MI-1254 experimental report

Proposal summary:

This proposal was a proof-of-principle experiment to demonstrate a setup capable to record multiple projections of an object in a single-shot manner with coherent diffraction imaging (CDI). The different projections are obtained by exciting simultaneously different Bragg reflections of a perfect thin crystal. Implementations of this concept are foreseen at X-ray free electron lasers (FEL) to access for the first time high resolution three-dimensional information from a single shot acquisition, adding the third dimension to the diffract-before-destruction mode [1].

Experimental methodology and results:

Following the requirement specified in our proposal [2], the energy of ID01 was set to 12.57 keV tuning the ondulators gap and using a double crystal monochromator. After the energy of the beamline was tuned to 12.57 keV, a Fresnel zone plate (FZP) was mounted to focus the beam. The FZP was aligned with the incoming beam, producing a vertical focal spot of 100 nm fwhm as depicted in Fig. 1, because in the vertical direction the whole FZP can be coherently illuminated, that is not the case for the horizontal direction. Therefore, to have a fully coherent illumination we horizontally slitted down the beam producing a larger focus in that direction. During the experiment the FZP was illuminated in two different modes, on one hand we used an asymmetric-horizontal illumination of the FZP, i.e., illuminating horizontally only the left side of the FZP to have a tighter and asymmetric focus; on the other hand, we used a symmetric-horizontal illumination of the FZP, obtaining a symmetric and less tight focus compared to the aforementioned scenario. For the final results presented below, the symmetric illumination was used.

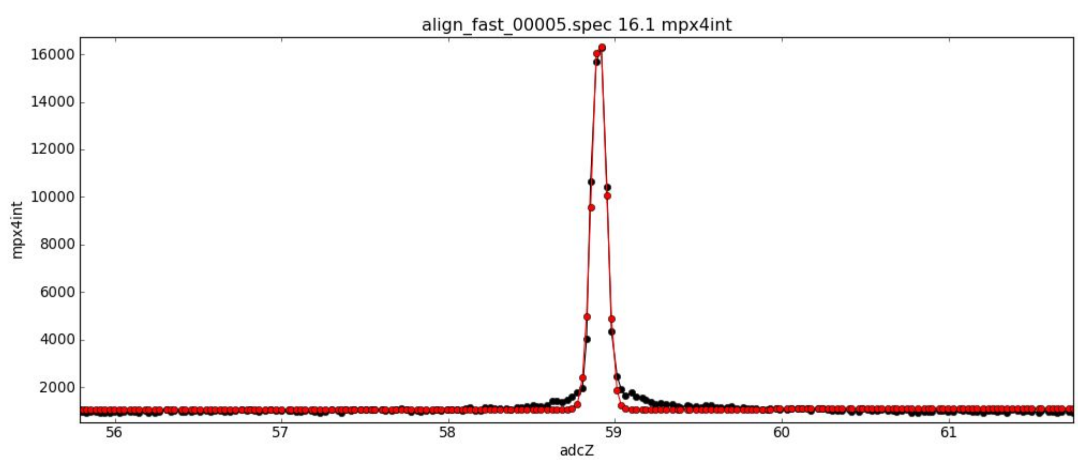

Fig. 1: Final alignment scan of the Fresnel zone plate. The obtained vertical focus was of 100 nm fwhm at 12.57 keV.

Once the focal spot was well defined, the ID01 diffractometer center of rotation was set to the focal spot, using a calibrated sample (silicon wedge), with an uncertainty smaller than the depth-of-focus. In order to characterize the illumination, ptychography scans were performed using a spiral scan on a Siemens star (well known object). In order to reduce the air scattering a 2 m flight tube was installed after the sample for this scan as our detector was positioned at 2.3 meters from the sample.

Once the beamline was setup to the required conditions for our experiment, we installed our sample [2]. The sample was a sandwich composed by a first layer of a perfect 100 μm thick Si crystal with an out-of-plane 100 and a second layer with 200 nm gold nanostructures. This Si crystal can generate 8 diffracted beams as depicted in Fig. 2 (a), when the out-of-plane direction matches the optical axis. The designed gold nanostructures were three-fold inverted mushrooms, as shown in Fig. 2 (b).

