ESRF	**Experiment title:** Nanodiffraction for local residual stress analysis	**Experiment number:** HS977
Beamline: BM5- ID13- ID32	**Date of experiment:** from: 8/09/99 to: 11/09/99 from: 6/10/99 to: 13/10/99 from: 19/01/00 to: 24/01/00	**Date of report:** 21/2/00
Shifts: BM5=9 ID13=15 ID32=12	**Local contact(s):** A. Freund M. Mueller B. Cowie	*Received at ESRF:*

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Report:

The proposal was intended to be a long-term proposal that however was not awarded; beam time was anyway given at three different beamlines with the aim to further develop the application of the x-ray waveguide and of the microdiffraction method [1]. We will describe in the following the result of the experiments at the three beamlines:

BM05:

Due to the limited flux available at the optics beamline we performed mainly the characterisation of a new generation x-ray waveguide having high efficiency and extended energy range. We measured both the reflectivity and the exit beam intensity vs. incident angle at several incoming beam energy from 8 to 25 KeV. In some cases also the image of the exit beam was recorded. Two waveguides were examined, one (#1) with the guiding layer thickness of 740 Angstrom, and the other (#2) of 1100 Angstroms. From these measurements we could derive the gain, defined as the ratio between the output flux density and the input flux density, as a function of energy. The result was that a gain as high as respectively 100 and 50 was obtained with the waveguide #1 and #2, with an improvement of an order of magnitude with respect to the previous waveguides, and of three orders of magnitude with respect to the first ones examined in 1995.

Furthermore the edge and the core from a human hair was examined in phase contrast microradiography. Data analysis is in progress.

ID13

One of the main goal of this experiment was to test the new version of the apparatus that has been designed and realized by ESRF for the specific needs of the waveguide. The old version had serious problems of reliability and for this reason was deeply modified adopting more standard mechanical components. A drawback is that the apparatus is now adapted only to the ID13 beamline and cannot be transferred to other beamlines. The test was positive, and the apparatus showed a satisfying reliability and flexibility. Some improvements have been discussed with the staff of ID13.

Concerning scientific activity, the attention has been concentrated on microscopic analysis of the domain walls in a LiNbO3 crystal. From previous measurements by other groups, who gave us the sample, a strong phase contrast effect at the domain walls with the typical periodicity of the domains was found. The beam from the waveguide is ideal to perform this kind of study with higher spatial resolution, and to test at the same time the presence of lattice distortion at the domain walls. Several measurements have been carried out in order to find a regular pattern, but unsuccesfully. A certain periodicity was found only for a limited spatial extent. The reason for this was clear after examination with standard x-ray topography of the sample surface that, unexpectedly, resulted full of defects, probably caused by a rough polishing procedure. Careful analysis of data is in process in order to extract all the possible information by this experiment.

We were then ready to start measurements on another kind of sample, i.e. a SiGe heterostructure, but a serious failure happened in the ring, and the last day of the run was cancelled.

ID32

The interest to carry-on waveguide experiments at the ID32 b.l. was due mainly to two reasons: i) to test the behaviour of the WG with an undulator on a high-beta section; ii) to use a high precision diffractometer to analyse the waveguide beam diffracted by the sample. In order to carry-on the experiment a strong effort has been made by the beamline staff to adapt standard mechanical components to the diffractometer to allow the waveguide operation. This was successful and we had a quite good control both of the waveguide and of the sample movements. The beam exiting from the waveguide was several times more intense than at ID13. The reason for this is still unclear. Because of the good beam conditions we tried to obtain from a specially prepared waveguide a beam compressed in two directions (standard waveguides compress the beam only in one direction). This was a challenging experiment that necessitated of high intensity and was not possible to perform at the optical beamline. Unfortunately we didn't reach the objective, probably because the waveguide used was not well enough optimized. We obtained however useful indications for further trials.

We then tried a novel diffraction geometry that can be very useful to study thin films. Data analysis is in progress.

In all these experiments we used the SENSICAM, a CCD camera given to us by the ID15 beamline, that resulted quite well adapted to the needs of waveguide experiments.

[1] Di Fonzo et al., Nature, 403,638 (2000)