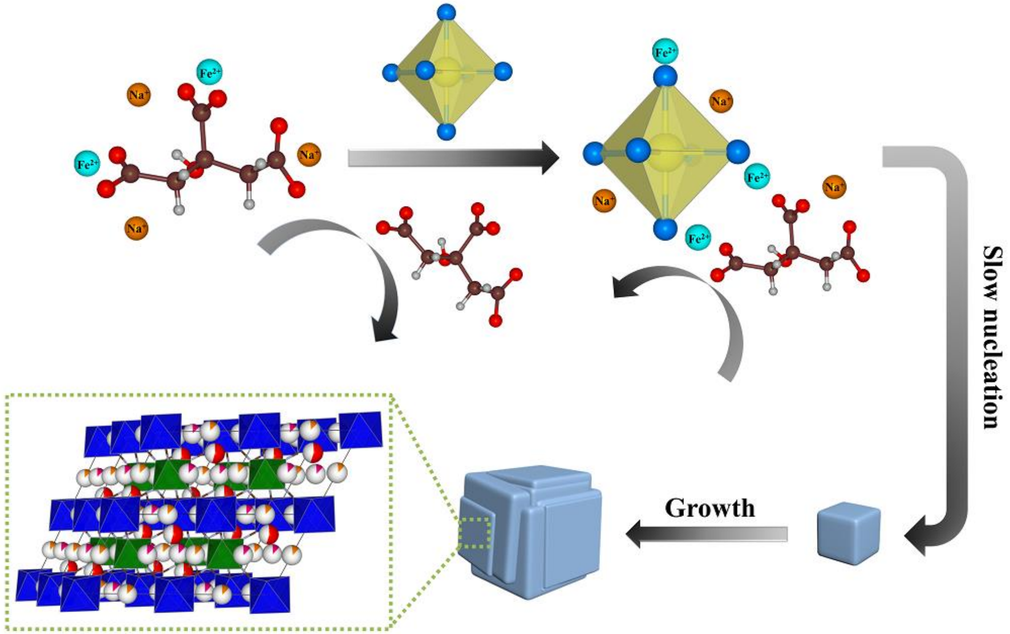
The Swing (1767) by Fragonard

Houses of Parliament (1899) by Claude Monet

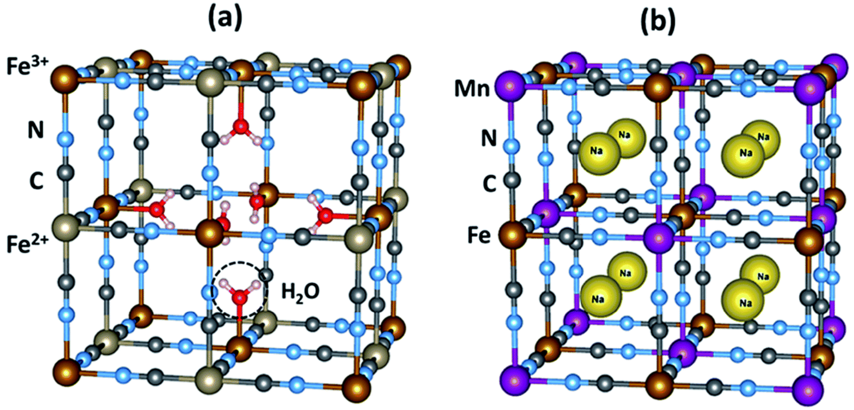
The Great Wave off Kanagawa by Hokusai (~1830)







