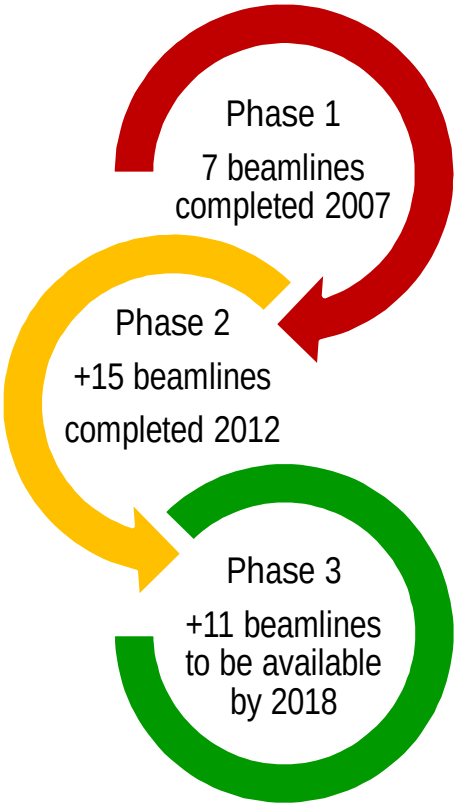
Shining a Light on Formulations: Advanced Materials Characterisation at the Diamond Light Source

Claire Pizzey

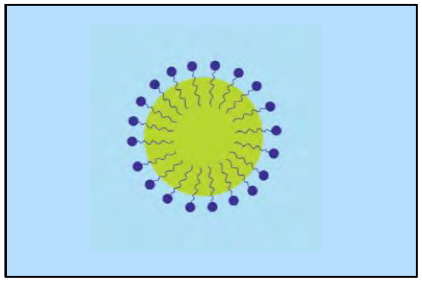
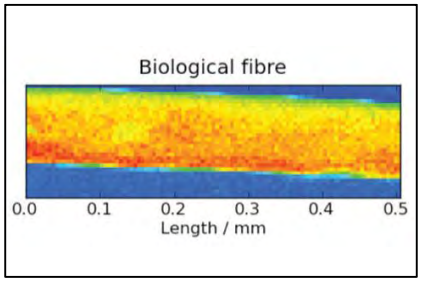
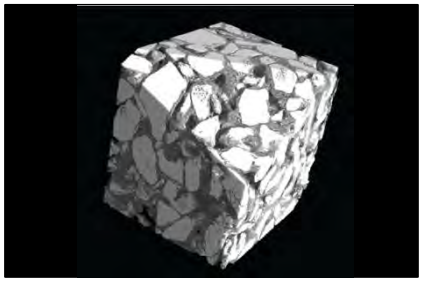
diamond

Diamond Light Source Overview

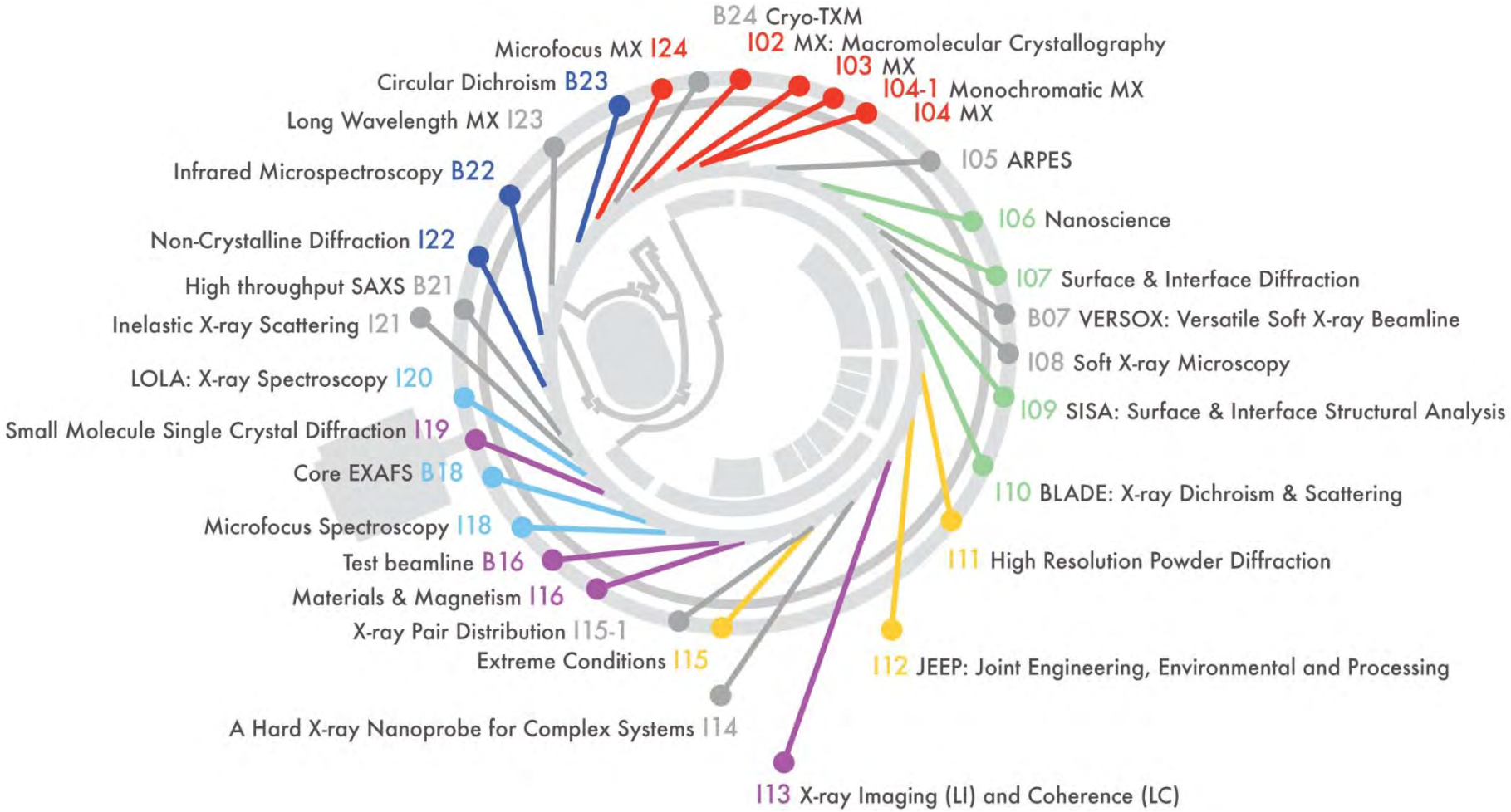
- Largest scientific facility to built in the UK for over 40 years
- Diamond is a private company formed as a joint venture between STFC (86%) and The Wellcome Trust (14%)
- All beamlines are owned and operated by Diamond



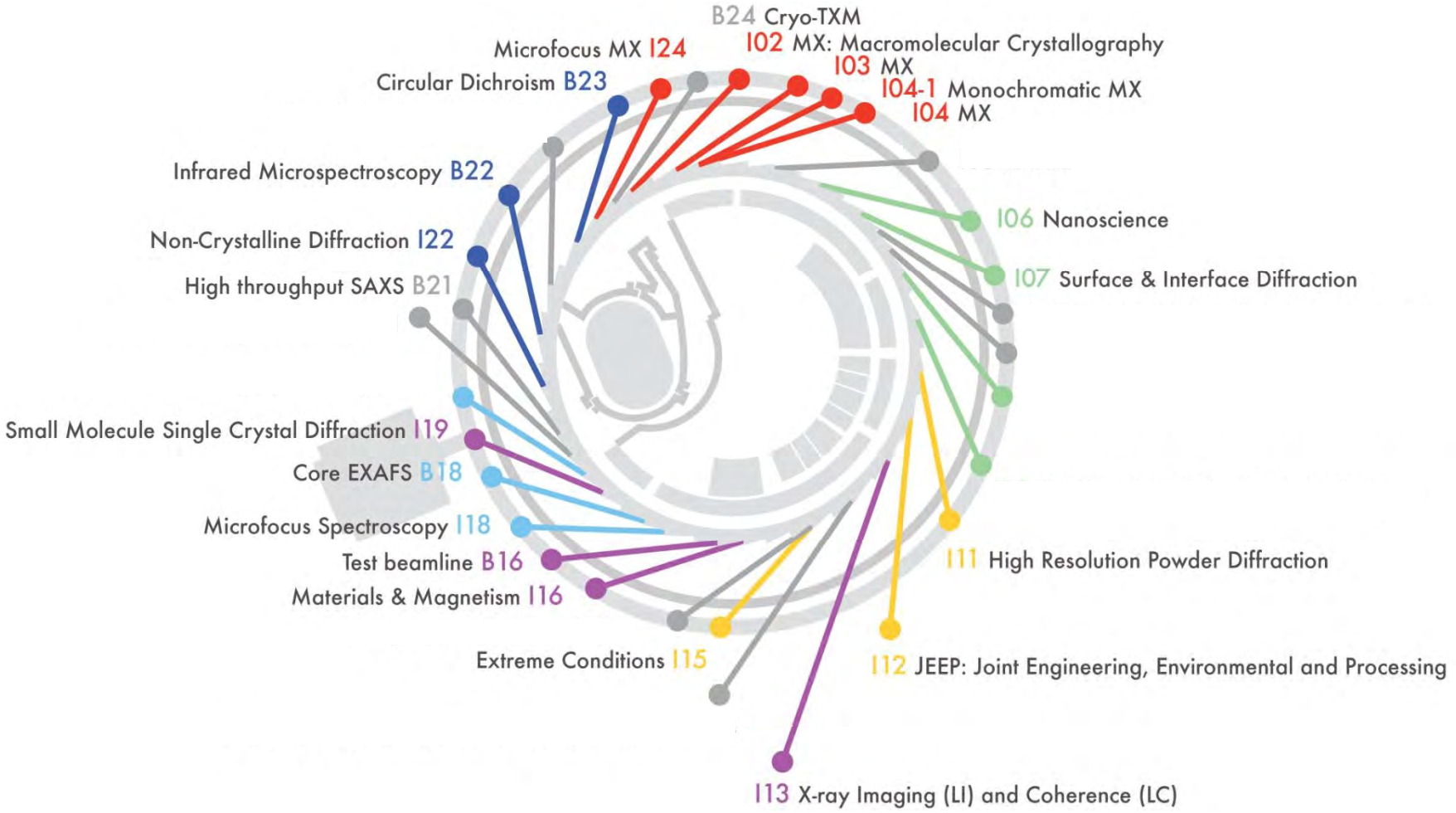
Formulations

Formulation control	Interfaces	Complex Materials	Processing
			
•Structural characterisation of dispersions, emulsions etc. •Phase behaviour in surfactants, lipids and polymers •Particle size and shape analysis •Behaviour of additives	•Surface structure and ordering in detergents, cosmetics •Interfacial interactions in surfactants, polymers and proteins •Adsorbed molecular films	•Element selective investigations (and mapping) of crystalline and amorphous materials •Structural characterisation under controlled environmental conditions	•Structure and thickness of thin films and coatings •Imaging microscale features in bulk samples (cracks, pores or bubbles) •Corrosion, oxidation and flow under *in situ* processing conditions

