ESRF	**Experiment title:** **X-ray Microfocusing - application to the study of micron-sized objects (part I - microfocus setup of the focusing device)**	**Experiment number**: BLC-2175
Beamline: ID-01	**Date of experiment**: from: 07 June 2005 to: 12 June 2005	**Date of report**: 23/08/2005
Shifts: 15	**Local contact(s)**: C.Mocuta	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Cristian MOCUTA (*), ESRF
Dr. Baerbel KRAUSE (*), ESRF
Dr. Till METZGER, ESRF

Report:

A microdiffraction setup was implemented and tested on ID-01, proving the great potential of this technique and its use in the study of small-sized objects. In contrast to *imaging* experiments, this technique combines diffraction **and** small size beams, thus giving access to the **crystalline structure** of the objects. Moreover, very different space constraints apply in diffraction, as the sample has to be moved independently of the focusing setup, apart scanning its position, around at least two rotation axes.
During this report we will mostly concentrate on the setup aspect and the technical issues, showing the results of the various tests which were carried out. The results obtained using this setup are described in more details in the experimental reports IH-HC-766 and MI-744.

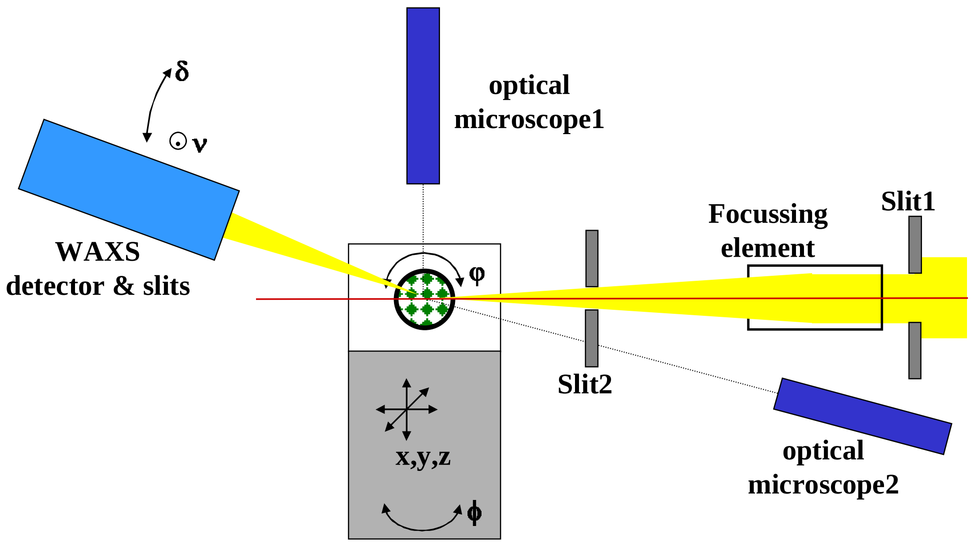

Figure 1. Schematic setup used for microdiffraction experiments. The sample is mounted on a motorized goniometer head allowing for its alignment. Slit1 and Slit2 act as entrance- (acceptance-) and cleaning- apertures respectively.

Either Be compound refractive lenses (CRL) or circular Fresnel Zone Plates (FZP) were used as focussing elements, yielding a similar result concerning the focused spot. Note that the use of FZP implies placing an aperture slit2 to clean the higher order focus, as close to the sample as possible. Figure 1 describes the setup using circular FZP.
The measured focused spot size at an energy of 10 keV was 3×5 μm^2(vertical × horizontal). A tungsten wire (15µm in diameter) was used to scan through the beam (vertical and horizontal directions – these data were also used to measure the size of the focused beam) to localize the position of the focal spot. This position is referenced by two optical microscopes placed in the vertical and horizontal plane respectively. The reference

position is then used for the prealignment of the sample – the single object of interest is brought as close as possible to the position of the focussed beam. Theoretically, the ID01 source size should be ~25×135 µm^2 (v × h), but the measured one (apparent source size) was ~200×200 µm^2. Several reasons explain this increase in size (especially in the vertical direction). It was found that the monochromator crystal shows high amplitude vibrations (~100 Hz) in the vertical plane [*Note1*], yielding thus an artificial spread of the source in this direction. For the horizontal direction, the existence of a pre-bended sagital focusing creates not only distortions of the wave-front, but also a virtual source at a different position. Consequently, the best focused spot obtained with the optical element (demagnification factor D~75) was ~3x5µm^2. The gain of the beam intensity inside the focused spot was measured, and found in agreement with the calculated value.