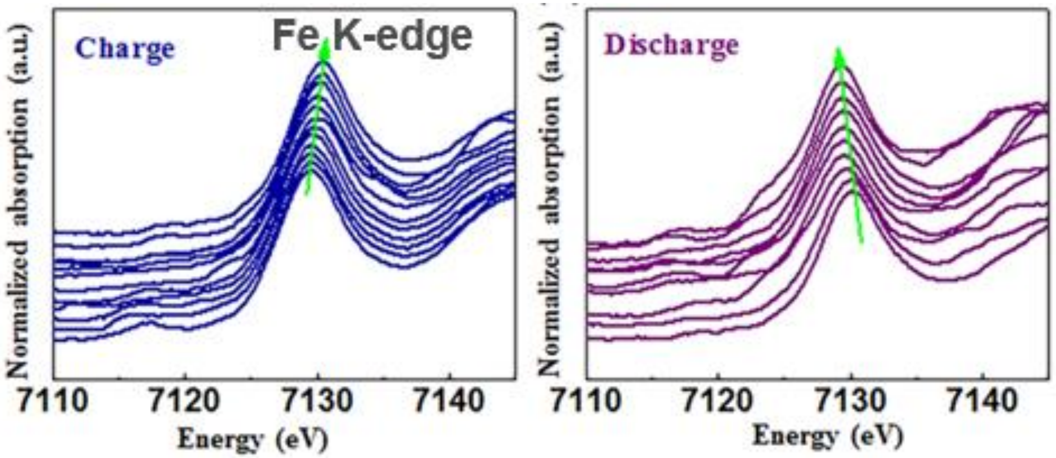
$$3K_4Fe(CN)_6 + 4FeCl_3 \rightarrow Fe_4[Fe(CN)_6]_3 + 12KCl$$

