EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** High-resolution monitoring of crystal deformation with X-ray diffuse multiple scattering	**Experiment number:** mi1157
Beamline: ID01	**Date of experiment:** from: 13.12.2013 to: 17.12.2013	**Date of report:** 20.12.2013
Shifts: 15	**Local contact(s):** Gilbert Chahine	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The goal of the experiment was to use a recently discovered diffraction signal to monitor the strain in sub-micron crystals as they are mechanically loaded *in situ* by a miniature indenter.

Recently, thanks to the availability of intense x-ray beams and low noise area detectors, we observed an unreported diffraction signal: “Kossel-like” lines were observed, but unlike Kossel lines their origin is not fluorescence but diffuse elastic scattering, hence the proposed name of 'Diffuse Multiple Scattering' lines [Nisbet *et al*, in preparation]. Their advantage over Kossel lines is that they can be observed at energies far for absorption edges of the crystal. On the other hand they are weaker, but since they can be measured at any energy, fluorescence background can be avoided and thus low noise can be achieved with a little care. Like Kossel lines, they do not require to sit on a Bragg position, and they are very sensitive to the lattice parameters, providing an interesting way to monitor strain. These lines had been so far observed only from (many) bulk crystals with full beam (Diamond I16) and from a bulk copper single crystal with KB-focused beam (ESRF ID03).

One of the achievements of this beamtime is the observation of DMS lines using Fresnel-Zone-Plate (FZP) focusing: this focusing mode, while achieving very small beam size (1.3x0.6 μm² hxv in this experiment) is a photon hungry method due to the rejection of a large part of the beam by the 300-μm diameter FZP and to the low efficiency of the FZP with respect to KB mirrors. Moreover the experiment was carried in 16-bunch mode (lower intensity). We had to reduce the noise as much as possible to observe the lines, which required a few shifts of optimisation. Figure 1 shows a couple of examples of these lines, as observed in the Maxipix detector. The intensity without focusing was found comparable to that measured at Diamond I16, which is well optimised for these measurements, but the background noise was here higher. The current state of the Maxipix detector of ID01, which has many damaged pixels and high noise on 2 chips out of 4, did not help: this higher background turned to be a major difficulty when using the FZP, due to the lower intensity of the beam.

