ESRF	**Experiment title:** Investigating the single crystal nature and domain size of the brittle-star lens array	**Experiment number**: ES 355
Beamline:	**Date of experiment**: from: 18/11/2015 to: 21/11/2015	**Date of report**:
Shifts:	**Local contact(s)**: Dr. Manfred Burghammer	*Received at ESRF:*

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Report:

The processed data was acquired at the ESRF beamline ID13 (nano-focus), during November 2015. The sample is a slice of calcite shell from the Ophiocoma wendtii brittle star, embedded in epoxy resin (Fig. 1). The slice includes the upper array of lenses (which are known to serve for optical purposes and to have a single-crystalline nature, from the work of J. Aizenberg et al. [1]) as well as the underlying "mesh" structure. The purposes of the analysis performed here are:

• To evaluate the quality of the single-crystalline structure of the lenses and of the underlying structure.

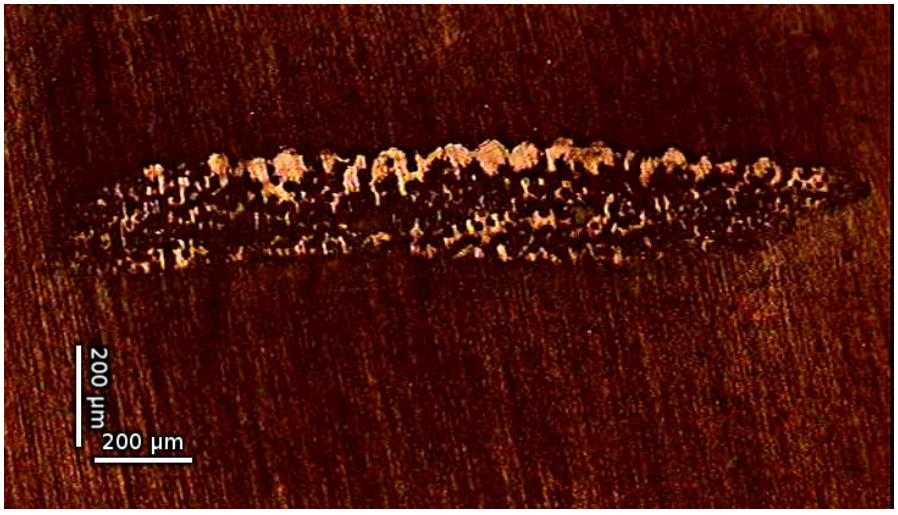

Figure 1.1: Optical micrograph of the mounted sample, in-place for the X-ray scans.

The sample was scanned using an X-ray beam focused roughly to 200 × 150 nm at FWHM. The wavelength used was λ=0.832109 Å. Two calibration measurements were performed on a standard corundum powder sample, one at the beginning of the experiment, and one at the end. Both the calibrations are in excellent agreement with each other (~0.01% difference in subsequent d-spacing calculations), and the calculated effective distance between the sample and the detector was 12.9216 cm. Several measurements were performed.

In figure 3.1 all the observed reflections can be seen. Additionally, it is evident from this image that the single-crystalline structure of the sample is of a high quality – the spots look very clear and are not smeared, although this image is averaged between 10,201 images, from rotations between −10° and 10°, with 0.2° intervals.