Beamlines at Diamond

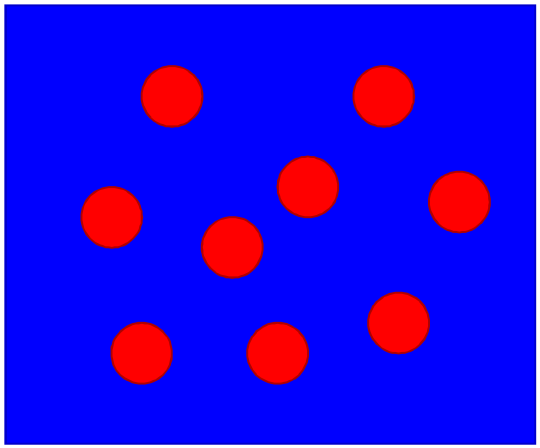


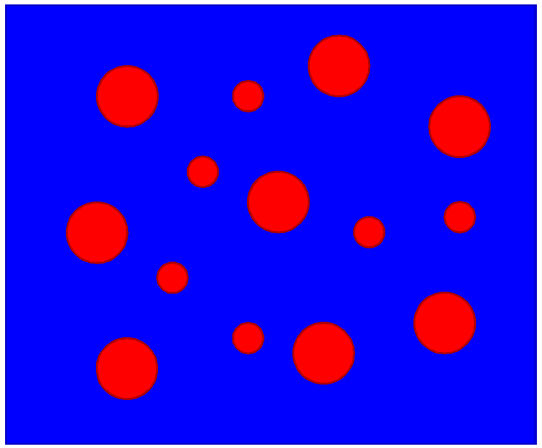
Beamlines at Diamond used by industry



Small Angle X-ray Scattering

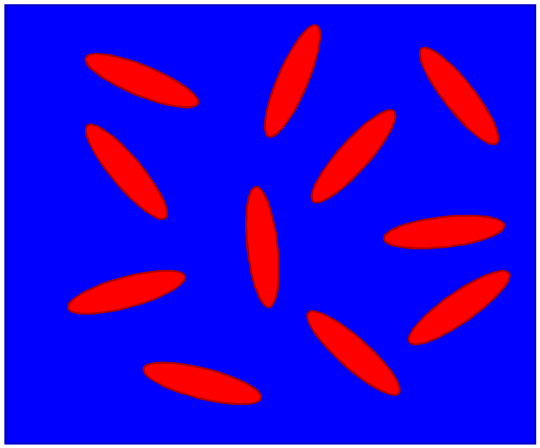
Particle size analysis

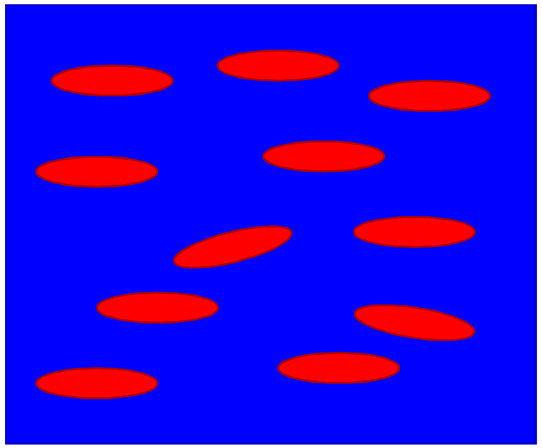




Particle size distribution

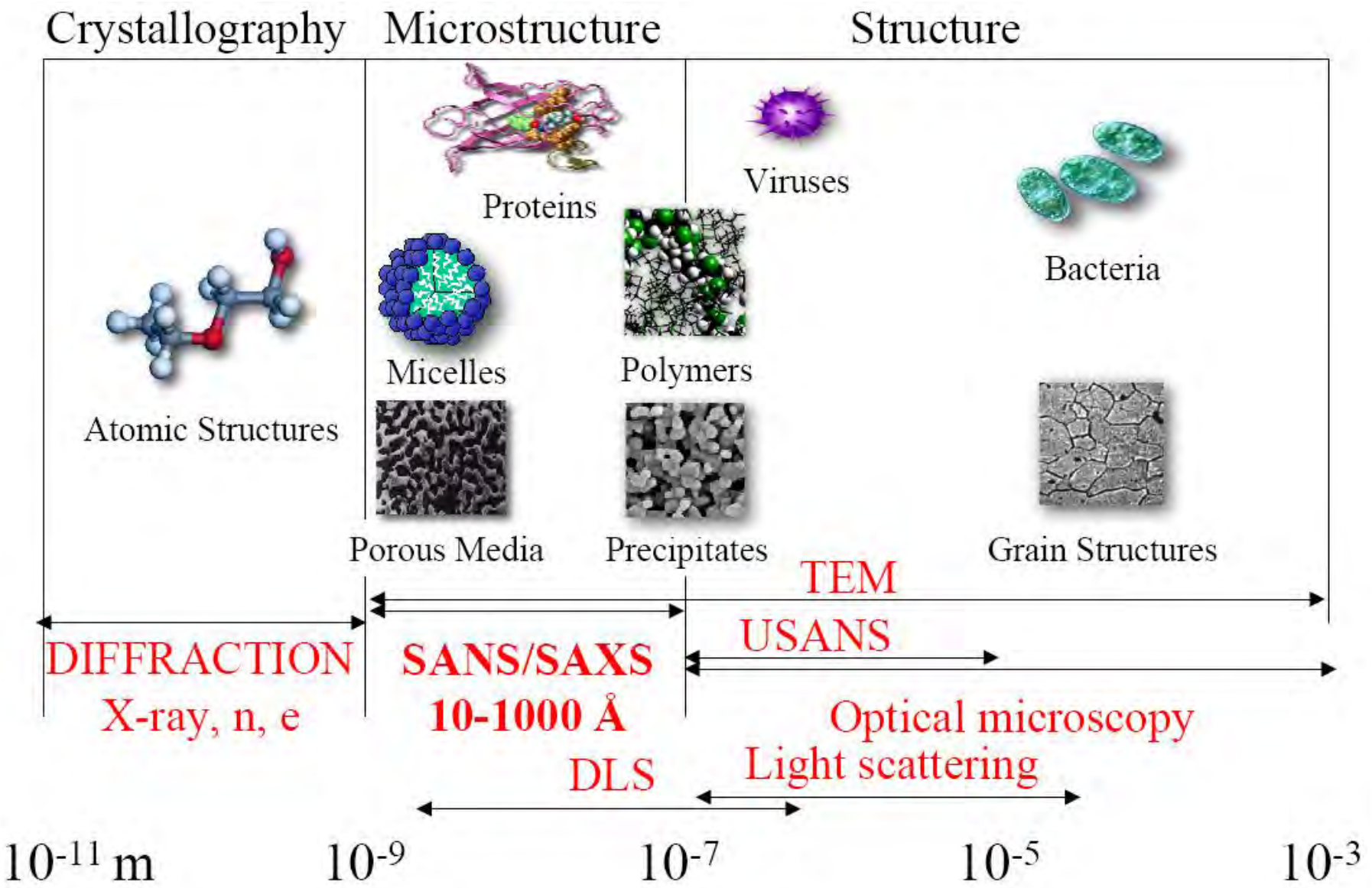
Particle shape analysis

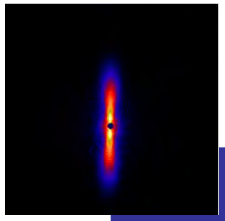




Particle orientation and alignment

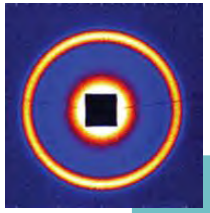
Why is SAXS useful?





Applications

- Structural information from partially ordered materials
- In situ processing
- Wide range of soft matter systems including polymers, surfactants, colloids and proteins
- Self-assembly and ordering



Benefits

- Wide range of sample types
- Can access length scales from <1nm up to 300nm
- Fast data collection & time resolution
- Sample environments e.g. temperature, pressure, shear

Undertaking hair care R&D with Unilever

Rapid innovation into market is key to growth in the consumer products sector. Increased product complexity requires greater understanding of the interplay between components and an accurate description of the product microstructure and its rheological and dispersion properties

Unilever scientists worked as a team with Diamond Industrial Liaison scientists. I22 was used to investigate product microstructure in diluted form for a new hair care product. Data was translated into understanding and communicated to the project team within just a few weeks of the original experiment.

Following the experiments at Diamond, the project accelerated to a working prototype product which went through “in home trial” in late 2013.

Unilever

“The excellent facilities, flexibility and “can do” attitude at the Diamond Light source aligned well with our project needs and objectives. Without this contribution we would not have our current best prototype option to hand. An excellent partnership which bodes well for the future.”
Dr Ian Tucker , Dr Cesar Mendoza, Dr Julia Li, Unilever R&D Port Sunlight

Making the most of waste products

Only a very small proportion of the surfactant used in cleaning products is actually needed to clean, the rest is used as a rheological modifier (thickener).

Cellulose-based materials, waste products of other processes, are abundant, non-food competitive, renewable, sustainable and low cost – the perfect alternative but their gelation behaviour was not well understood.

Researchers used I22 to investigate gel microstructure under a wide range of formulation conditions including variable salt concentration, salt types, surfactant types, pH levels and effect of addition of other formulation ingredients such as perfumes and low molecular weight alcohols to control the behaviour of formulations.