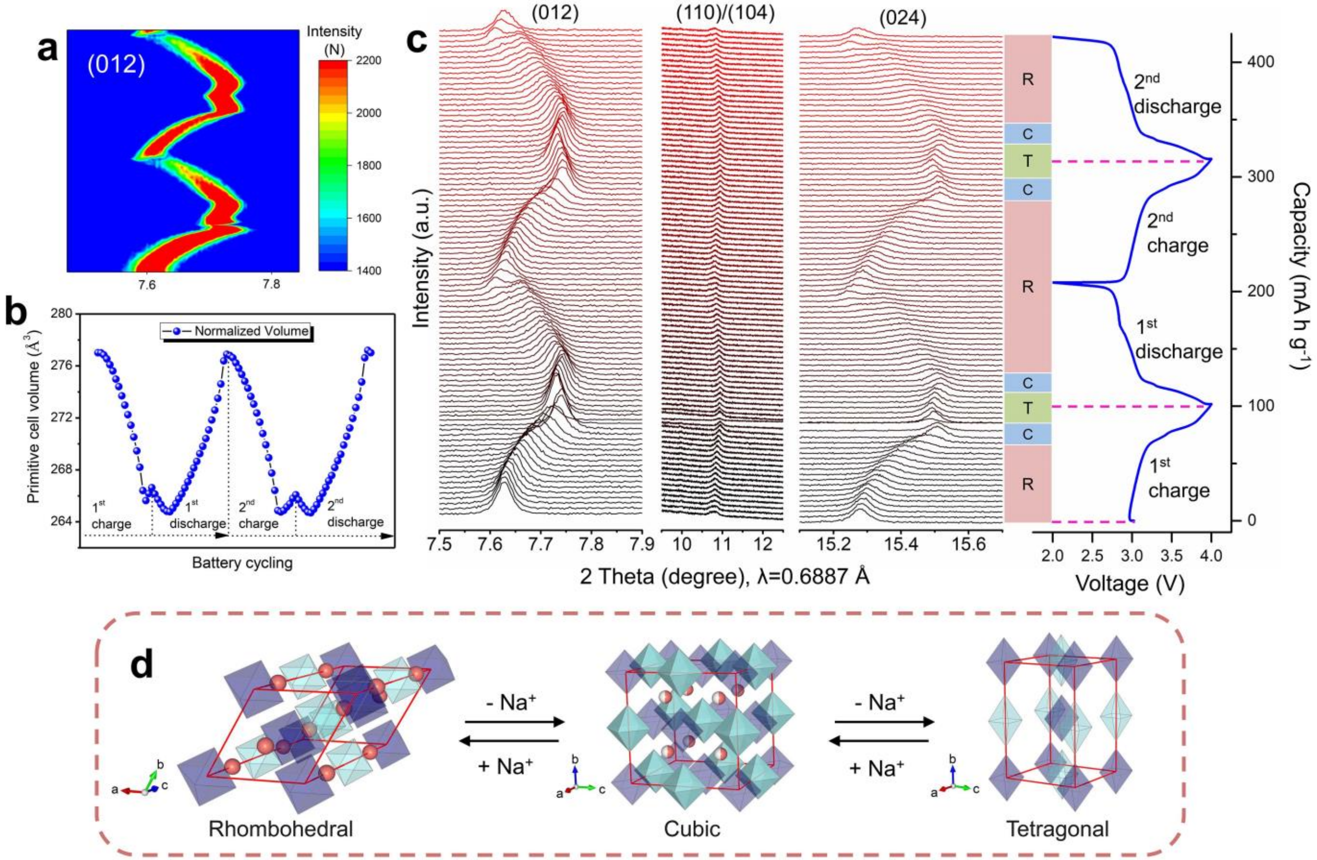


Prussian Blue Analogues (PBAs)

Prussian Blue has been studied since 1978 for its application in electrochemical energy storage and conversion. However, replacing some Fe metal centres with other metal ions (Mn, Co, Ni, etc) to form analogous materials have recently resulted in markedly more efficient materials. For example, Mn centred PBAs:

- Stabilizes the structure -> excellent cycle lifespan
- Eliminates dissolution of the active Mn^{2+} ions
- 80% capacity retention over 300 cycles
- High energy density
- Can be mass produced





Nature Communications, 11 (1), 980, (2020)
Adv. Energy Mater., 10, 1903006, (2020)
Energy Storage Materials, (2020)

Sodium ion batteries applications

❖ SIBs show overall advantage to Lead acid and Lithium manganate batteries.

	Lead acid	Lithium manganate Li_2MnO_4	Sodium ion battery PBAs	Lithium iron phosphate LiFePO4
Cost ($usd/kWh)	46-78	78-109	46-92	78-125
Energy density (Wh/kg)	30-40	130-150	100-150	130-170
Cycling	<400	500-1000	1000-5000	1000-5000
Low temp performance	bad	good	very good	bad
High temp performance	ok	bad	good	good
Rate performance	1C	3C	3-6C	3C
Environmental friendly	bad	good	good	good
Commercial scale	large	large	small	large

✓ **Replace Pb-acid and LFPs in some applications**

Energy storage	Low speed vehicle
Renewable energy grid	Golf cart, wheelchair
Domestic/industry grid	E-bike
5G, 6G mobile station	E-boat
Data centre	E-bus

❖ SIBs show lower energy density to LIBs.

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Prussian Blue pathways to success

Taking advantage of both PD and XAS beamlines:

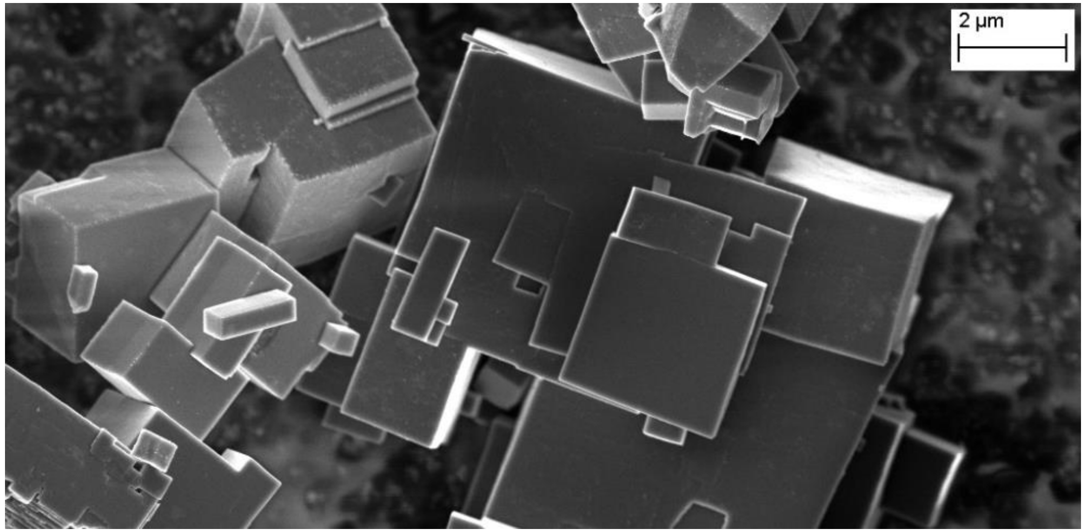
✓ **Shulei Chou**, (Wollongang University) now at Wenzhou University in China, collaborates with CATL who is a major supplier for energy applications worldwide.