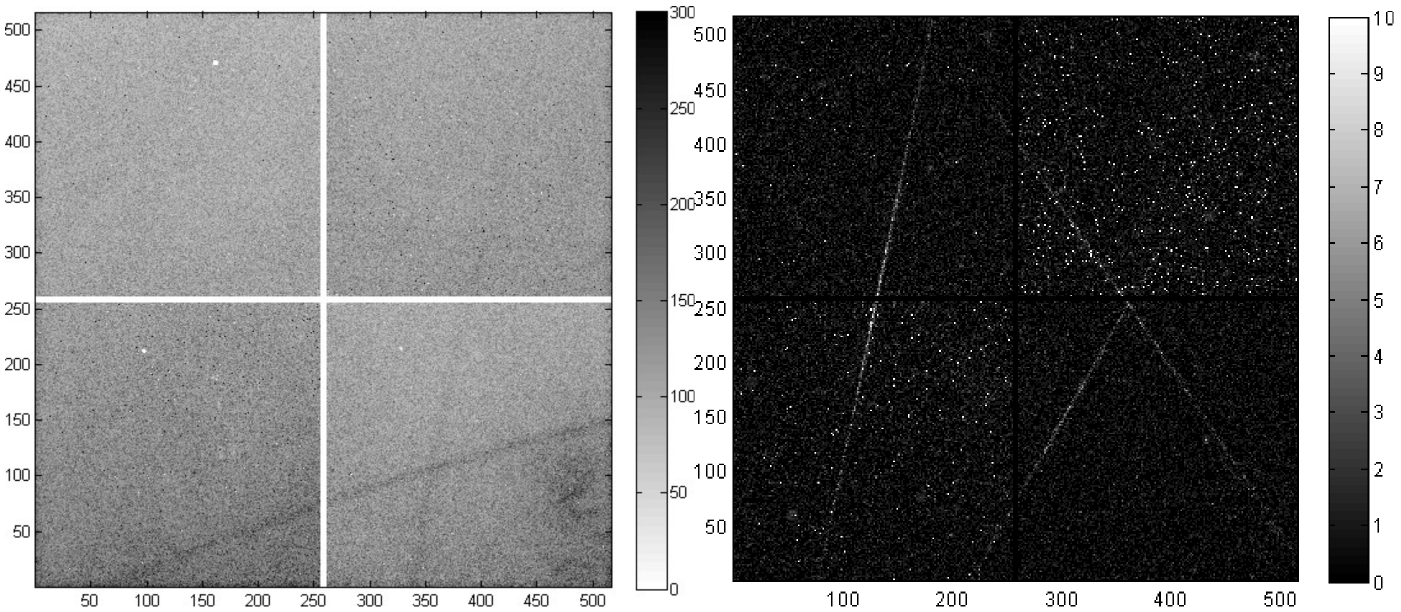

Figure 1: DMS lines observed with full unfocused beam from a large gold single crystal in 1000 seconds (left, 2 visible lines), and with FZP focusing from a large Si single crystal in 600 seconds (right, 3 visible lines). Note the difference of sharpness, due to the difference of mosaic spread (moreover the sample-detector distance was roughly doubled on the right image).

But for the proposed experiment we needed to measure DMS lines from sub-micron crystals (we chose gold crystals to maximise the scattering cross-section) and we did not manage to observe them. We identified 3 reasons for this: 1) reduced flux due to FZP focusing and 16-bunch mode 2) background noise not low enough 3) scattering volume: while the focused beam was smaller than the crystals, the penetration depth was much bigger than the crystals height (~0.5 µm): 50% of the beam was transmitted across the crystal for the 111 reflection at 8.5 keV. We still think that it should be possible to see these lines from such gold crystals, possibly at lower energy, with KB mirrors focusing instead of FZP for higher efficiency, and with a lower background set-up.

Another aspect of the experiment was to use a nano-indenter to apply load *in situ* and measure force-displacement curves while monitoring DMS lines which are thought very sensitive to strain. Our instrument was installed on the diffractometer of ID01 (Figure 2). The noise on the diffractometer was found very high: ~300 nm peak-to-peak. For comparison, it was ~80 nm in ID01 preparation lab, and ~20 nm in our lab. A frequency analysis reveals a particular low frequency at 0.86 Hz, which may be attributed to the closed loop of one of the diffractometer axes or its hexapod stage. Such high noise would be a critical issue when trying to measure the first stages of plasticity, for instance with a coherent beam. Moreover, it prevented us from measuring the elastic regime from metal samples.
The tip was in tungsten carbide, with an end-radius of 5 µm: the contact area on our gold microcrystals, which have a top facet of a few 100 nm, can be approximated as a flat punch.

