

CASE STUDY

Changing how and when materials crystallise

The control of crystallisation is a perennial problem in many aspects of life – for example: the manufacturing of foodstuffs, polymer products, pharmaceuticals and the operation of transportation fuels. There has always been a wish to be able to change the nucleation and growth of crystallising species simply and efficiently. This would enable the control of wax crystals from diesel fuel, speed up polymer processing, and make pharmaceuticals and dairy products more efficiently.



The Challenge

Ultrasound has routinely been used across many sectors to monitor industrial manufacturing processes, but instead of simply monitoring the process of crystallisation, LST wanted to see if it was possible to use ultrasound to modify and affect the actual crystallisation process.

The Solution

Working with Professor Megan Povey from University of Leeds, LST monitored crystallisation with ultrasound and, to begin with, the energies needed to affect the 'critical nucleation processes' (when a viable crystal is formed that can grow) were calculated. It was predicted that only low power ultrasound was needed and the first experiment was successful in stopping the nucleation of wax from solution and then starting it again when the low power ultrasound was switched off. This idea has now been patented around the world.

Using A4I and EPSRC grant funding, the team were able to further test their theory *in situ* on Diamond's I22 beamline to fully understand the process at which crystallisation occurs. 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.

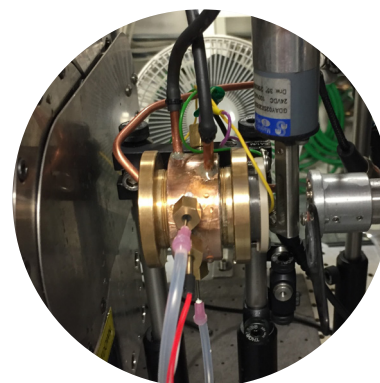
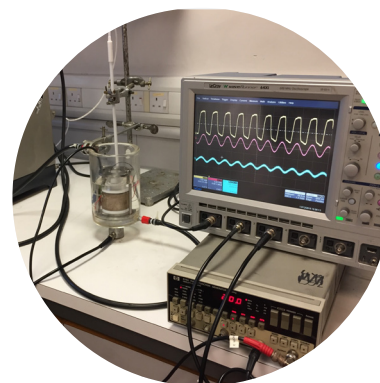
In addition, they are extending this invention into the high-pressure regimes with Dr Nick Brooks at Imperial College London. Modern diesel engines are now using extremely high pressures (3 kbar) and this means that wax will crystallise at higher temperatures. Research at Diamond has shed further light on this phenomenon.

The Benefits

By using the instruments at Diamond, LST, Leeds and Imperial were able to monitor the process of crystallisation at the molecular and meso-scales. They were able to combine synchrotron techniques with acoustic methods to understand crystallisation processes happening at a molecular level. By using Diamond's world-class instruments they were able to watch *in situ* as the process evolved. By understanding and manipulating the process at which crystallisation occurs in foodstuff and diesel fuel, we will be better able to create efficient food processes and diesel fuel systems which effectively control vehicle emissions. Future developments are in progress include extending the process to other industries such as polymer processing, e.g. extrusion and injection moulding of semi-crystalline polymers.



“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**



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