



Experiment title: EC-419 (Proposal ID 20750)

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Report:

We report on a Coherent X-ray Diffraction Imaging (CXDI) Experiment on biomineralized carbonate and greigite specimen and biological samples at the ID13 Microfocus Beamline. The spherulites of the coralline demosponge of *Astrosclera willeyana*, symbiotic associations of methanogenic archaea and sulfate reducing eubacterian, namely ANME-2 (anaerobic methane-oxidizing archaea)/ greigite-bearing *Desulfosarcina/ Desulfococcus* group (DSS), unstained freeze-dried and cryogenically cooled cells of the bacterium *Deinococcus radiodurans* as well as a simple Pollen have been studied at 15.25 keV photon energy in different imaging modes.

For the experiment, we have used the nanobeam of the hard x-ray scanning microscope based on nanofocusing refractive x-ray lenses. Each of these cylindrical lenses focuses the beam in one dimension. In the microscope two of them are aligned behind each other. Two of the lenses are aligned behind each other in crossed geometry to form a point focus about 13 mm behind the last lens. At an x-ray energy of $E = 15.25$ keV a highly coherent portion of the x-ray beam is collected and a full width at half maximum (FWHM) focus size of $67 \times 83 \text{ nm}^2$ is expected in horizontal and vertical direction, respectively.

As a first step, ptychographic CXDI was applied to a lithographic test structure in order to retrieve the complex-valued wave field of the nanobeam and to verify object reconstruction (cf. fig 1 for reconstruction of test structure). The setup was placed in the focal plane. No beamstop was used. A sub-area of a two-dimensional resolution test chart¹¹ manufactured by NTT-AT was scanned in focus with 126×126 steps and a step size of 40 nm, covering an area of about $5 \times 5 \mu\text{m}^2$. At each position of the scan, a far-field diffraction pattern (without beam stop) was recorded by a single photon counting and noise-free MAXIPIX detector having 256×256 pixels with an area of $55 \times 55 \mu\text{m}^2$, each. The detector was positioned at a distance of 1926mm from the sample. A tube filled

with helium was introduced between the sample and the detector in order to reduce background scattering from air. Fig. 2 a) shows the reconstructed phase of the transmission function of the object with a pixel size of 17.8 nm. Simultaneously to the CDXI phase contrast data set the wave field of the sample we have recorded simultaneously high resolution fluorescence data of the very same part of Tantalum test structure.

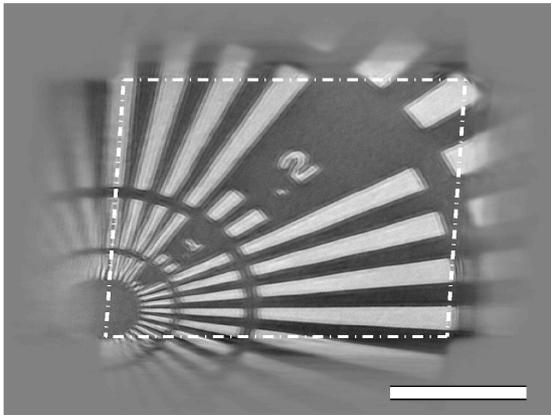


Fig. 1 Phase of the reconstructed transmission function of test structure [2]. Dashed line marks scanned area. Scale bar denotes 2 μm . The complex values illumination function enables a detailed quantitative characterization of the focusing optics, i.e. the wavefront formed by the refractive X-ray silicon lenses [1].

In the next step ptychographic CXDI data sets as well as Fresnel CDI data sets of the biomineralized specimen were collected. Note, that the larger samples were moved to defocus, e.g. the spherulites of *A. willeyana*, in order to enlarge the field of view. Again, the measurements were accompanied by fluorescence scans in parallel. The farfield diffraction data was used to obtain high resolution images of different contrast schemes, e.g. differential phase contrast (DPC) and darkfield (DF) (c.f. fig. 2 *A. willeyana*). Data analysis is still in progress. Along with further improvements, we are convinced that the hard x-ray nanoprobe is able to yield high resolution chemical and structural information on the investigated samples, possibly in 3D after extension to tomography. Wide angle scans to simultaneously assess the crystal structure can also be envisioned. Among all contrast modes, phase contrast is the most challenging, related to the reconstruction problem. In future, reconstruction should be facilitated primarily by detector improvements (beamstop, choice of distance, improved bad pixel and sensitivity corrections) as well as positioning accuracy of the sample to exploit the overlap constraints in ptychographic reconstruction.

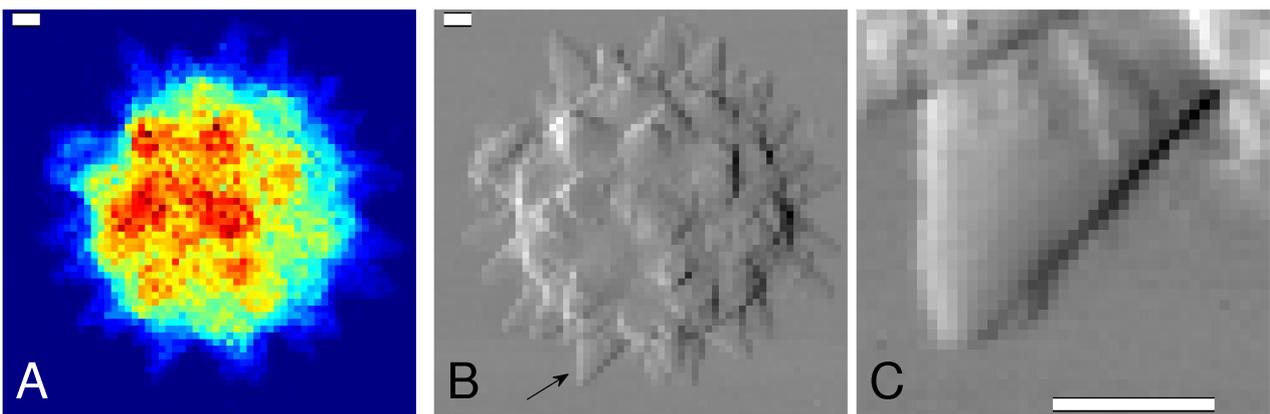


Fig. 2 (A) Fluorescence map (Ca K - radiation) of an aragonitic spherulite of the CaCO_3 skeleton of *A. willeyana* [3]. (B) Differential phase contrast image of the same specimen. (C) DPC image of region within (B) (marked by arrow). Scale bar denotes 2 μm in all images.

- [1] Schroer, C. G. et al. , *Appl. Phys. Lett.* **87**, 124103 (2005).
- [2] Schropp, A. et al. , *Appl. Phys. Lett.*, accepted for publication (2010)
- [3] Jackson, D. J. et al. , *Science*, **316** (2007), 1893.