"SAXS experiments at Diamond have enabled us to probe the gelation behaviour of oxidised cellulose nanofibres to explain gel formation under a range of formulation conditions. We have demonstrated that oxidised cellulose nanofibres show great promise for future use as sustainable and low cost formulation ingredients."
Dr Karen Edler, University of Bath

Unilever

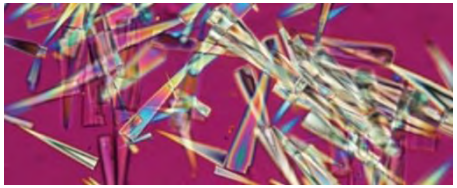
Centre for Sustainable Chemical Technologies

UNIVERSITY OF BATH

Controlling crystallisation in fuels and biofuels

"Freezing" of diesel fuel in winter is a major issue. Wax crystals nucleate and grow and block fuel lines and filters which can lead to vehicle failures and motorists being stranded. The use of biofuels within diesel blends has become increasingly common and can adversely affect the low temperature operability of the fuel.

Infineum scientists have used a range of techniques at Diamond to gain a deeper understanding of the crystallisation processes in biofuels which occur on different length- and time-scales. The results can now be used to direct Infineum's additive modifier design.



"The combination of world-leading capabilities and scientists at Diamond together with the knowledge of Infineum scientists has created unique academic/industrial teamwork with a clear line-of-sight to real-world industrial problems. This has enabled advances in both fundamental understanding and industrial solutions."
Prof. Ken Lewtas, Infineum UK Limited

Smart pigments for energy efficient displays

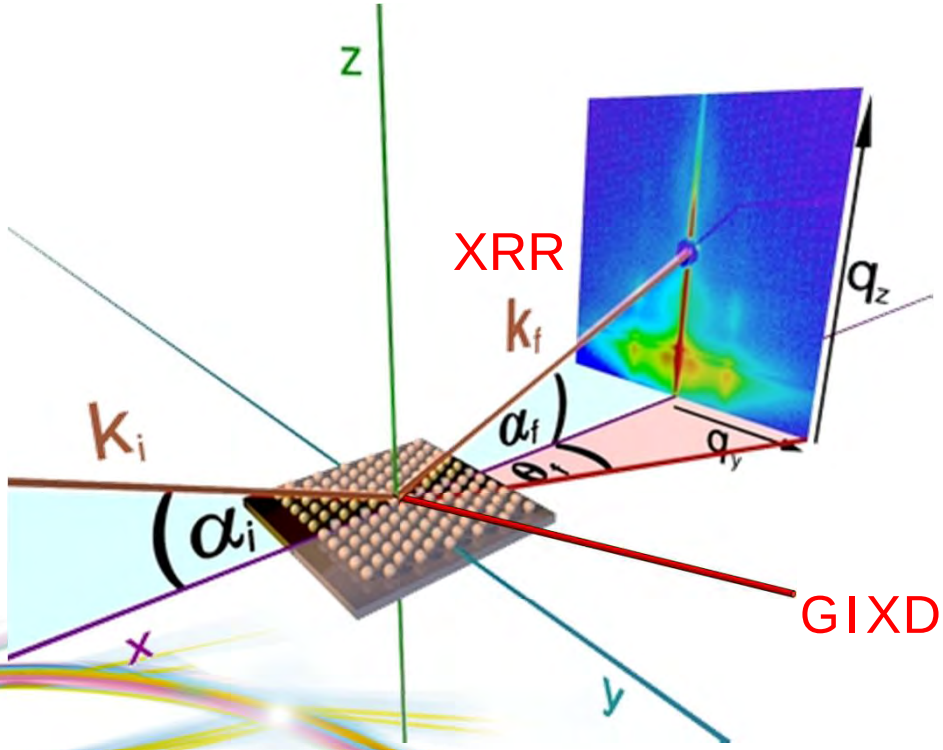
HP were investigating new technologies to replace the inherently energy inefficient liquid crystal display. One promising technology is the use of pigment suspensions which react to an applied electric field with a change of hue.

The team used SAXS to investigate pigment nanoparticle ordering and orientation within suspension under the application of an electric field (required to switch a pixel on or off) on a fast timescale.

The experiments allowed the team to control the behaviour of the suspensions, vital for optimising performance, and select the most promising candidates for future device development.

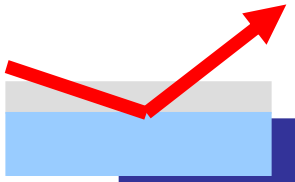
"Small angle X-ray Scattering at I22 allows us to distinguish between the influence of an applied electric field on the pigment particles themselves and on the solvent, a low molecular weight liquid crystal. This information is vital for the design of the most effective pigment suspensions for display application."
Dr Susanne Klein, HP Labs

Surfaces and Interfaces



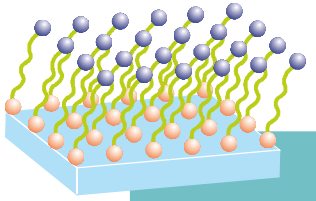
- Structure of thin films (GIXD)
- Changes in film structure with high temperature or pressure (GIXD)
- Structure of layered systems (GIXD)
- Number of layers (XRR)
- Layer thickness (XRR)
- Layer roughness (XRR)

- grazing incidence X-ray diffraction (GIXD)
  - X-ray reflectivity (XRR)



Applications

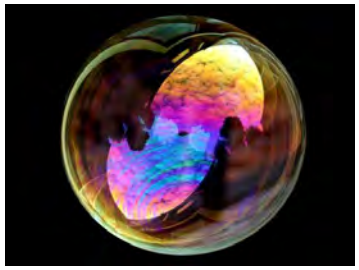
- Phase transitions at surfaces
- Layer growth and structure
- Roughness and wear
- Self-assembly
- Aggregation from solution
- Multilayered materials
- Depth profiling of thin layers



Benefits

- Wide range of interfaces: solid-air, solid-solid, solid-liquid, liquid-liquid and air-liquid
- High resolution structural information
- Down to monolayer coverage

Nanofilms at the mineral-water interface



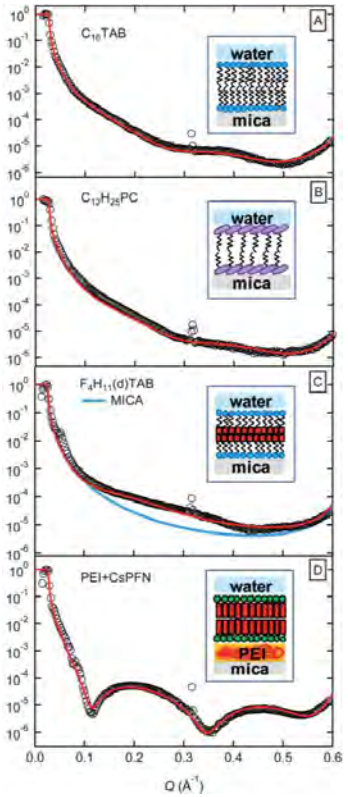
Surfactant behaviour at interfaces is critical to many industrial processes.

Environmental variations can lead to solution composition changes or competitive adsorption processes, strongly influencing the performance of the soft film.

X-ray reflectivity is sensitive to structure of thin films. Provides information on thickness, density, roughness, crystallinity and compositional profile of interfacial layers.

Results show differences in the adsorption of cationic (A), zwitter-ionic (B) and semi-fluorinated (C) surfactants at the water-mineral interface and competitive adsorption processes.

Polymer-surfactant interactions at mineral interfaces rarely reported – experiment allows investigation of an anionic surfactant and a positively charged polymer (D) at the water–mineral interface.

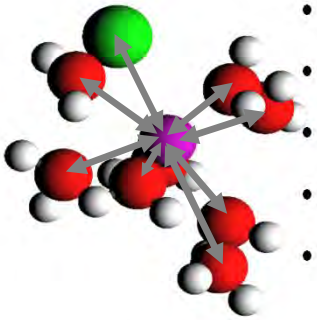


University of BRISTOL

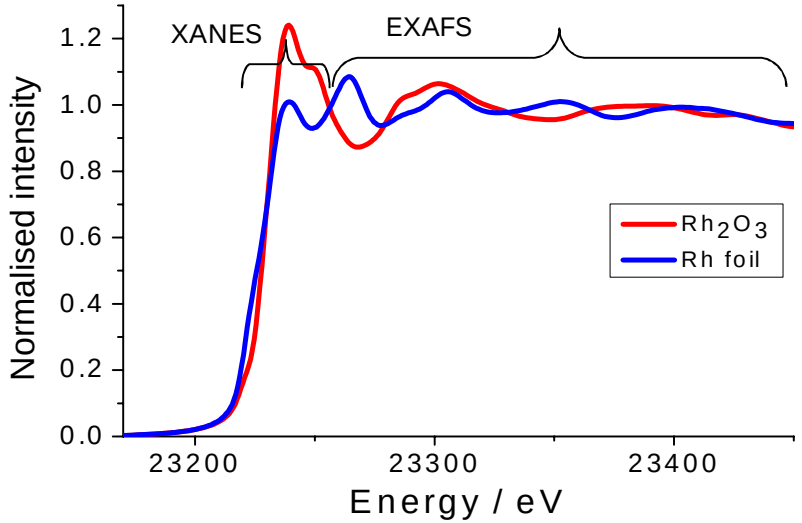
W. Briscoe et al, Soft Matter, (2012) 8, 5055-5068.