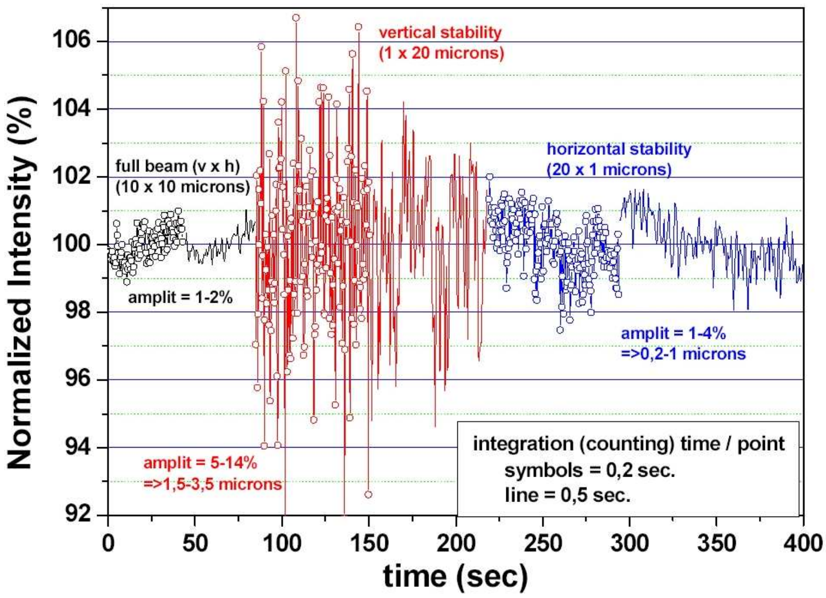

Figure 2. Intensity in the focused direct beam measured after a rectangular slit (values reported on the figure). The amplitude of intensity variation is translated into amplitude of vibration using the measured beam profile.

Figure 2 shows an evidence of the vibrations to occur mostly in the vertical direction. A rectangular slit was placed at the focused beam position, either vertically or horizontally. The intensity variation of the direct beam after the slit is showing higher amplitudes when it is horizontal, corresponding to higher amplitudes of vibrations in the vertical plane. Using the measured intensity beam profile(s), these intensity variations are translated in movement amplitudes (1-3 microns vertically and up to 1 micron horizontally). These values are rather similar if Be CRLs are used, and they are in agreement with values measured using mechanical vibration tests on the sample stage: up to 1.5 microns vertically and 0.5 microns horizontally.
These results show, if microdiffraction experiments are to be done on ID-01, the need of:

- A better and more stable monochromator
- A better and dedicated diffraction stage (diffractometer)

Considering the spreading of the beam, it was still possible to perform experiments on single isolated objects. The constraint was to have objects well separated (distance between objects bigger than the size of the beam). The first tests were performed on patterned arrays of Nb dots (spacinngs and sizes from 10 to 1 microns). The sphere of confusion of the diffractometer is rather big (50µm), the sample (single object) can be pre-aligned within 20µm. Considering that a reciprocal space map typically involves rotations of the angles of 1-2^o, this translates into sample movements of at most 1µm. Thus a beam of a size of few microns has, in this case, the advantage of still illuminating the single object during the whole measurement (see exp. Report MI-744).
We noted the high mechanical stability (within a micron) of both the sample and optical elements stages. We also evidenced a strong sensitivity of the experiment to machine instabilities and refills. For the moment, the only mode in which these experiments seem to be feasible is the uniform mode (eventually 2/3 filling).

Using the above described setup, two experiments were performed on ID-01: the study of single GaAs/InGaAs rolled nanotubes (IH-HC-766) and the study of individual Ge pyramids on Si(100) (MI-744).

[*Note1*] The vibrations were evidenced as 50% intensity fluctuations measured on the 3rd harmonic (no mirrors, E_3=24 keV) of the direct beam. The angular amplitude of vibrations was estimated to the corresponding Darwin width (~3µrad). This value is in good agreement with the measured movement of the center of mass of the beam using a fluorescence-screen CCD.