



	<b>Experiment title:</b> X-ray microscopy and microdiffraction experiments with X-ray waveguides (Long Term Project 2001/I - 2002/II)	<b>Experiment number:</b> MI490
<b>Beamline:</b> BM5	<b>Date of experiment:</b> from: 06/02/2002 to: 06/02/2002	<b>Date of report:</b> 28/2/2003  <i>Received at ESRF:</i>
<b>Shifts:</b> 15	<b>Local contact(s):</b> T. Bigault	
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## Report:

The run was part of a Long Term Proposal involving experiments at ID13, ID32 and BM5. The sessions at BM5 have been mainly used to assess improved experimental procedures for x-ray microdiffraction experiments aimed at measuring local deformation in perfect crystals. We recall here that we used the properties of the waveguide to obtain high spatial resolution in the vertical plane, while maintaining the incident beam characteristics in the horizontal plane which contain the incident and diffracted beams. In these conditions, since the beamline monochromator disperses in the vertical plane, diffraction in the horizontal plane suffers of the angular dispersion due to the energy band width (of the order of  $10^{-4}$ ). To overcome this limitation on strain sensitivity we put a second monochromator which disperses in the horizontal plane in order to achieve a double crystal parallel geometry. The flexibility of BM5 was particularly adapted to carry out this test. In the first run (3-5- may 2001) we successfully tested this new geometry, with a Si crystal on the first tower, the waveguide and the sample on the second tower and the detector on the third. Fig.1 shows the difference in the instrumental function between the experimental set-ups with and without the second monochromator.

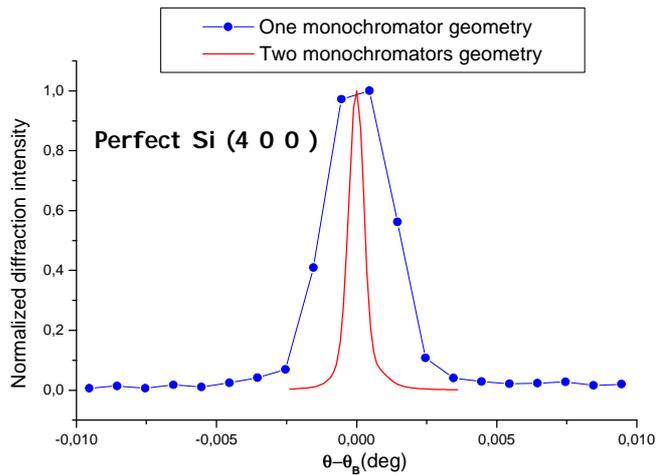


Fig.1 Instrumental function with (red solid line) and without (blue line with dots) a second monochromator dispersing in the horizontal plane

Another difference with respect to previous microdiffraction measurements was that instead to adopt a projection geometry that requires a CCD camera, we adopted a scanning geometry with the sample close to the waveguide exit and a standard scintillation detector. We were clearly pushed to this solution due to the low intensity levels at BM5, in particular after the second monochromator.

The first session demonstrated the usefulness of the insertion of the second monochromator. Measurements were carried out on samples from ST microelectronics with the aim to measure the local strain under sub-micrometer Shallow Trench Isolation structures (these measurements were made in the frame of the european project STREAM). Due to the complexity of the alignment procedures and to the low intensity we were able to measure successfully only one sample.

Fig. 2 shows an example of the measurement on 3  $\mu$  active regions separated by 1  $\mu$  space.

The same kind of measurements were carried out in the second run at BM5 (6-11 February 2002), with improvements concerning background levels and amplitude of the angular region probed. However, strong instabilities appeared, probably due to some mechanical component. We are not able to clearly explain the origin of these instabilities, but the experimental set-up, practically equal to that previously tested, was in fact quite complex and any small failure can give disastrous effects. We succeeded anyway to collect some useful data, however much less than scheduled.

It is interesting to note that the spatial variation of a given strain can be easily derived recording the diffracted intensity as a function of the vertical position maintaining the angle of incidence at the value corresponding to the given strain. Fig 3 gives an example: the periodicity (5 stripes 0.2  $\mu$  wide separated by 5  $\mu$  space region) is perfectly reproduced.

A paper related to this work: " X-ray characterization of Si microstructures with high spatial resolution" by A. Cedola et al. has been submitted for publication

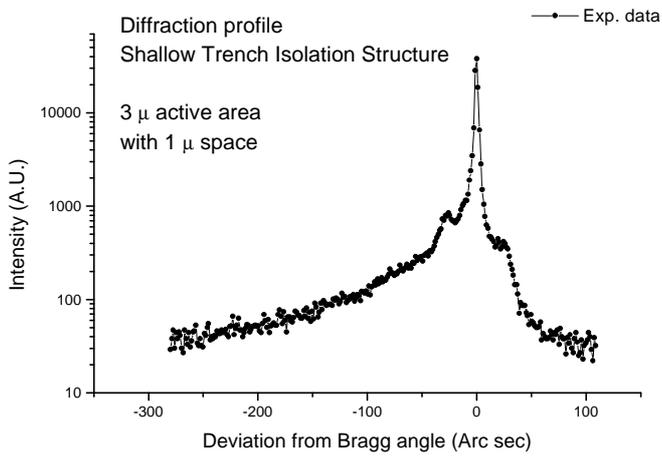


Fig. 2 Example of diffraction profile in correspondence of a 3  $\mu$  active area separated by 1  $\mu$  space in an STI structure

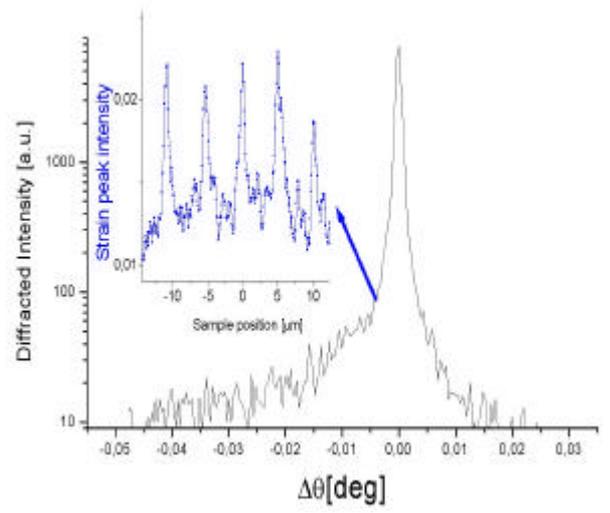


Fig. 3 Spatial variation of strain in correspondence of 0.2  $\mu$  wide active areas separated by 5  $\mu$  space in Shallow Trench Isolation structures.

