Shining a Light on Particle Characterisation at the Diamond Light Source

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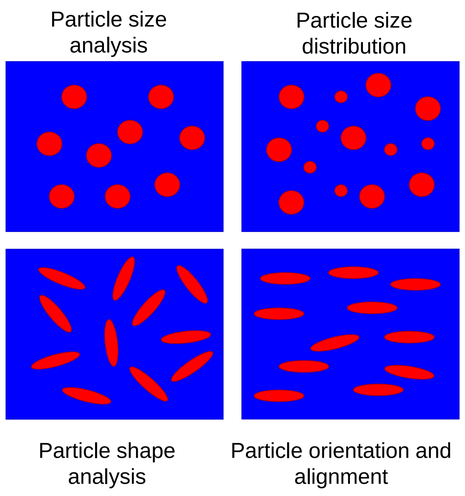
Diamond Light Source, Harwell Science and Innovation Campus, OX11 0DE, UK.

Diamond Light Source is the UK's national synchrotron science facility, located at the Harwell Science and Innovation Campus in Oxfordshire. Diamond generates brilliant beams of light from infra-red to X-rays which are used to investigate physical and chemical behaviour of materials and processes on the atomic, molecular and cellular scale. Clients range from multinationals to start-ups and SMEs working in sectors as diverse as drug discovery and development, food, electronics, formulation, energy, automotive and aerospace engineering. Investigations of microstructure of formulations are of particular interest to a variety of clients with small angle X-ray scattering among the available techniques used to non-destructively probe nanoparticle morphology and suspension microstructure.

Microstructure of Particles and Suspensions

Small Angle X-ray Scattering (SAXS) is a powerful tool for elucidating structural information from complex systems in low ordered environments, for example proteins in solution, colloidal suspensions, liquid crystals and polymeric systems. It is commonly used to probe the nanoparticle suspensions and for characterisation of nanoparticles.

What can we learn?

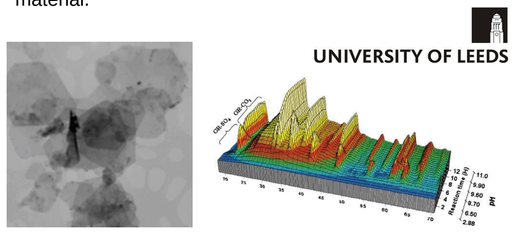


Small angle X-ray scattering arises from differences in electron density between particle and medium, so suitable for opaque and non-volatile samples.

How has this been successfully applied?

Challenge: monitoring nanoparticle formation in air sensitive green rust suspensions

Solution: SAXS experiments were used to follow kinetics and mechanism of particle nucleation and growth in complex experimental conditions (wet, oxygen free). Green rust is able to absorb heavy metals from the environment and is a potential environmental barrier material.



"Diamond is enabling us carry out our experiments incredibly fast, and effectively allows us to bring the contaminated land site onto the beamline. This means that we are, for the first time, seeing exactly how the green rust is effecting the toxic and radioactive species in the soil. On the beamline, we can do in one day what it would take years for us to achieve back in the laboratory. This may bring the reality of a green rust remediation technology within our grasp within years."
Dr Sam Shaw, University of Leeds

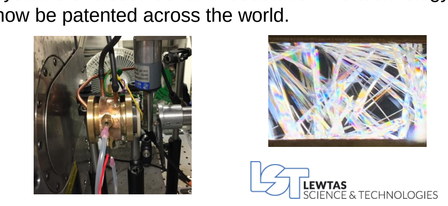
Challenge: microstructural investigations of pigment nanoparticles dispersed in a complex liquid crystal matrix for in built colour in display applications.

Solution: Suspensions in an optically opaque, highly ordered and non-volatile solvent made analysis challenging. SAXS experiments were performed both with and without an applied electric field to switch the hue of the overall suspension and follow structural changes.

"Small Angle X-ray Scattering at I22 allows us to distinguish between the influence of an applied electric field on the pigment particle 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 Bristol

Challenge: demonstrating how ultrasound could be used to control crystallisation from solution

Solution: SAXS experiments were used to follow particle nucleation and growth at molecular and mesoscales. Using specially designed and built acoustic cells, they applied SAXS and WAXS analytical techniques to a mixture of waxes, fats and cocoa butter to simulate model systems of diesel fuel and foodstuffs. The technology has now be patented across the world.