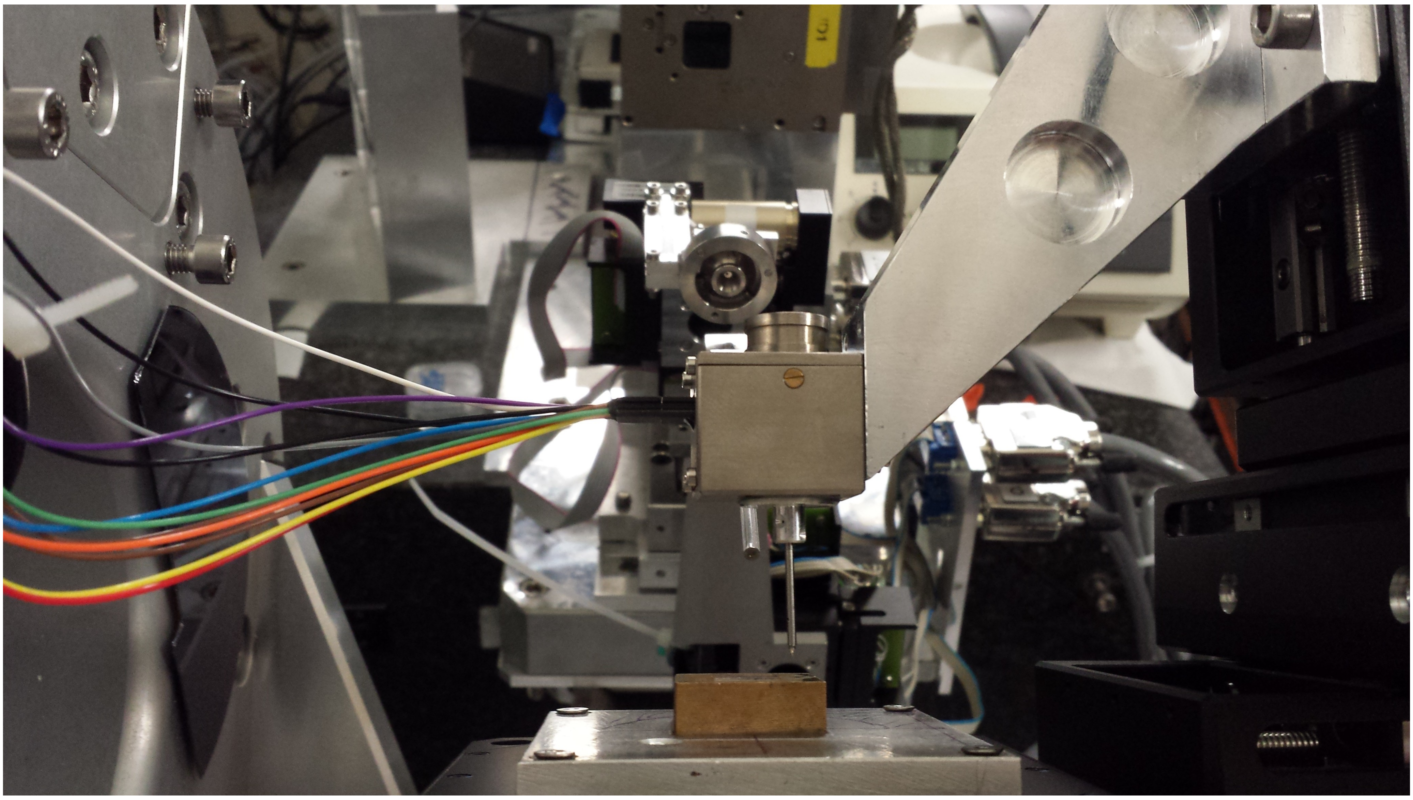

Figure 2: Front view of the set-up with the nano-indenter. From top to bottom: slits, FZP, nano-indenter head, order-sorting aperture of the FZP, nano-indenter tip, sample, tungsten block for height adjustment.

As stated above, we could not measure DMS lines from individual microcrystals, due the weakness of the signal, so we measured the 111 reflection instead. With the help of ID01 staff, we implemented a fast mapping procedure to scan the indenter stepper motors in a similar way to the fast mapping of the diffractometer piezo stage. The tip was scanned above the surface in diffraction geometry: its 2 shadows (when blocking respectively the incident and the diffracted beam) give a precise determination of its position (Figure 3). Using both fast mapping routines, we were able to bring the tip and the microcrystal of interest together in the beam in a few tens of minutes.