➢ www.catl.com

✓ **William Brant**, now at Uppsala University in Sweden, is a co-founder of Altris AB who is producing Fennac® a commercial Prussian Blue derivative.

➢ www.altris.se

ALTRIS



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Access modes

Merit

- 3 rounds a year
- Peer-reviewed based on scientific merit
- Proposals are ranked
- Contact beamline before submitting

Mail-in

- Up to 20 room temperature capillaries OR up to 3 hours beamtime
- Peer reviewed as Merit proposal
- 2 energies
- Low hazard materials only

Commercial

- Confidential results
- IP owned by the client
- Fast and guaranteed access
- Standard service or full assistance

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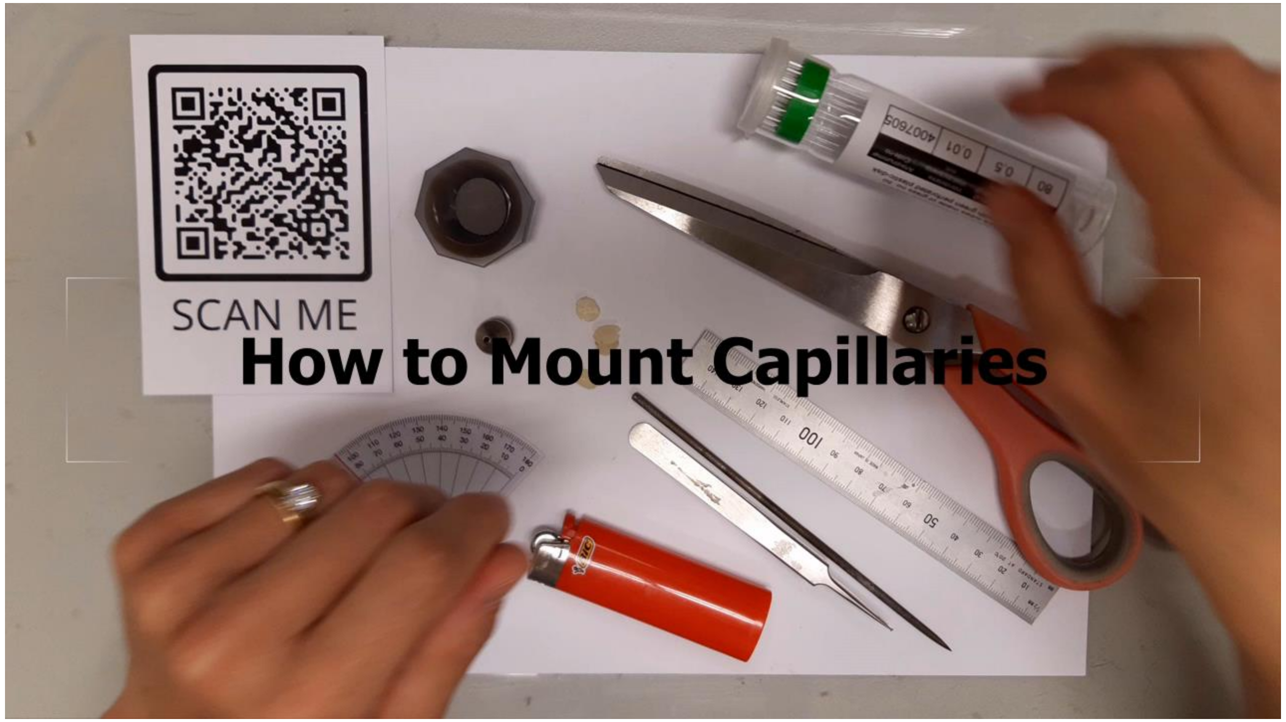
Mail – in