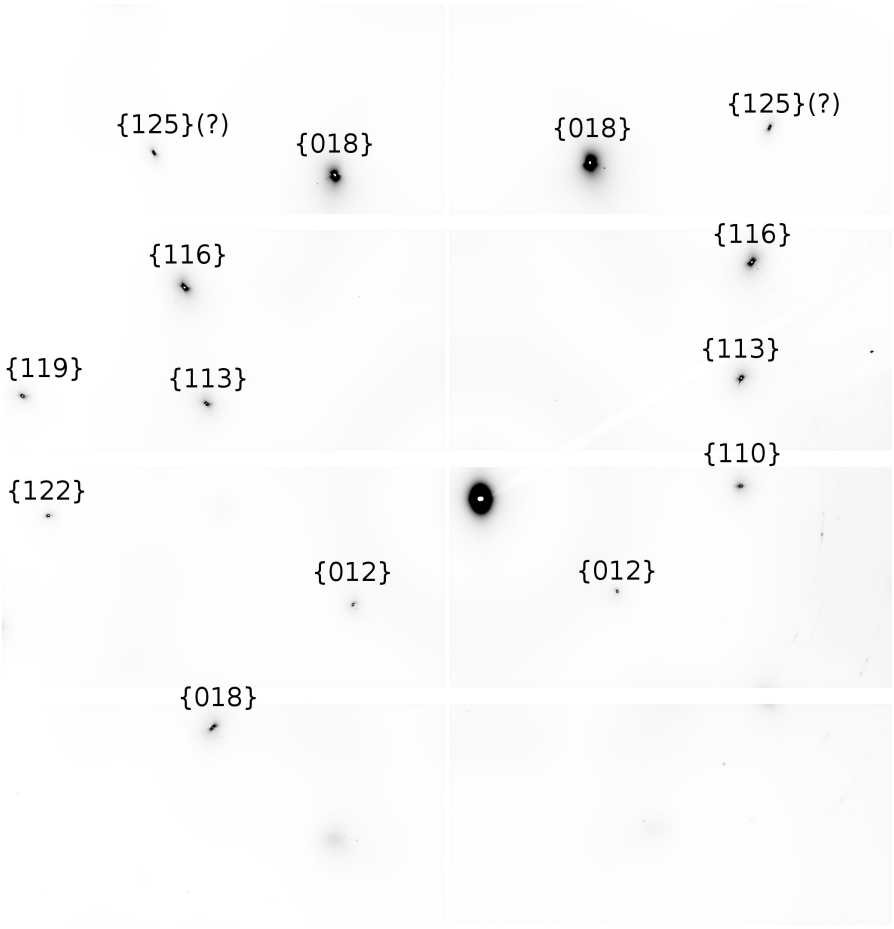

Figure 2: An averaged image from all the studied rotations (from a rotational line scan), with markings of the observed reflections.

Figures 3. is a map of single peaks from the {113}. Each spot location on the map is taken from exactly the same location of the individual diffraction at that area of the sample, and from these figures it is clear that even though some variations in intensity of the peaks occur throughout the sample, the peak location stays practically the same over the entire area.

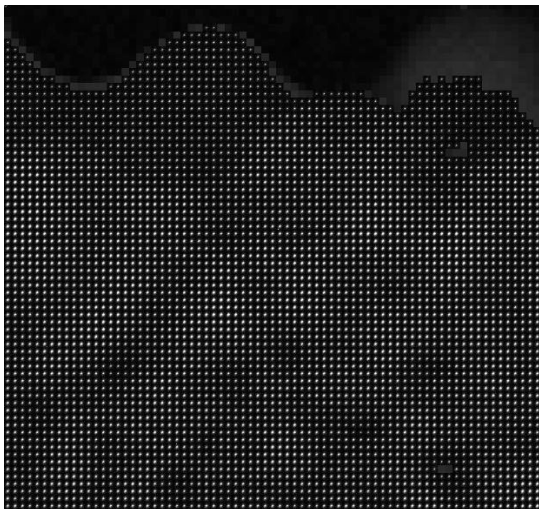

Figure 3: Large area scan, map of (113) plane. Evidently, all the peaks are at the same azimuthal/radial location, although they vary in intensity.

Figures 4.A—F show the rocking curves that were obtained by this rotational scan. Each rocking curve shows the areas between the 10 to 90 and 30 to 70 percentiles, as well as the mean of all the intensities of a specific peak at the various rotation angles. This is done to give a better visual representation of the statistics. Even these rocking curves, taken with 0.5° intervals, have a very sharp profile. But it's still not a match to the rocking curves taken with 0.05° intervals, which will be shown later. There the sharpness is much greater.

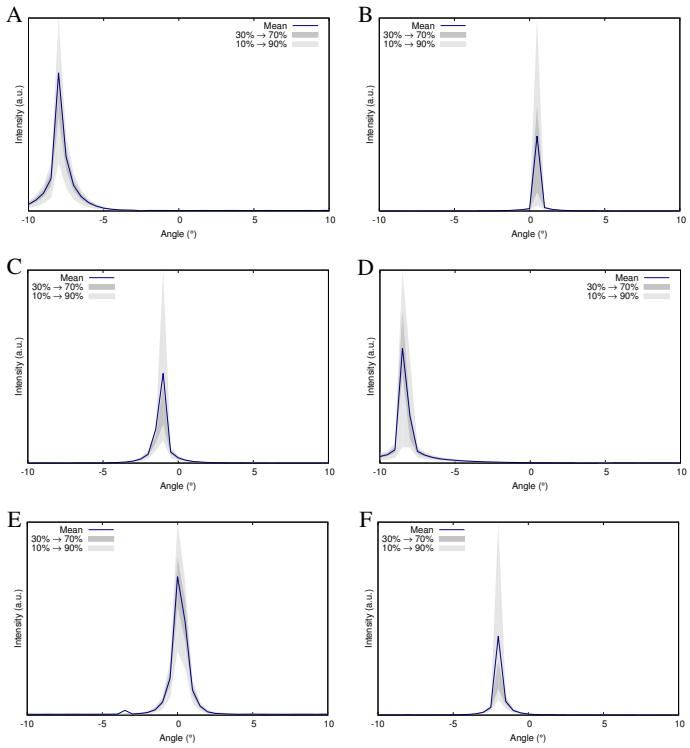

Figure 4: Rocking curves of various reflections from the 0.5°-interval rotational scan. A: (110), B: (113), C: (116), D: (118), E: (119), F: (125).

From these experments it is clear that indeed this complex strucure is a single crystal despite the intricate morphology