ESRF	**Experiment title: Coherent diffraction Imaging of Hollow Particles of $CaCO_3$ for Encapsulation of Proteins**	**Experiment number**:
Beamline:	**Date of experiment**: from: 4/02/2015 to: 10/02/2015	**Date of report**: 01/03/2015
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Report:

The aim of this experiment was to carry out coherent scattering by hollow spheres of vaterite (metastable phase of $CaCO_3$) to image the inner structure of these beads and their porosity. The hollow core is about 0.7µm in diameter and the shell is also porous with pores which are mesoscopic in size) [1,2].This information was recently achieved by conventional SAXS experiments performed at ID02. It was shown that the beads exhibit hierarchical porosity [2]. Such beads are of great interest as a host matrix for proteins [3]. We have already shown using fluorescence spectroscopy that proteins can be embedded in such porous mineral structures [3] (see Fig. 2). One of the key issues of this experiment was to apply Coherent Diffraction Imaging (CDI) on such beads with and without the proteins inside.

In such an experiment the particles are deposited on a Si_3N_4 membrane and one of them is selected by the help of a microscope to carry out the CDI.

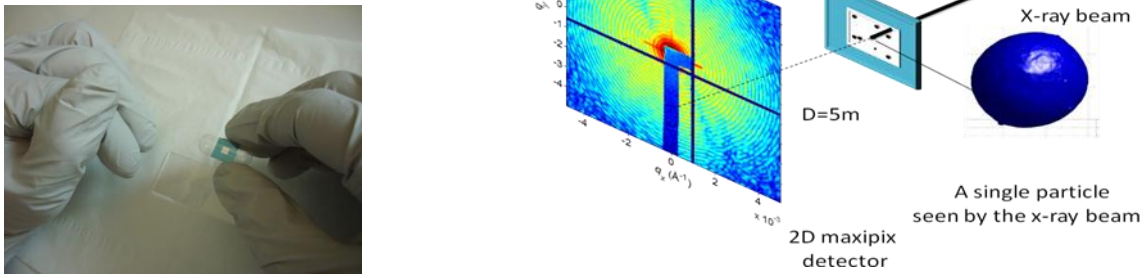

Fig1 : Si3N4 membrane and a sketch of a the experimental set-up

The scattering by a single particle is then collected on a 2D maxipix detector at a given angle as shown in Fig.1. In order to obtain the tomography of the particle, the membrane is rotated about the vertical axis from -80° to +80° with a 0.5° step. At each angle a scattering pattern is collected. One can see that the scattering pattern is mainly defined by a speckle pattern which contains information about heterogeneities of electron densities inside the scattering object. After collecting all the patterns, the 3D Fourier matrix is assembled and the reconstruction of the scattering object in the real space is obtained by phasing the 3D Fourier data using a phase retrieval algorithm.

As this experiment was run in February 2015, we have tested a few reconstruction and we report here some of the very stunning images that we were able to extract from this run. In order to fully validate the algorithm of reconstruction we compare the CDI results with those obtained by SEM on the same particles. Note that the position of the particle was carefully monitored on the membrane to be able to carry out such a cross-check.

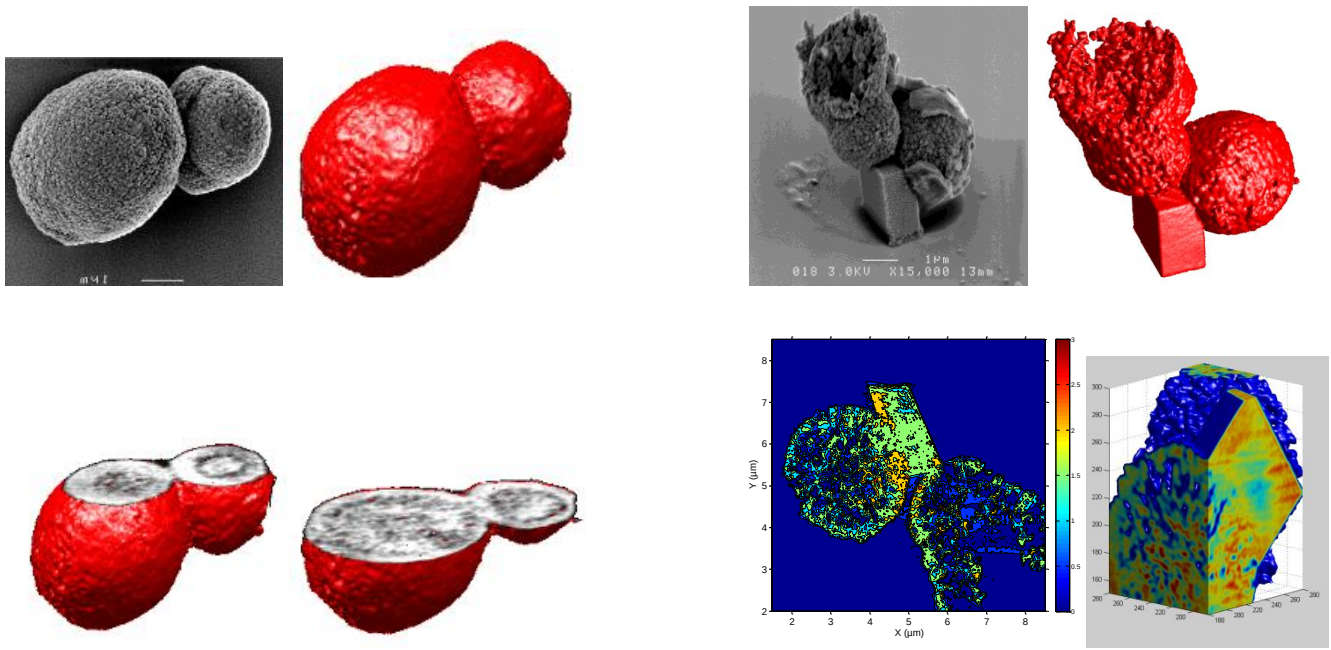

Fig. 2 : SEM and CDI reconstructions of the same particles. On the left panel we show a vaterite particle made of the agglomeration of two particles. The bottom part of the figure shows iso-contour intensity plots of the inner part of the particle where one can see the porosity (in grey). On the right part of the figure we show three agglomerated vaterite particles that are hollow in their center and that are transforming into the more stable calcite phase of $CaCO_3$. Iso-contour representation of a slice in the previous particle, showing the very large porosity in vaterite and a more compact particle of calcite. The same with slice at 90° inside the particle. Note that the angle of the calcite particle is either 104° or 76° which is in perfect agreement with the rhombohedral structure of calcite.

The two above examples are given to demonstrate the validity of the reconstruction procedure. So far we are still in the process of analyzing the data. In particular we want to extract as much information as possible from the reconstructions such as the overall porosity and the density profile of the particles (as shown in the slice on the right panel). We expect to be able to go beyond these first representations. We would like to observe if proteins are inserted in the porous particles of vaterite, for which it is compulsory to make sure that the reconstructed density profile inside the particle is reliable.

References

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