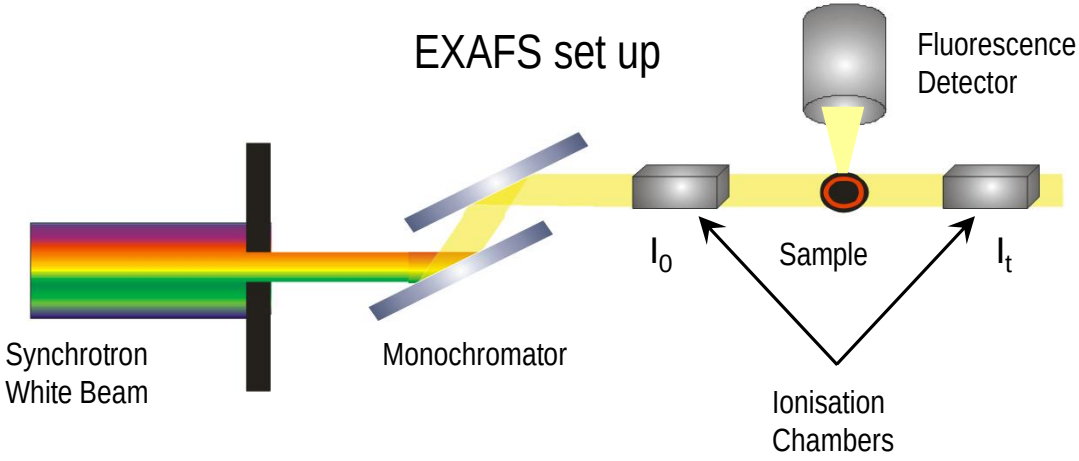
X-ray Absorption Spectroscopy

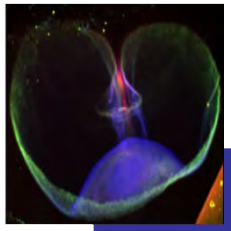
Parameters obtained by XAS analysis



- **Chemical** composition
- **Bond le**ngths
- **Coordin**ation number
- **Geome**try
- **Oxidati**on state

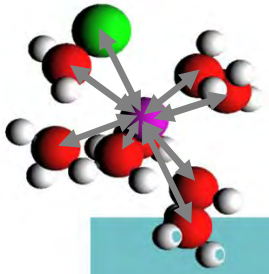






Applications

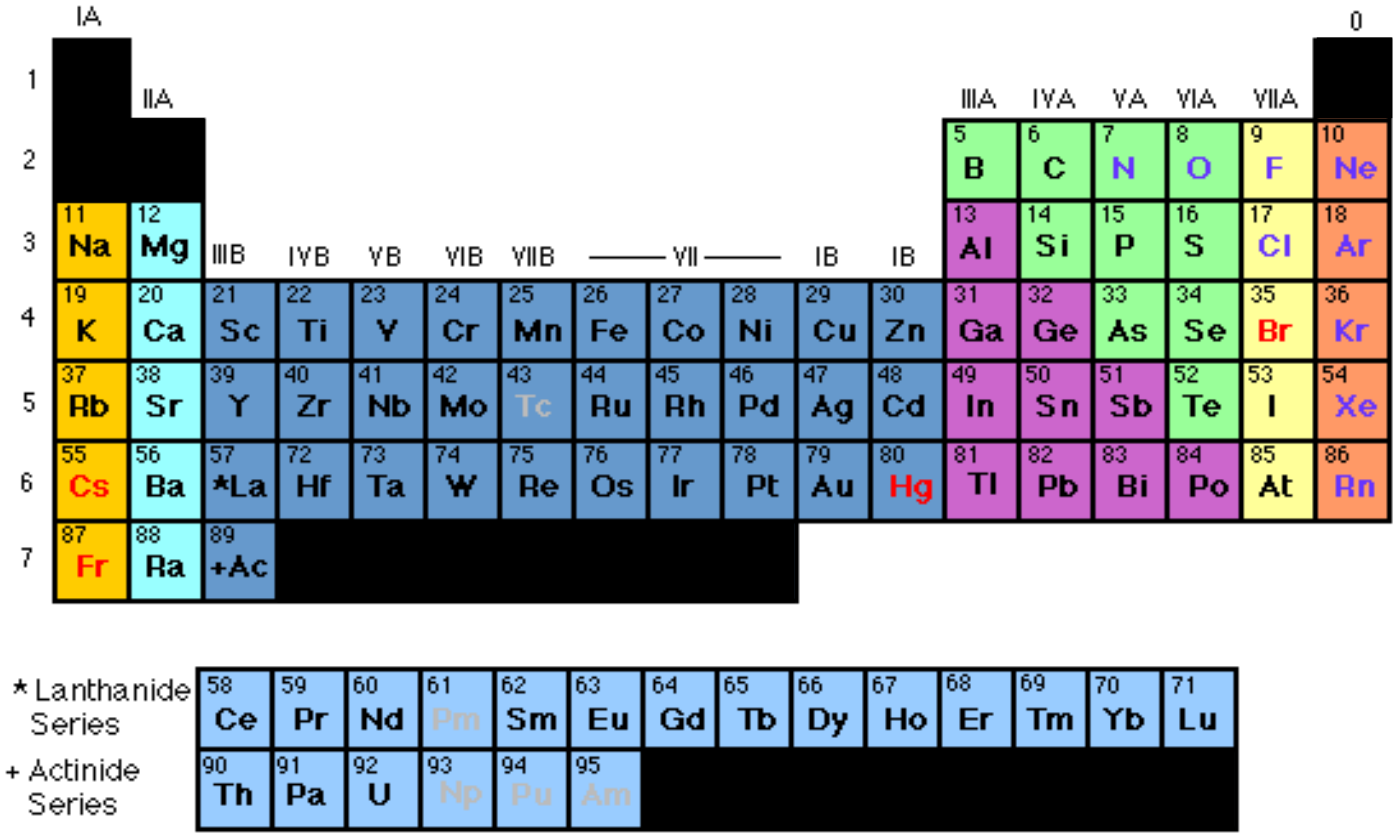
- Oxidation state determination
- Elemental mapping
- Chemical reaction monitoring
- Catalysis
- Electrochemistry
- Photochemistry
- Glasses
- Medical Implants
- Food



Benefits

- Wide variety of sample types; gas, liquid, solid - crystalline or amorphous
- Time-resolved in situ conditions
- Low concentrations possible (down to ppm)
- Sample environments

Accessible elements at Diamond



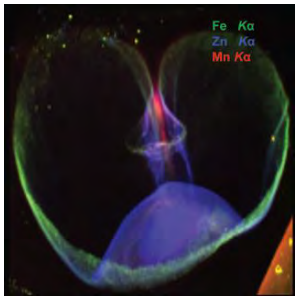
* vacuum conditions may be required for some techniques

Elemental mapping of wheat grain

Iron and zinc deficiencies are particularly serious in Africa, the eastern Mediterranean and south-east Asia where chronic health problems are related to the low intake and bioavailability of minerals from traditional diets comprising little or no meat, fruit and vegetables.

Elemental mapping of wheat grain cross sections was performed on I18 provide localised information metal complexation information.

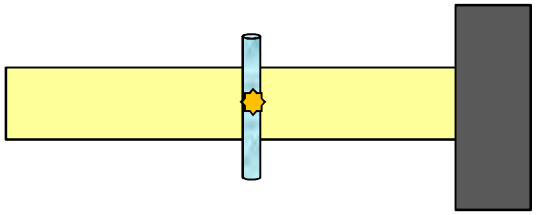
Speciation and location of metals such as Fe and Zn within the wheat grain can give information about the bioavailability of nutrients in different strains of wheat, informing the process of breeding mineral enriched wheat.



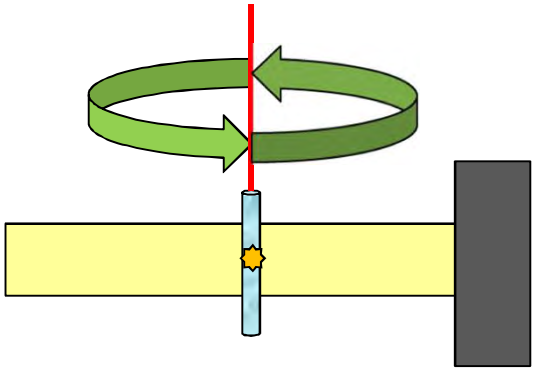
"Even today one billion people are still permanently hungry and millions die each year as a consequence of deficiencies of iron and zinc. This is not good enough. Whether this is a problem of politics, production or distribution doesn't matter: we must explore all avenues to correct this, and it starts with basic scientific investigation"
Dr Andy Neal, Rothamsted Research

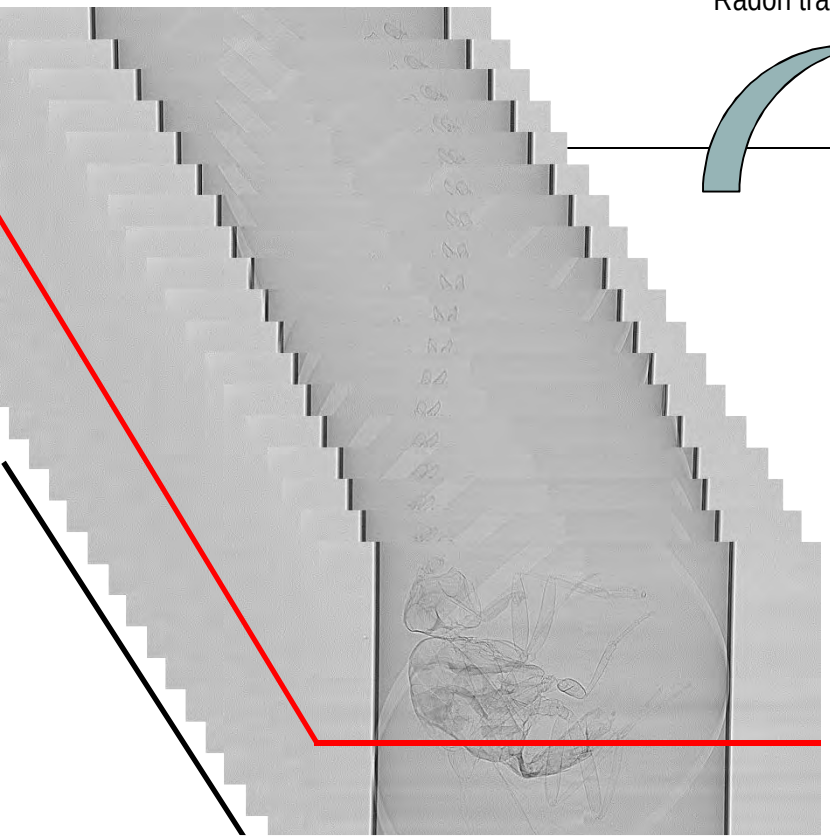
Imaging

Phase contrast or absorption contrast imaging



- Radiography
  - Gives 2D projection of sample; useful for:
    - High speed measurements in dynamic experiments (camera speed >5000fps)
    - Complex experiments
    - Simple samples without too many overlapping structures.
- Tomography
  - 3D construction from a series of projections