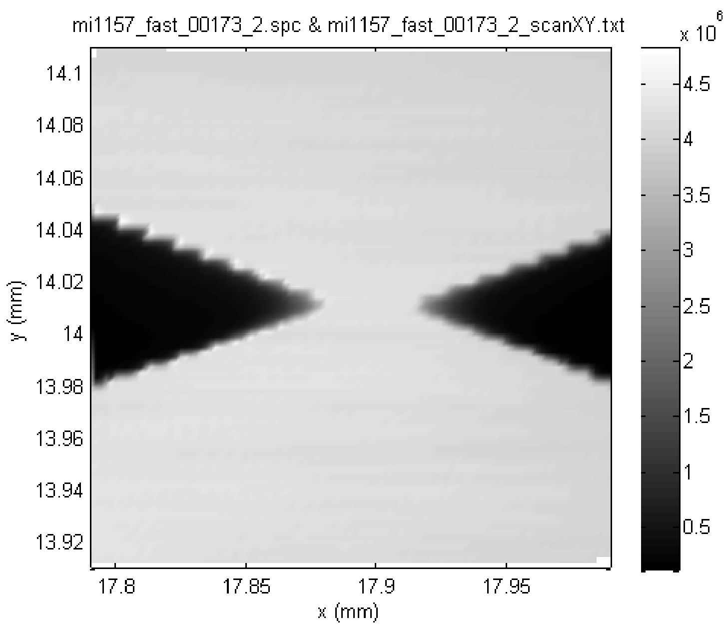

Figure 3: Fast scan of the nano-indenter tip in diffraction geometry. The position of the beam is at the middle point between the 2 shadows.

The microcrystal was then loaded with controlled force, and the intensity was recorded on the maxipix detector together with the force and the displacement of the tip (Figure 4). This is to our knowledge the first time that a force- and height-controlled indenter has been used *in situ* in diffraction geometry. However the high vibration noise of the diffractometer recorded on the height signal did not allow to record meaningful force-height curves.

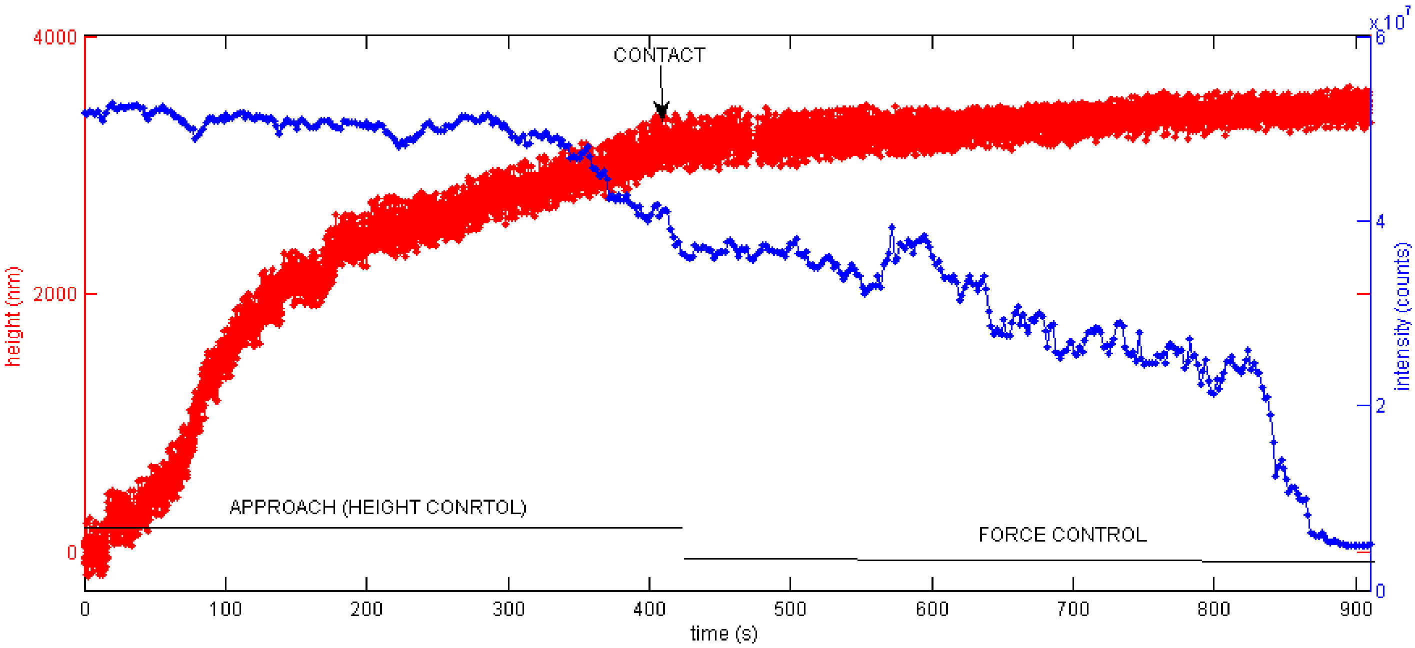

Figure 4: Indentation test of a gold microcrystal. The approach is controlled in height (1st part of the curve), and the indentation itself is controlled in force (2nd part of the curve). Both are recorded at all time together with the intensity on the detector.

