Coherent X-ray diffraction imaging to investigate structure and morphological evolution of calcium carbonate microparticles

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Coherent X-ray diffraction imaging (CXDI) is a powerful lensless imaging technique utilizing X-rays with a high degree of coherence [1]. Conceptually, CXDI can image isolated microscopic objects with high resolution and since its first demonstration in 1999 [2] it has laid the foundation for the development of other methodologies such as ptychography and Bragg CXDI [1]. With the new low-emittance storage ring [3] at ESRF combined with a state-of-the-art Eiger 4M detector and efficient iterative algorithms, the ID10 beamline is optimised for CXDI experiments. Three-dimensional imaging of crystalline and amorphous particles at ~14 nm resolution [4] has recently been demonstrated. In contrast to conventional characterisation methodologies such as electron microscopies, CXDI is ideally suited to study the surface morphology and interior of such microscopic particles without sectioning or ion-milling.

We used CXDI to image in 3D a series of CaCO₃ microparticles prepared under different crystallization and growth conditions, revealing that the microparticles systematically assume a wide range of morphologies as function of temperature [5]. In this presentation, we demonstrate 3D CXDI imaging of CaCO₃ particles 3-6 μ m in diameter, with 16 nm voxel size. Wide-angle X-ray diffraction (WAXD) patterns [6] were recorded in combination with the CXDI datasets to identify the crystalline phase of the CaCO₃ microparticles and obtain information of characteristic crystal planes. Figure 1 shows the evolution of CaCO₃ particle morphology as a function of precipitation temperature: from the nested hexagonal morphology at $T = 25^{\circ}$ C to an appearance of spikes at $T = 35^{\circ}$, and finally transforming to an extended rod-shape morphology at $T = 45^{\circ}$ C. In addition, the internal structures of the particles and density variations within the particles can also be appreciated in 3D. Finally, we discuss the challenges arising due to radiation damage and how to resolve them, and also the future prospects of dynamic or serial CXDI experiments.



Figure 1. 3D iso-surface renderings of calcium carbonate particles precipitated in solution at a) 25°C b) 35°C c) 45°C

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