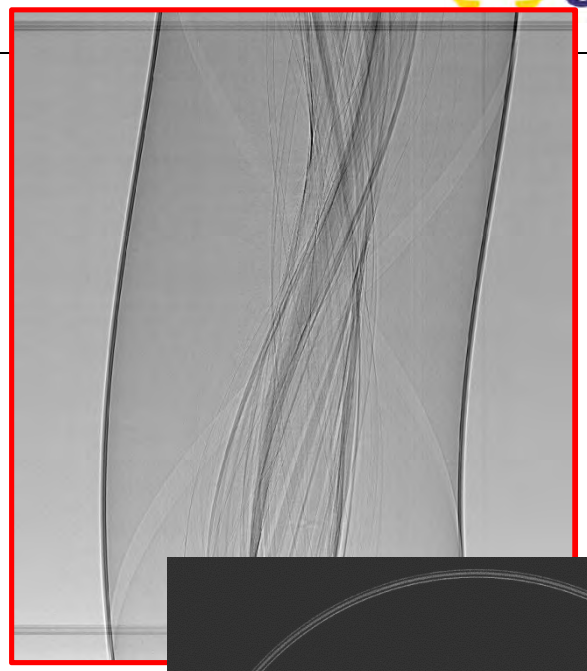


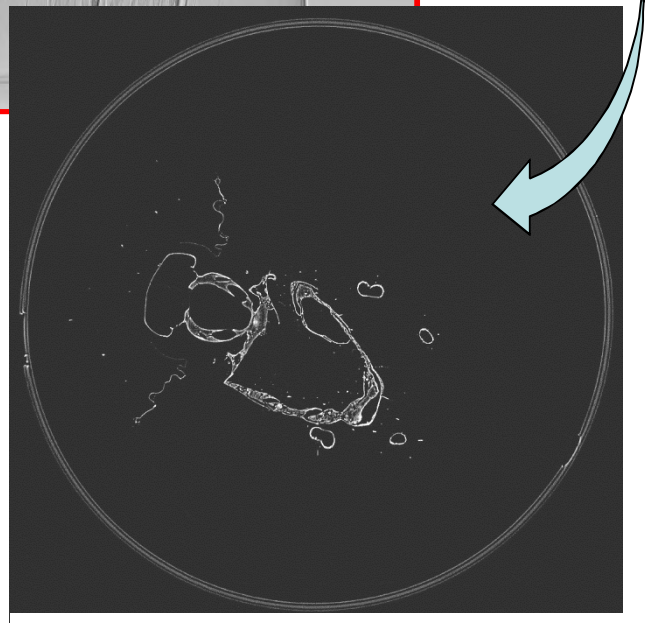
Radon transform

sinogram

θ = 180°

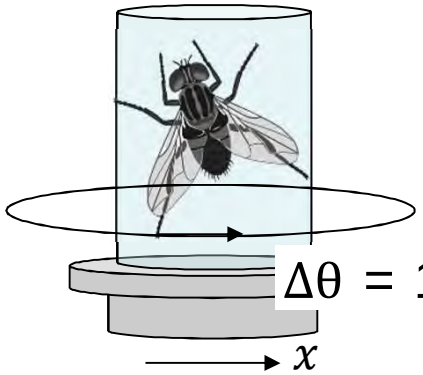
θ





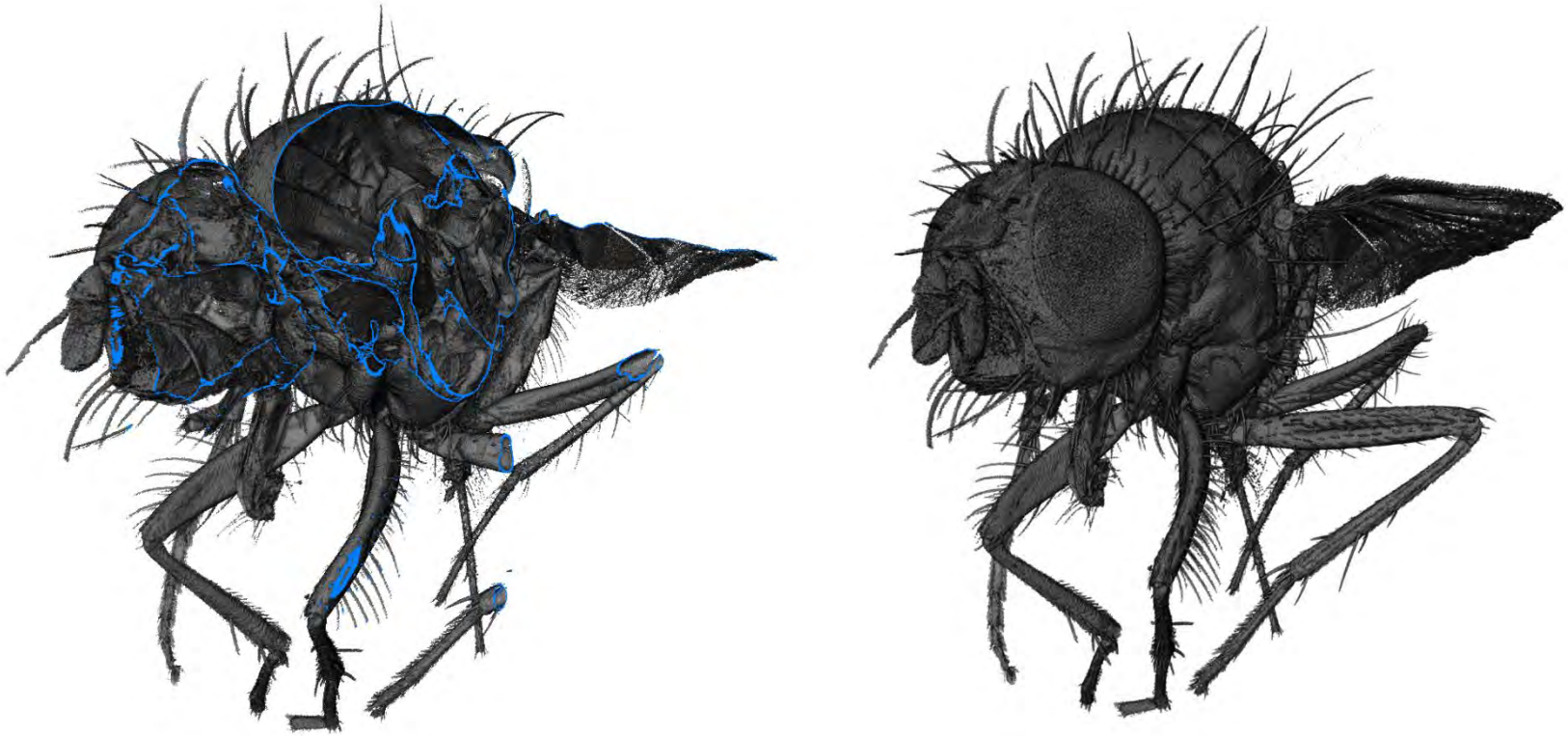
tomogram

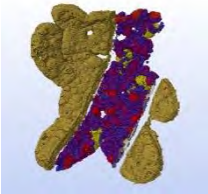
Then we take the inverse radon transform….



How tomography reconstruction works

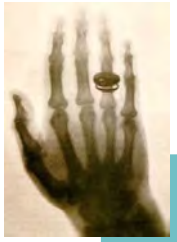
Florian the Fly





Applications

- In situ internal microstructures
- Cracks, voids and bubble development
- Radiographic imaging of complex materials e.g. composites or foams
- Imaging of complex materials e.g. food, medicines, devices,



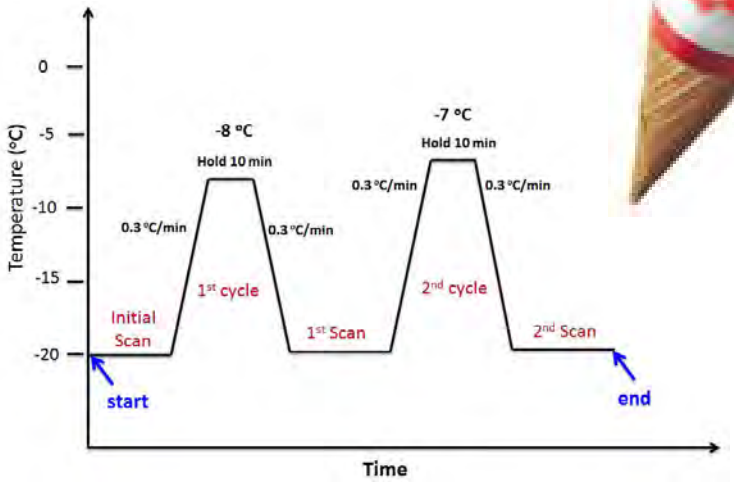
Benefits

- High resolution images
- High speed imaging
- Tomographic reconstruction allows 3D visualisation of materials
- Flexible experiments
- In situ processes
- Non destructive imaging