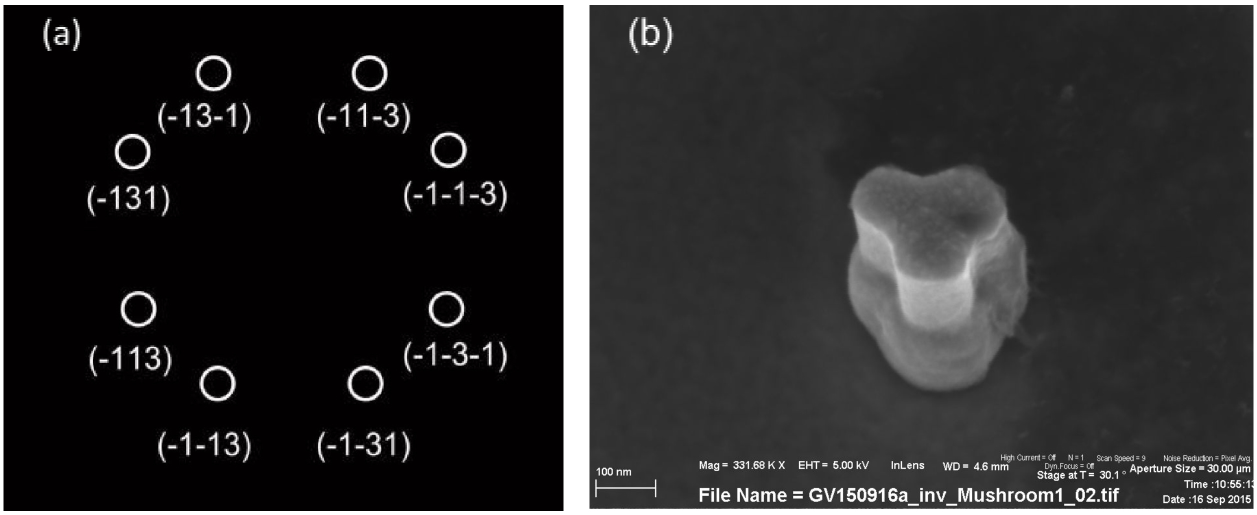

Fig. 2 (a) Family of planes in Laue condition for a Si crystal with the out of plane and the incident beam in the (100) orientation, at 12.57 keV. (b) Three-fold gold nanostructure illuminated by the 8 deflected beams and the direct beam.

With the sample mounted, our first goal is to align the crystal to obtain different deflected beams. The initial strategy was to observe the different deflected beams on a phosphor screen with an optical camera. Unfortunately the intensity we got from the phosphor screen was too low to observe the deflected beams on the camera. Therefore, we used the Maxipix mounted really close to sample (85 pixels per degree). In order to avoid any damage on the detector we attenuated the direct beam using a beamviewer. The positioning of the detector respect to the sample can be observed in Fig. 3 (a). The intensity of the deflected beams was so low compared to the intensity reported in the proposal (material science beamline at SLS), because for the ID01 experiment we were using a high numerical aperture optics to focus the beam. This implies that the divergence is larger than the experiment at material science beamline, where we were just collimating the beam using mirrors. The perfect 100 μm thick Si crystal, used in both experiments, it only deflects a portion of the photons which travel in the out-of-plane crystal direction. Therefore, a small fraction of the photons were travelling in this direction compared to the almost total quantity of the photons in the material science experiment, due to the use of high-numerical focusing optics in the ID01 experiment. Furthermore, we were using a double crystal monochromator during our experiments at ID01, which made the alignment of the crystal more difficult, because neglecting dynamical effects and using Bragg's law it can be observed that the angular acceptance of the crystal is proportional to the bandwidth. Taking into account the intensity reduction due to the focusing optics and the required alignment precision given by the monochromatic beam, we required around a shift to align the crystal. Two deflected beams were observed on the Maxipix detector, as depicted in Fig. 3 (b).

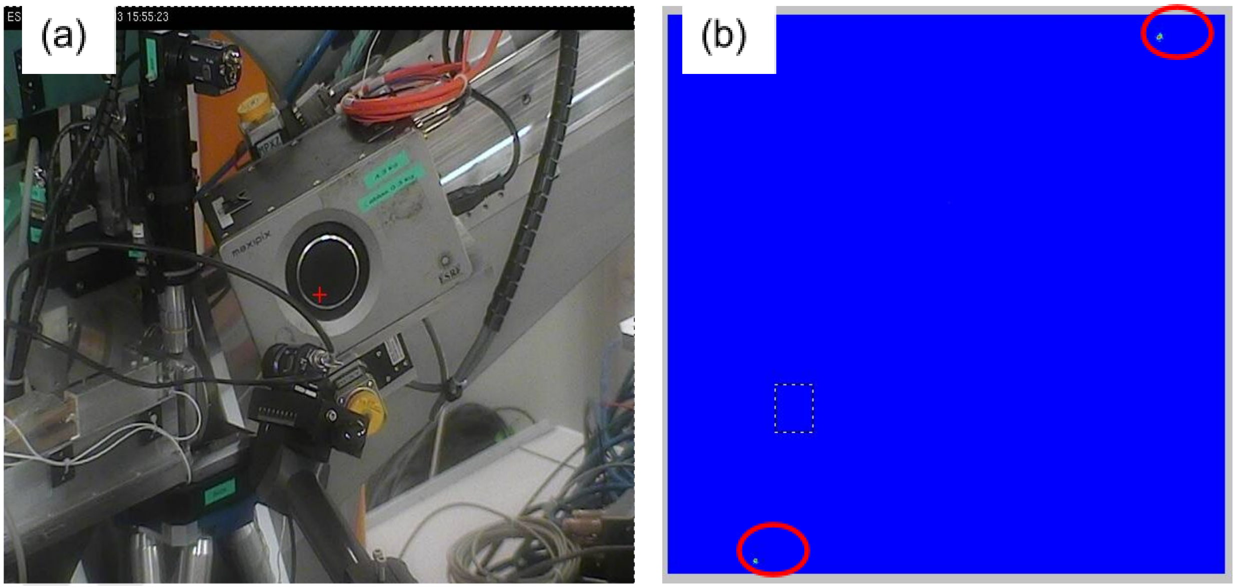

Fig. 3 (a) Positioning of the Maxipix detector close to the sample to align the crystal to generate different deflected beams. (b) Two peaks observed in the Maxipix detector with similar intensity after the alignment of the crystal