At the end of the test presented here, the crystal is broken in pieces: a much broader rocking curve is observed after the test, and the top intensity has dropped enough to make the crystal disappear on a diffraction map (Figure 5).

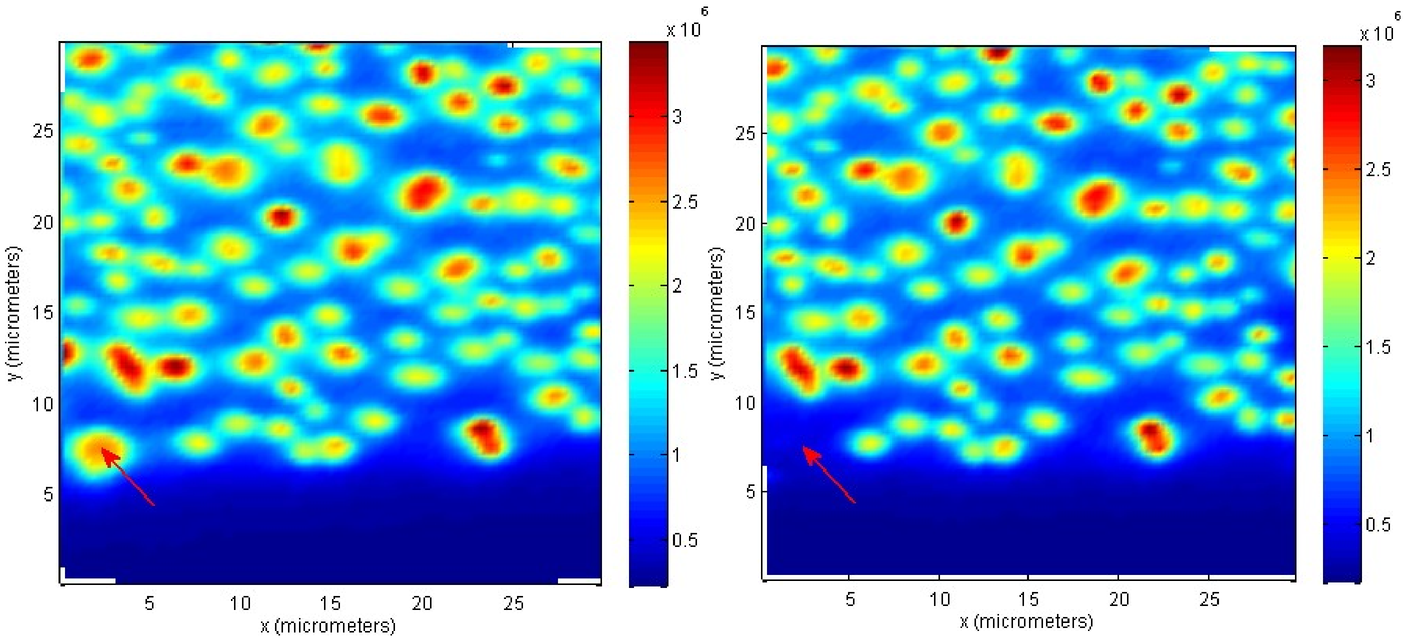

Figure 5: Diffraction maps of the gold microcrystals before (left) and after (right) indenting one of them.

To conclude, the experiment yielded several positive results: 1) observation of DMS lines for the first time on ID01, and with FZP focused beam for the first time anywhere; 2) implementation of a nano-indenter controlled in force and height and successful indentation of a microcrystal. On the other hand, there are also negative results: 1) we could not measure DMS lines from microcrystals because of the lack of intensity and the background noise; 2) we could not measure accurate force-displacement curves because of the high vibration noise of the diffractometer axes/stage. Hopefully these drawbacks will be improved on the new ID01 beamline, where we plan to continue developing such experiments.