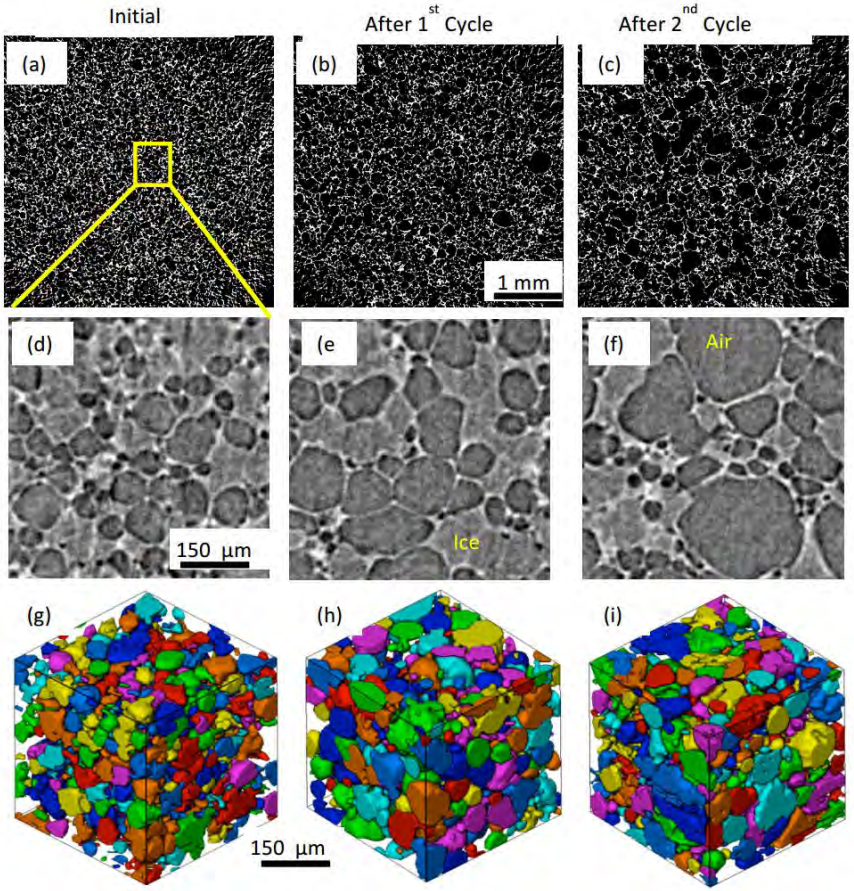
Application: ice cream studies

Growth of ice crystals with freeze-thaw cycles

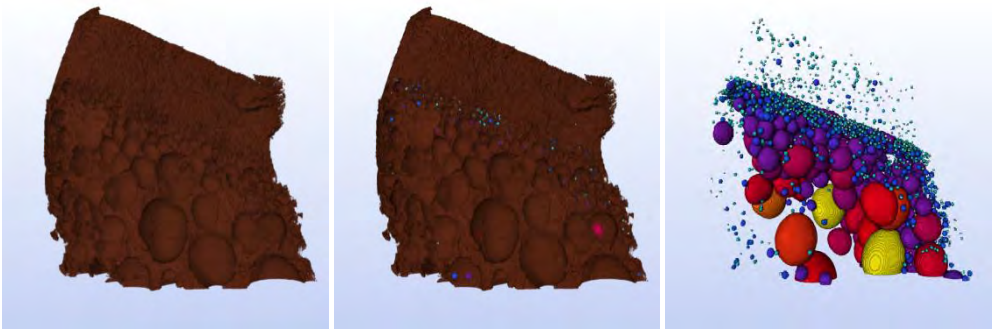
A 4-D dataset for validation of crystal growth in a complex three-phase material, ice cream, P. Rockett , S. Karagadde, E. Guo , J. Bent , J. Hazekamp, M. Kingsley, J. Vila-Comamala, and P.D. Lee. **IOP Conf. Ser.: Mater. Sci. Eng. 84 012076, 2015**

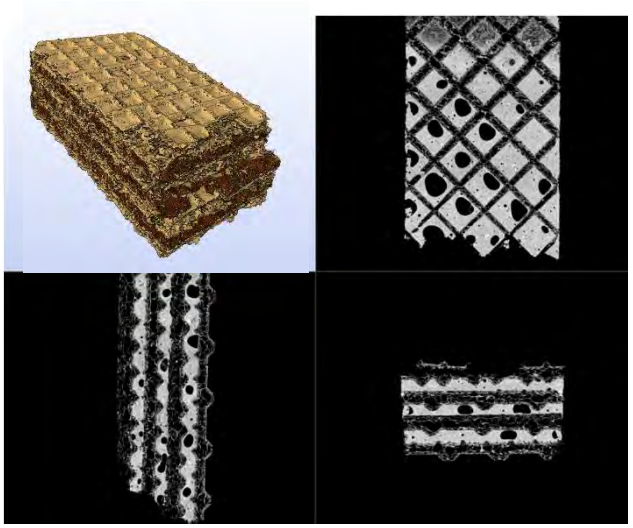


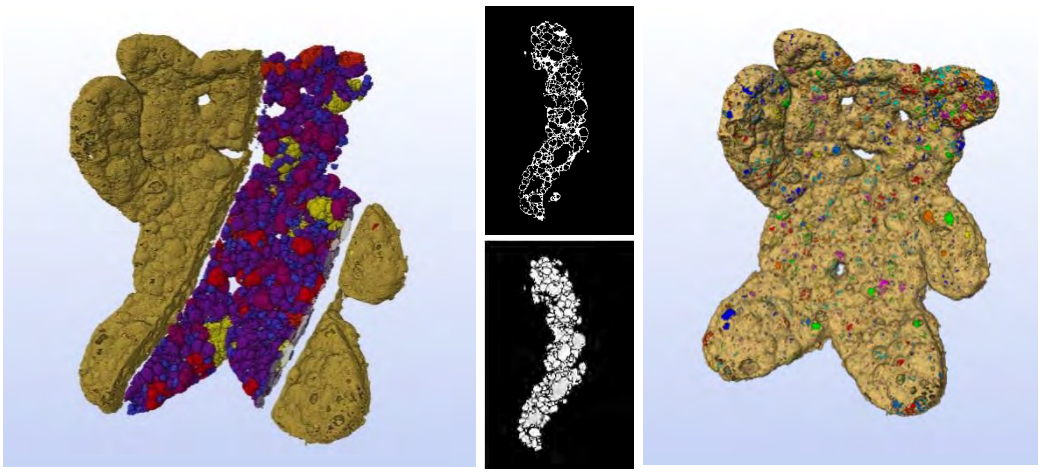
Temperature cycling graph

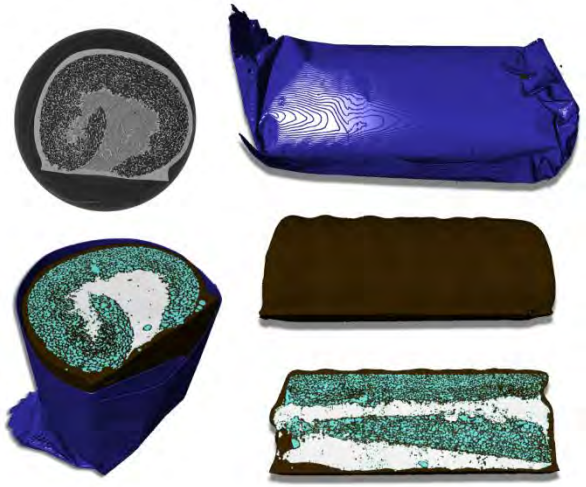


Food imaging examples

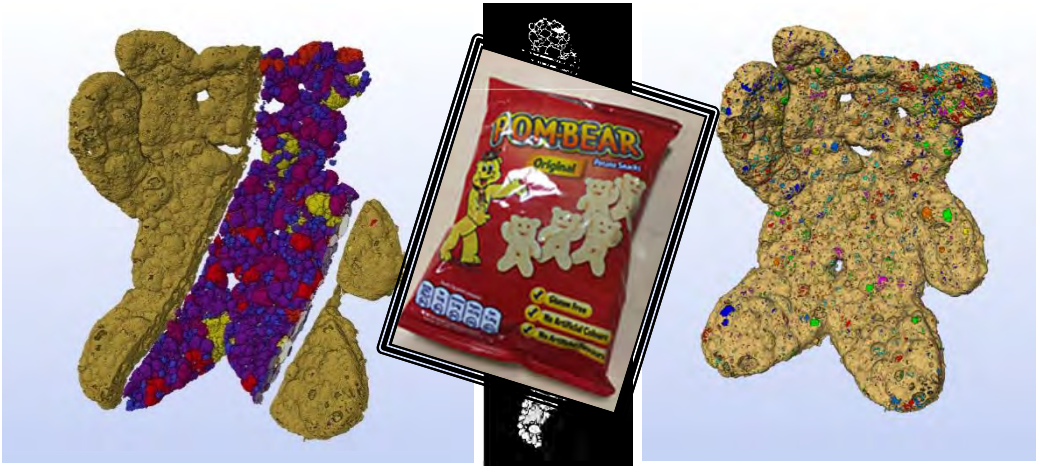




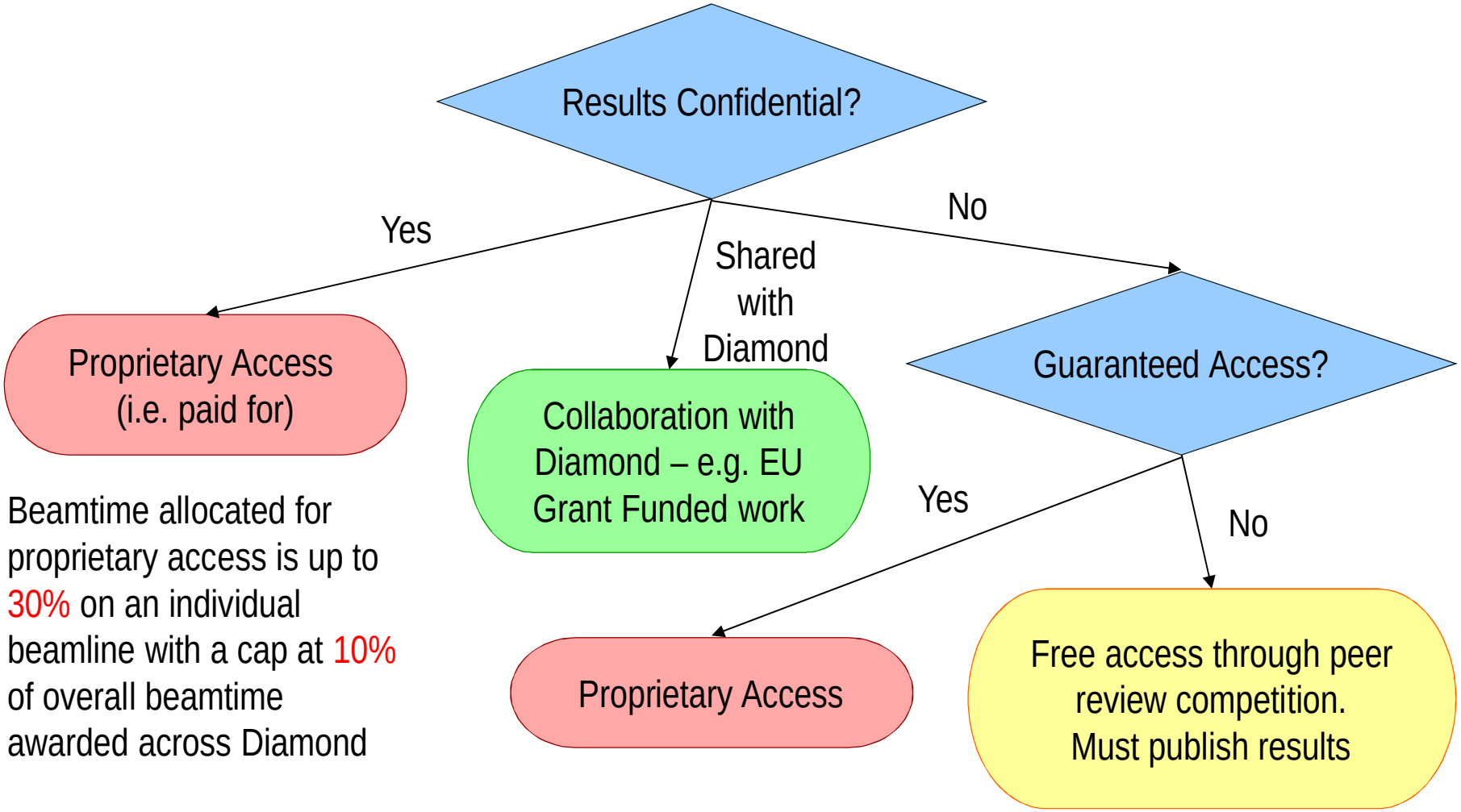




Food imaging examples



Access Modes for Industrial Users



Beamtime allocated for proprietary access is up to 30% on an individual beamline with a cap at 10% of overall beamtime awarded across Diamond