After aligning the crystal, we perform our imaging experiments. First, we cleaned the beam in the direct direction (non-deflected beam). To clean the beam to perform CDI, we removed all the filters and control elements in the direct beam direction, changed damaged kapton foil windows, cleaned the order selecting aperture (OSA) of the fresnel zone plate, and installed the 1 m flight tube after the sample. The sample to detector distance for the final experiment was 1.22 m, to match the oversampling criterion required by CDI, and the Maxipix was the used detector. As aforementioned, we illuminated the FZP symmetrically in our imaging experiments. The diffraction patters of our object with the Si crystal in the direct beam are shown in Fig. 4 (a). The CDI reconstructed object, using a modified version of the reconstruction code developed by Y. Chushkin for ID10 at ESRF, is depicted in Fig. 4 (b). The resolution of the reconstructed object is 15 nm evaluated using phase-retrieval transfer function [3].

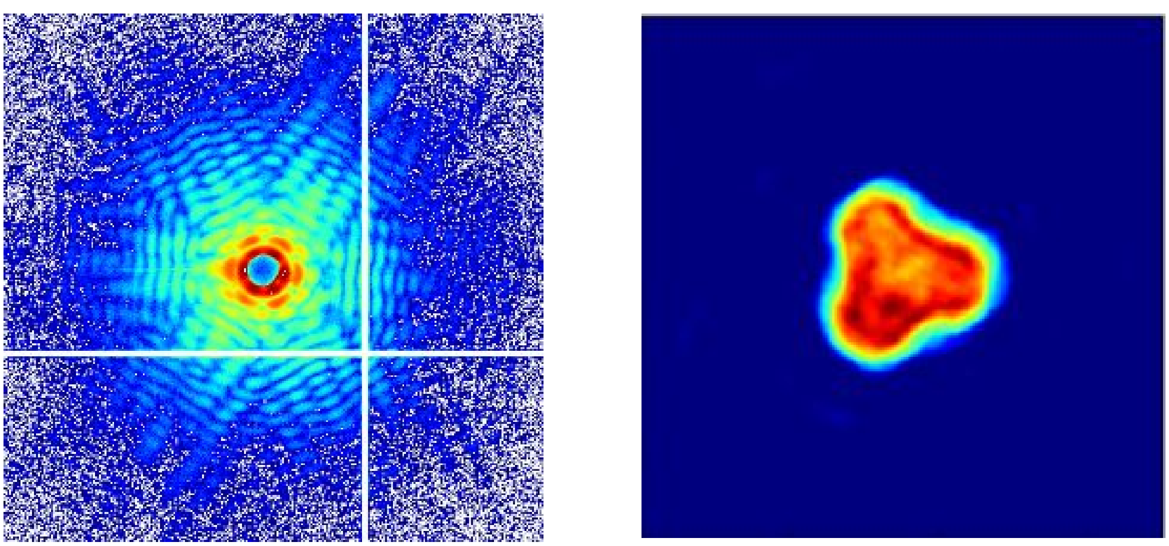

Fig. 4 (a) Diffraction patter recorded after cleaning the beam. (b) CDI reconstructed sample with a resolution of 15 nm, it correspond to the sample depicted in Fig. 2 (b)

Unfortunately on the deflected beams, we could not perform imaging because the deflected intensity was not enough to acquire images with a reasonable exposure time and the focused beam was not big enough to illuminate the sample simultaneously by all the deflected beams, thus a realignment of the sample was required.

Discussion of the results and future work

During our beamtime, we managed to perform CDI imaging experiments. It was the first time CDI was performed on the direct beam at ID01, therefore several tasks were done to clean the beam to achieve this goal. The object was reconstructed with a resolution of 15 nm after exposing for 200 s. Unfortunately, we did not succeed to perform CDI in the deflected beams by the perfect Si crystal. In order to succeed with this last goal several future modifications to the setup are required:

1) Bigger samples to increase the number of diffracted photons (around 1.3 μm to fulfill the oversampling criterion and have an efficient focus).
2) Use more efficient optics with smaller numerical aperture to increase the number of photons on the deflected beams, e.g. compound refractive lenses. Unfortunately, during the experiment we did not have available CRLs at ID01 with the required parameters.
3) Use pink beam instead of double crystal monochromator, e.g., using a multilayer monochromator. Unfortunately, this was not possible as the multilayer monochromators of the beamline were not efficient for the requested energy.

References:

[1] R. Neutze et al., Nature 406, 752-757 (2000).

[2] P. Villanueva-Perez, B. Pedrini, R. Mokso, and M. Stampanoni, proposal mi-1254.

[3] H. N. Chapman, et al, JOSA A, vol. 23, no. 5, 1179–1200, 2006