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Report:
It has been proven that the phase problem can be solved by over-sampling the diffraction pattern. Although it is well established for optics, the 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 performed at BNL, ALS and APL synchrotron sources (for review article see [1]). 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 with iterative algorithms and the oversampling leads to the uniqueness of the solution without the need for *a priori* hypothesis. The technique is particularly promising to investigate strains in single crystal particles with typical size in the nanometer range. The limiting factor becomes the flux at the sample position and no more the coherence length. In ref [2], the possibility to measure the coherent diffraction pattern using a focusing element has been demonstrated on a 160 nm Ag particle.
The aim of the present experiment was to investigate the strains in a single nanoparticle using inversion algorithms based on coherent x-ray diffraction measurements. A 9.5 keV beam was focused on a 8×3 μm^2 spot in the center of the diffractometer using 31 Be Compound Refractive Lenses (CRL) located 0.9 m upstream. The integrated intensity was evaluated to $2 \cdot 10^9$ ph.s^{-1}. A beamstop on the direct beam was placed a few cm after the sample, followed by a pair of anti-scattering slits. The acquisition was performed with a direct illumination CCD (384×576 pixels of 22.5×22.5 μm^2 size each) mounted at 30 cm from the sample. A droplet of ethanol containing the powder sample in solution was deposited on a support.This allowed for a surface density of a few particles per μm^2.

For measurement in transmission geometry, the support was a Cu electron microscopy grid recovered by a C replica (with holes of 100 μm size), while for reflection geometry a SiO_2/Si substrate (also recovered by a C thin film) was used. The later one was leading to a reduced background and allowed for a good evacuation of electrostatic charges (proven by the good stability of the sample in the beam). Three kinds of samples have been investigated : (*i*) ZrC crystals (figure 1(a)), (*ii*) ZrC crystals embedded in C (named ZrC-C) and (*iii*) ZrO_2 crystals. Due to the small size of the particles (ZrO_2 and ZrC-C are about 30 nm and 100 nm, respectively), to their tendency to form agglomerates and to the large background produced by the C shell (in case of ZrC-C), the experiment was difficult to perform for the two later samples and did not lead to a large enough signal to background ratio. However satisfying measurements have been obtained on the ZrC crystals. The 2D coherent diffraction pattern obtained on the (111) reflection is shown on figure 1(b). It results from the accumulation of 2000 frames with a 2 s exposure time. Each frame is analyzed by the droplet algorithm to recover the single photon signal [3]. The intensity exhibits a dynamics of about 1000. The five interference fringes result from the crystal finite size effects ($\approx$ 120 nm). The 2D inversion procedure was performed during the experiment. In order to take into account the possibility of strains, the object is defined by a complex function, whose amplitude is the electron density and whose phase is related to the displacement field (the codes were previously checked on numerical complex test objects). The 2D inversion of the ZrC (111) reflection showed good convergence properties and successfully led to the real space electron density (projected onto a plane inclined by a few degrees from the (111) direction). The obtained displacement field map shows no indication of strains. The 3D diffraction pattern was also measured and will be used for the 3D inversion.

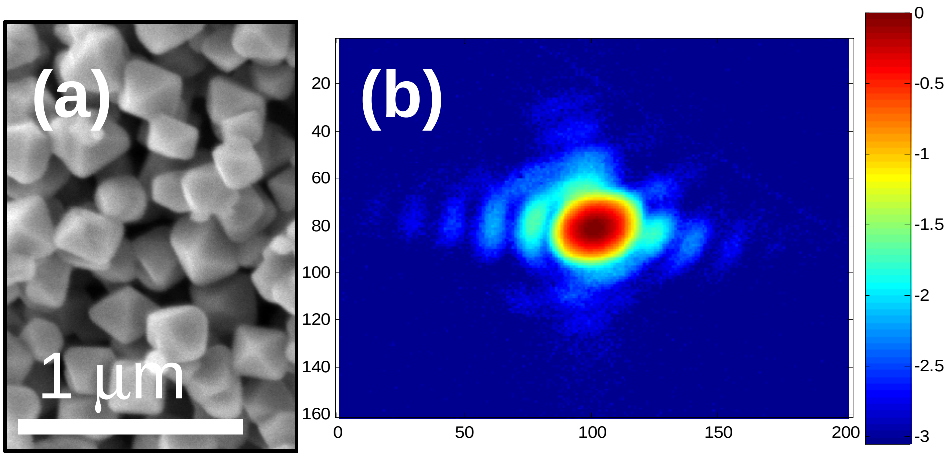

Figure 1: (a) Scanning electron microscopy investigation of the ZrC nanocrystals and (b) coherent diffraction pattern measured on a 120 nm ZrC crystal at the (111) Bragg reflexion.

References

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[2] I. K. Robinson *et al.*, Optics Express **11**, 2329 (2003).
[3] F. Livet, *et al.* Nucl. Instr. Meth. A **451**, 596 (2000).