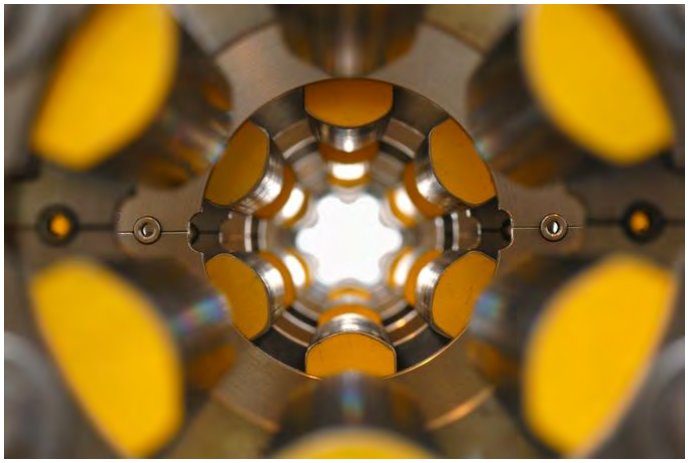
Leading the way for industrial research

Beamtime only

- Priority access
- Pay for what you need
- Ideal for experts

Consultancy

- Dedicated scientific team
- From experiment design to reporting
- No prior knowledge required



Mail-in data collection service

- Submit 1 to 100s of samples
- Rapid turnaround
- Multiple techniques

Remote access

- Collect data from your home lab
- Minimise travel
- All team can participate

Peer review

- With Diamond
- With university
- Apply directly
- Free if awarded time

Collaboration

- Studentships
- Post-docs
- Grants – RCs, EU etc

The Industrial Liaison Group

Elizabeth Shotton

Head of Industrial Liaison

XRPD, SXTAL

Claire Pizzey

Deputy Head of Industrial Liaison

SAXS

Anna Kroner

Senior Industrial Liaison Scientist

XAS

Leigh Connor

Senior Industrial Liaison Scientist

XRPD, Engineering

Alex Dias

Senior Industrial Liaison Scientist

MX

Jitka Waterman

Industrial Liaison Scientist

MX

Sally Irvine

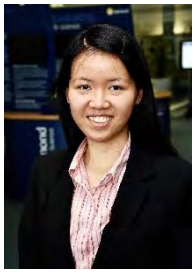
Industrial Liaison Scientist

Imaging

Jason van Rooyen

Industrial Liaison Scientist

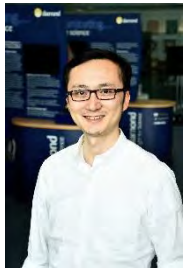
Cryo-EM



Sin-Yuen Chang

PDRA

Soft X-ray Spectroscopy



Renjie Zhang

Lab Manager

Fragment Screening Facility

?

Currently Recruiting

Marketing Manager

DISCo members and some of our partners

Johnson Matthey

Rolls-Royce

GlaxoSmithKline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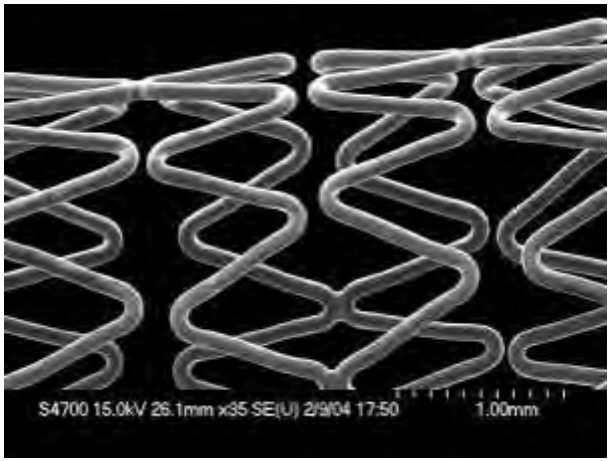
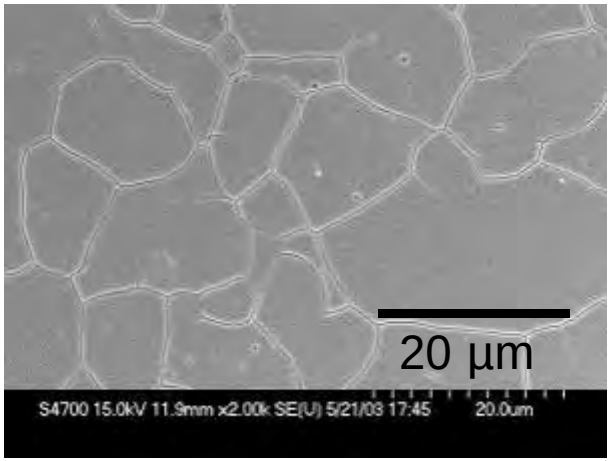
High energy diffraction and imaging

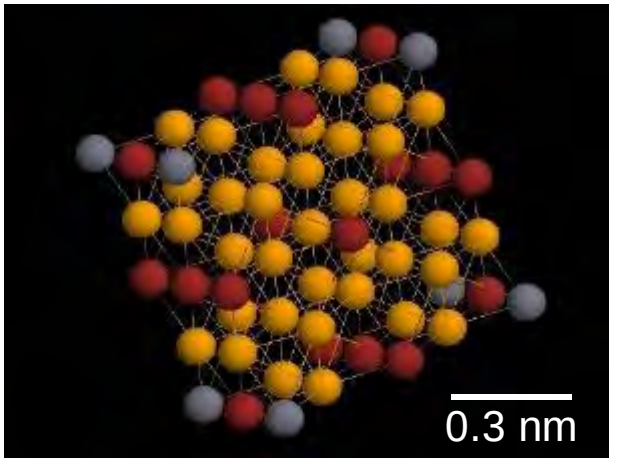
Macroscopic Structure

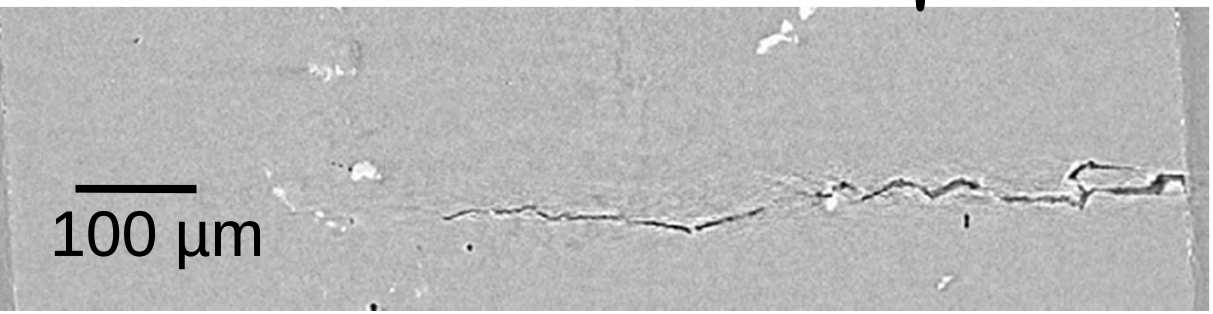


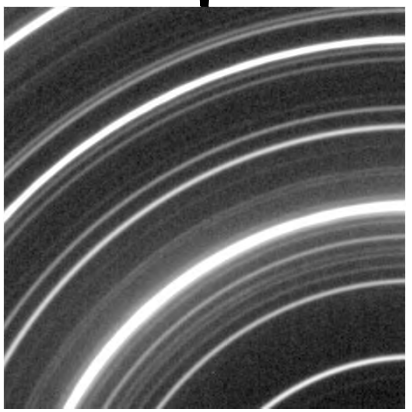
Microstructure

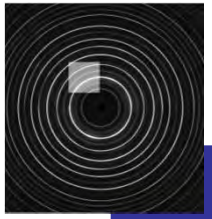


Atomic structure



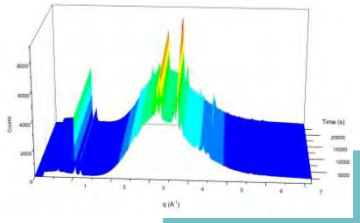






Applications

- Stress/strain measurements
- Cracks, voids and fatigue development
- 3D radiographic imaging of complex materials e.g. composites or foams
- Imaging of flow and compaction processes or chemical reactions