We send you this

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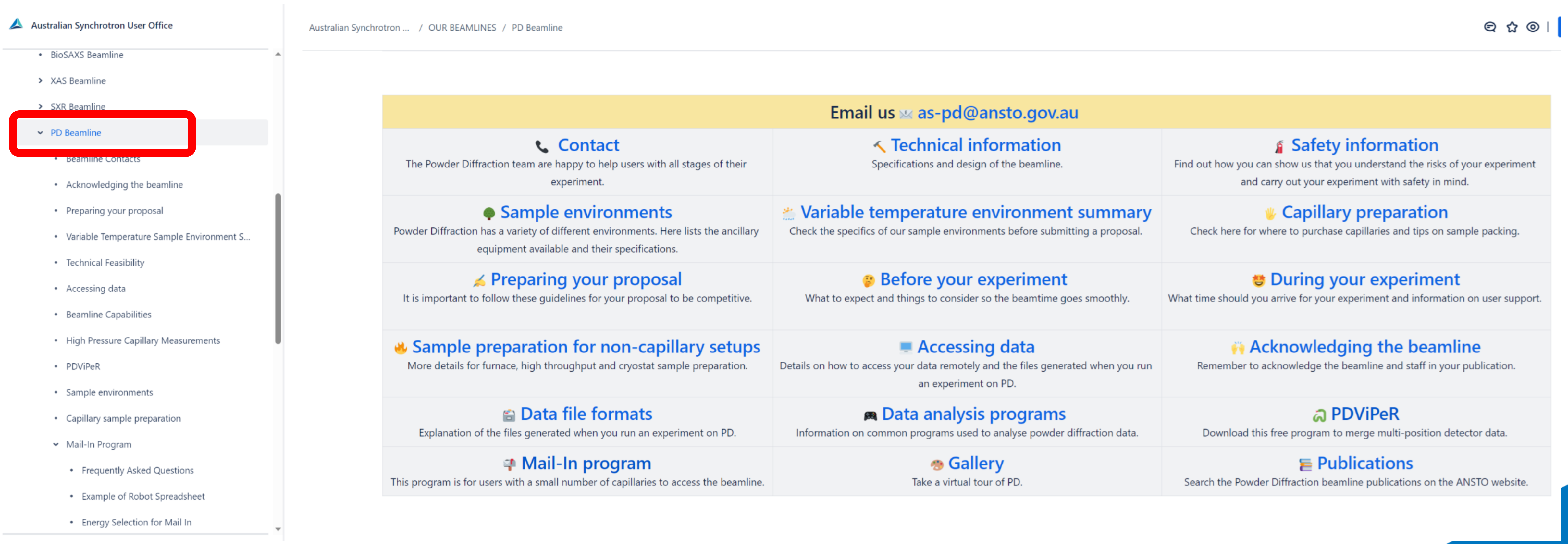
Beamline guides

https://asuserwiki.atlassian.net/wiki/spaces/UO/overview



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Things to think about

Science

What do I want to know?

Why is this science exciting?

Why is this science of national benefit?

Do I need a synchrotron, can I do this on a lab machine? Have I tried to do it on a lab machine?

Experimental

SAFETY

One setup per day

What experimental conditions do I actually need?

What elements are in my sample?

Ramp rates, temperature profiles, pressure? Gas? – **Include table!**

Setup, cooling and heating time

Procedures
Sample hazards/handling issues

Data analysis

What do I want to know?

How will I analyse the data? What software? Do I have expertise?

Do I want kinetics?

Structural changes?

Quantitative phase analysis?

Absorption?

What data collection strategy do I need for these?

Talk to your Beamline Scientist!

Principal Scientist
Qinfen Gu

Scientist
Anita D'Angelo

Scientist
Liam Tan

as-pd@ansto.gov.au

Australian Government | ANSTO