



Beamline: ID01	Experiment title: Coherent diffraction from micrometric crystal: phase retrieval	Experiment number: MI689
	Date of experiment: from: 05/07/04 to: 12/07/04	Date of report: 20/07/04
Shifts: 18	Local contact(s): T. H. Metzger	<i>Received at ESRF:</i>
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Report:

It has been recently proven that the phase problem can be solved by over-sampling the diffraction pattern. Although it is well established for optics, such a measurement is difficult to perform with x-rays due to the lack of coherent x-ray sources. Previous coherent x-ray diffraction measurements have been already performed at BNL, ALS and APL synchrotron sources, either in the forward direction for amorphous samples or at a Bragg reflection for micro-crystals. The 3D diffraction pattern can then be used for the inversion in order to reconstruct the full 3D real space density. The phase is retrieved through iterative algorithms and the oversampling leads to the uniqueness of the solution without the need for *a priori* hypothesis.

The aim of the present experiment is to perform at ESRF on ID1 beamline a coherent x-ray diffraction measurement on a micrometric single crystal. We used gold crystals obtained by the dewetting of a thin gold film (100 nm thick). Electron scanning microscopy investigation of the sample shows well faceted isolated particles, that size varies between 0.5 and 2 μm (Fig. 1). The (111) directions of the particles are mostly along the perpendicular to the substrate surface. A non focused monochromatic beam of 8.2 keV is used. The (111) powder ring measured with a large Princeton CCD located at 60 cm after the sample exhibits some intense spots which come from the single Au particles. Once a crystal is chosen, a pair of slits placed at 37 cm in front of the sample are used to define a $40 \times 40 \mu\text{m}^2$ area in order to illuminate a small amount of particles. As the crystals are slightly disoriented one to the other, only the chosen particle exhibits a diffraction pattern in the 3D space we probe.

The full diffraction pattern is measured by scanning the theta angle and recorded with a direct illumination CCD (384×576 pixels of $22.5 \times 22.5 \mu\text{m}^2$ size each) placed at 122 cm from the sample.

The Fig. 2(a) shows the diffraction pattern measured at the center of the Bragg spot. This picture results from the accumulation of 200 frames of 0.8 s each. A 0.5 mm diameter beamstop is used to hide the intense central part of the pattern and to avoid the use of filters. (The central part itself is measured later by reducing the incoming intensity.) The streaks are coming from the facets and the large number of interference fringes, due to finite size effects, show the good coherence property of the beam. From the periodicity of the fringes, a particle size of about $1.5 \mu\text{m}$ can be deduced. In absence of strain, a centro-symmetric 3D pattern is expected. This property is fairly visible on the Fig. 2(a). The Fig. 2(b) and (c) present the diffraction patterns measured at $\pm 0.08^\circ$ from the Bragg maximum (resulting from the accumulation of 1000 frames of 1.8 s each). These patterns are highly centro-symmetric one to the other.

The full 3D pattern is recorded and will now be used to retrieve the 3D real space shape of the Au crystal.

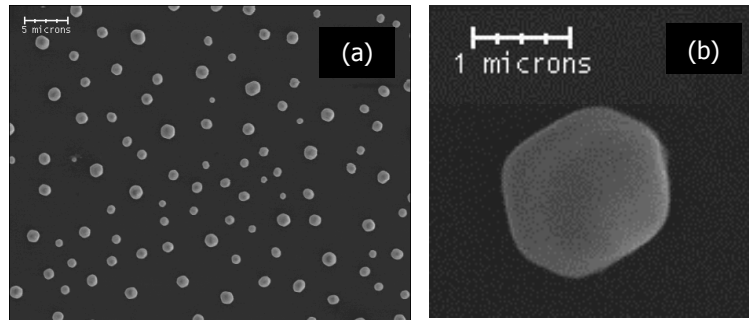


Figure 1: Scanning electron microscopy investigation of (a) the Au crystals and (b) view on a typical single crystal.

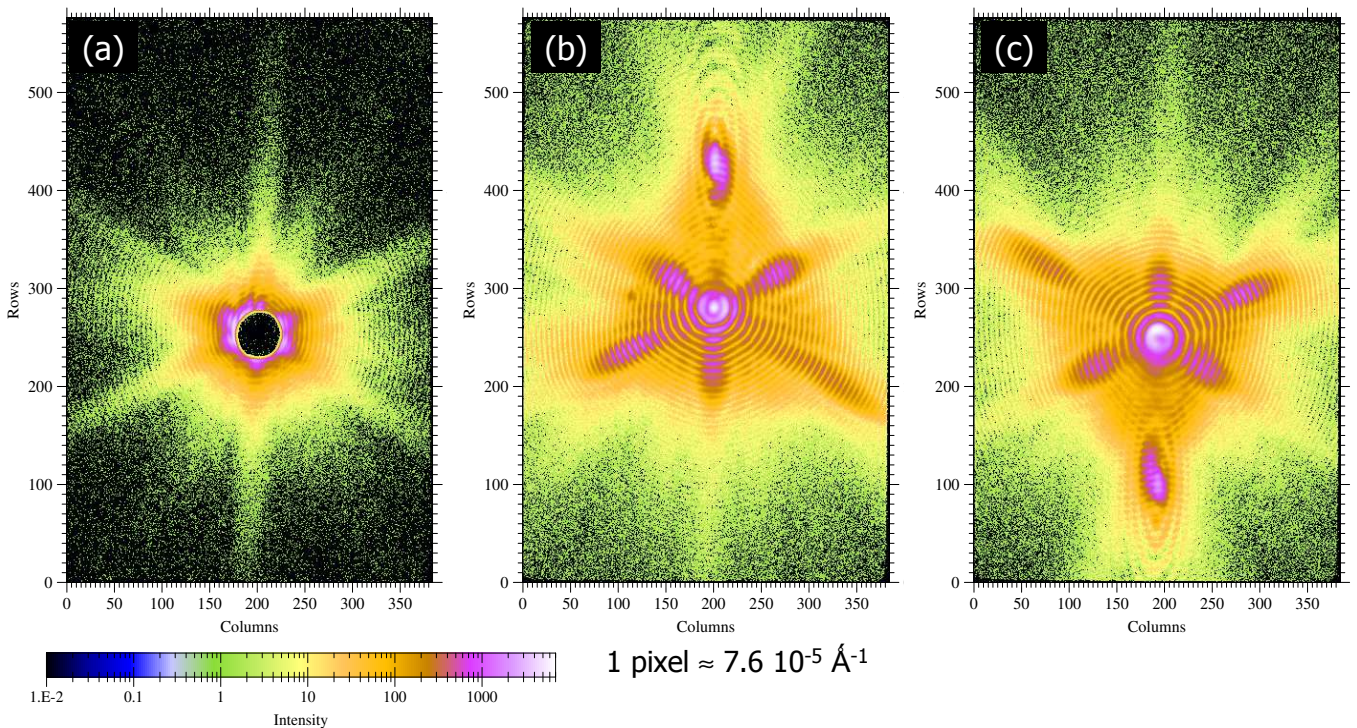


Figure 2: 2D coherent x-ray diffraction patterns measured on a single Au crystal (a) in the center of the Bragg spot and (b) -0.08° and (b) $+0.08^\circ$ from the Bragg maximum.