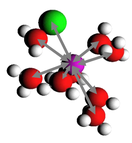
"Wonderful things can happen when you bring together imaginative academic, industrial and Diamond people together with the best scientific facilities! Access to I22 SAXS beamline at Diamond made a real difference to the development of our project. We were able to apply a purpose-built liquid cell with acoustic transducers into the beam and look at the associations and dissociations of molecules with and without an applied acoustic field. Using model compounds to simulate food and fuels we were able to observe all the changes and make adjustments in real time. The support of the team at Diamond was invaluable in making this happen and we were able to immediately check our theories and later verify them when all the data was examined."
Ken Lewtas, Director, LST

Understanding Chemical Speciation

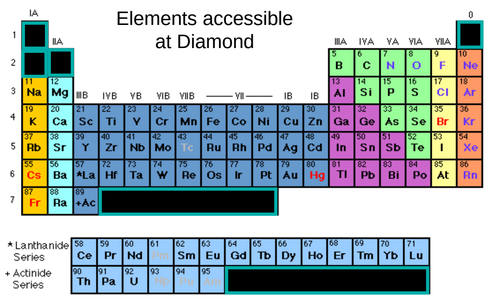
X-ray absorption spectroscopy (XAS) is a powerful chemical probe providing element specific information on solid liquid or gas samples.

What can we learn?

Parameters obtained by XAS analysis



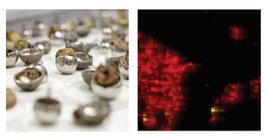
- Chemical composition
- Bond lengths
- Coordination number
- Geometry
- Oxidation state



How has this been successfully applied?

Challenge: understand chemical speciation in nanoparticle wear from metal-on-metal hip implants

Solution: chemical mapping to locate nanoparticles dispersed in tissue surrounding hip implants and identify chemical speciation and correlate with clinical mode of failure such as infection or allergic response.

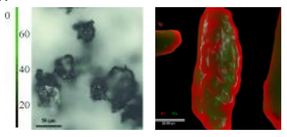


UCL Royal National Orthopaedic Hospital NHS Trust

"This is the first time synchrotron radiation has been used to chemically characterise implant derived tissue from MOM hips. We have demonstrated the usefulness of synchrotron techniques in being able to identify low concentrations of material and in determining the metal speciation."
Mr Alister Hart, University College London and Royal National Orthopaedic Hospital

Challenge: monitor micro-distribution of elements within a heterogeneous catalyst particle (solid in liquid reaction).

Solution: chemical tomography to identify where Pt and Mo metals are located in the particles and identify how different preparation methods affect the activity of the catalyst.



UCL

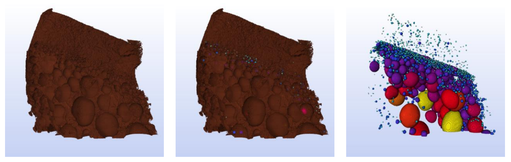
"Imaging a working catalyst gives even more information on the exact working mechanism of the metals and stabilizer. For such imaging, it is important to use conditions that are close to the working conditions of a commercial catalyst. These catalysts are most often used in fine chemical applications, which are typically run in liquid phase at mild conditions. The current work shows that the stabilizer is removed from the metal surface under reaction conditions. It is most likely that different results would be obtained if this catalyst were to be imaged under (unrealistic) gas phase conditions."
Dr Peter Witte, BASF

Visualising Particle Structure and Morphology

X-ray imaging allows 2D or 3D visualisation of samples without the need to destroy the sample.

What can we learn?

- Radiography (2D)
- Tomographic reconstruction allows 3D visualisation of materials
- High speed
- High resolution

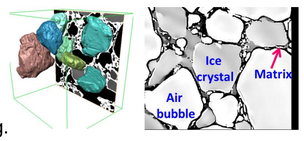


**Disclaimer: purchased and used as a test sample only*

How has this been successfully applied?

Challenge: understanding changes in ice cream microstructure during repeat freeze-thaw cycles.

Solution: X-ray tomography was performed with high temporal and spatial resolution to probe the complex microstructural evolution throughout temperature cycling.

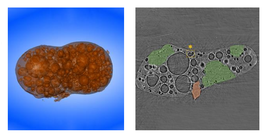


MANCHESTER 1824 Unilever

"The unique qualities of the Diamond-Manchester branchline (in-line phase contrast in pink beam) allowed us to study how processing conditions and ingredients affect the taste of ice cream, helping us better understand our product. By collaborating with Manchester we developed an in situ rig that replicated the processing conditions making it all possible."
Julian Bent, Unilever

Challenge: understand the physical and chemical nature of the radioactive particles from Fukushima restricted zone.

Solution: X-ray tomography and microfluorescence measurements performed to analyse its internal structure and 3D elemental distribution, providing insight into the mechanism of dispersion of radioactive elements into the environment.



JAEA University of BRISTOL

"The analytical capabilities, techniques and equipment afforded by Diamond Light Source are unique and have enabled us to undertake crucial research into the Fukushima Daiichi Nuclear Power Plant accident that would not otherwise be possible. The results from Diamond have directly influenced decommissioning strategies on the accident site, resulting in significant cost and human dose reductions."
Dr Peter Martin, South West Nuclear Hub

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