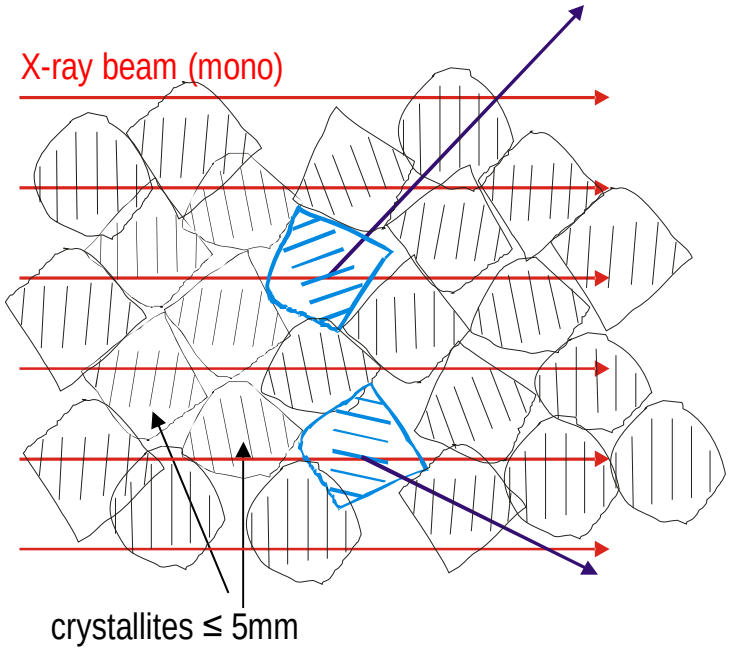


Benefits

- Large samples possible (up to 2 tonnes)
- Large scanning area (1m x 1m)
- Flexible experiments
- In situ processing
- High resolution data and imaging
- Non destructive testing

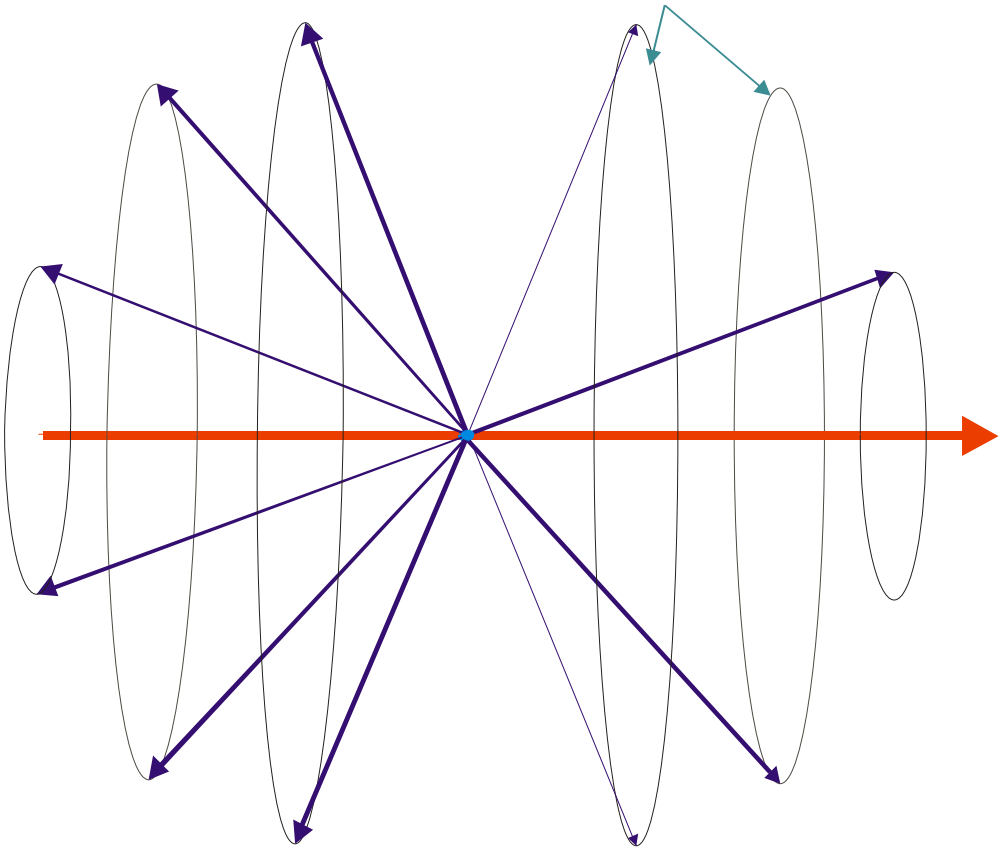
X-ray powder (polycrystalline) diffraction

In powder diffraction, the ideal sample consists of a vast number of very tiny crystallites of random orientation.



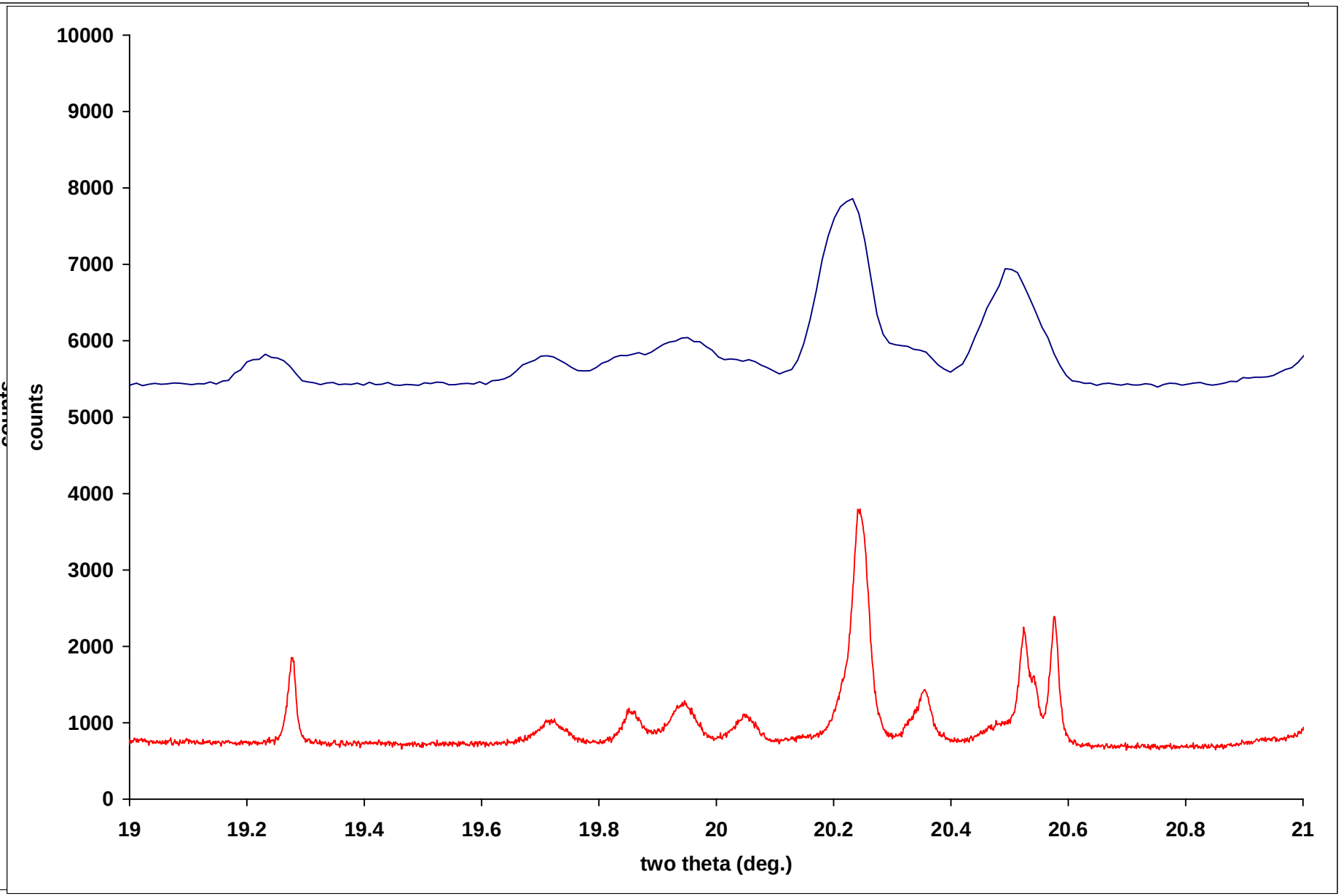
Sample size ~ 0.1 - 5 mm in diameter
~ millions of crystallites

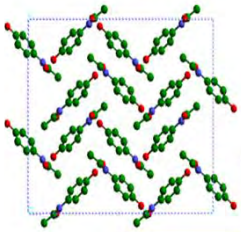
Powder (Debye-Scherrer) rings



... Angular Dispersive

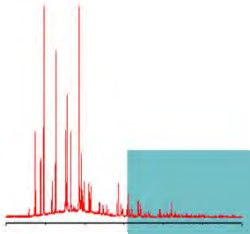
Powder diffraction





Applications

- Structure determination
- In situ reaction monitoring
- Stress/strain measurements
- Particle size measurements
- Polymorph studies



Benefits

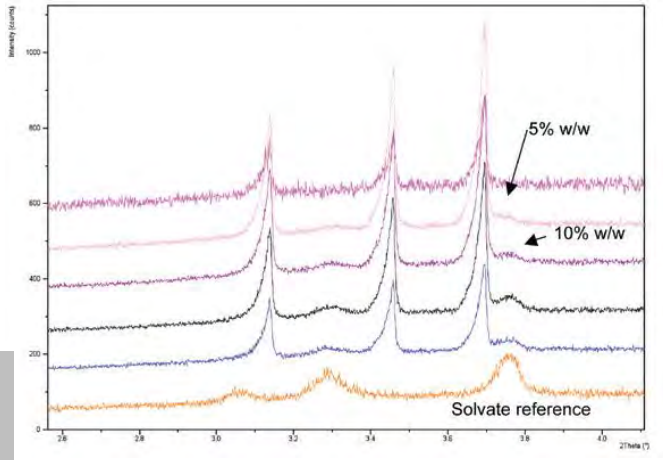
- High resolution data collection
- Fast data collection & time resolution
- High throughput
- Sample environments

Detecting low levels of crystalline API

GSK used I11 to determine the detection limit for the presence of a poorly crystalline solvate within a manufactured drug batch. Lab methods could only detect to 15% w/w.

The samples were loaded in capillaries, flash cooled to 90K and data collected in 1 minute using a helical scan to prevent radiation damage.

A 10% w/w detection limit was confidently achieved which was suitable for specification purposes.



"Without the use of I11, we would not have been able to reach these detection limits that have led to greater understanding and control over the solvate material, thereby allowing us to have confidence in the reproducibility of our manufacturing process."
Dr Matthew Johnson, GlaxoSmithKline