ESRF	**Experiment title:** **Mechanical properties and pressure induced phase transformations of low-dimensional bismuth**	**Experiment number**: HS 3903
Beamline: ID01	**Date of experiment**: from: 28/10/2009 to: 03/11/2009	**Date of report**: 31/08/2010
Shifts: 18	**Local contact(s)**: T.H. Metzger	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

***T.W. Cornelius, ESRF, Grenoble, France**

***T. Scheler, University of Edinburgh, Edinburgh, United Kingdom**

R. Neumann, GSI Helmholtz Zentrum for Heavy Ion Research, Darmstadt, Germany

S. Müller, GSI Helmholtz Zentrum for Heavy Ion Research, Darmstadt, Germany

Report:

We studied the mechanical properties of bismuth by combining the *in-situ* atomic force microscope (AFM, Small Infinity) of ID01 and microfocused X-ray diffraction. For this purpose, the X-ray beam was focused to 1.2 µm x 2.8 µm by means of Be compound refractive lenses. The bismuth strucures under investigation were prepared by evaporating a continuous film of thickness 500 nm on a Si wafer. Subsequently, this polycrystalline film was structured by UV lithography creating 10 x 10 μm^2 large Bi islands that contain 500 nm sized crystals. A scanning electron microscopy (SEM) image of these islands is presented in Fig. 1a.

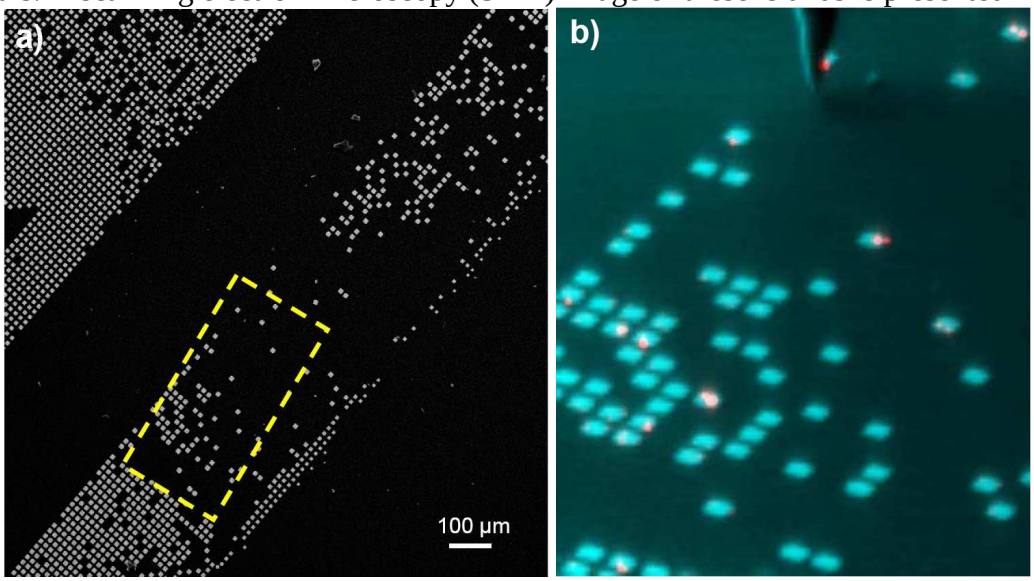

Fig. 1: a) Scanning electron micrograph. b) Superposition of photocurrent image and X-ray diffraction map

For mutual alignment of the sample, the AFM-tip, and the X-ray beam we used AFM imaging, photocurrent imaging (PI), and scanning X-ray diffraction (SXD) mapping, respectively. In Fig. 1b, a superposition of a PI

and a SXD map around the Bi(012) Bragg peak is displayed. The scanned area is indicated by the box in the SEM image (Fig. 1a). The Bi islands as well as the AFM-tip are clearly visible in the photocurrent image. The superposition with the SXD map allows the unambiguous correlation of specific Bi grains fulfilling the Bragg condition. Thus, this superposition demonstrates the excellent alignment capabilities by the combination of these techniques.

After aligning the AFM-tip with respect to both a specific Bi grain and the microfocused X-ray beam the sample was moved against the AFM-tip, thus, applying a force on the crystal. While pressure application the resonance frequency of the AFM tuning fork as well as the X-ray diffraction pattern of the Bi grain were recorded. A sequence of XRD images is presented in Fig. 2a revealing significant changes in the patterns during pressure application. The visible part of the Debye-Scherrer-ring moves to lower Bragg angles, i.e. the lattice parameter increases during compression. Moreover, the Bragg peaks move horizontally on the CCD image indicating a tilting of the corresponding grains. These findings are attributed to the lateral expansion of the grain under pressure leading to a lateral compression of neighbouring grains and, thus, to their vertical expansion and tilting. The last image of the sequence was taken after pressure release. The pattern is totally different to the original one indicating plastic deformation which is proven by the SEM image in Fig. 3a. Two indentation areas are visible in the micrograph due to the fact that two compression testst on this Bi structure was performed.

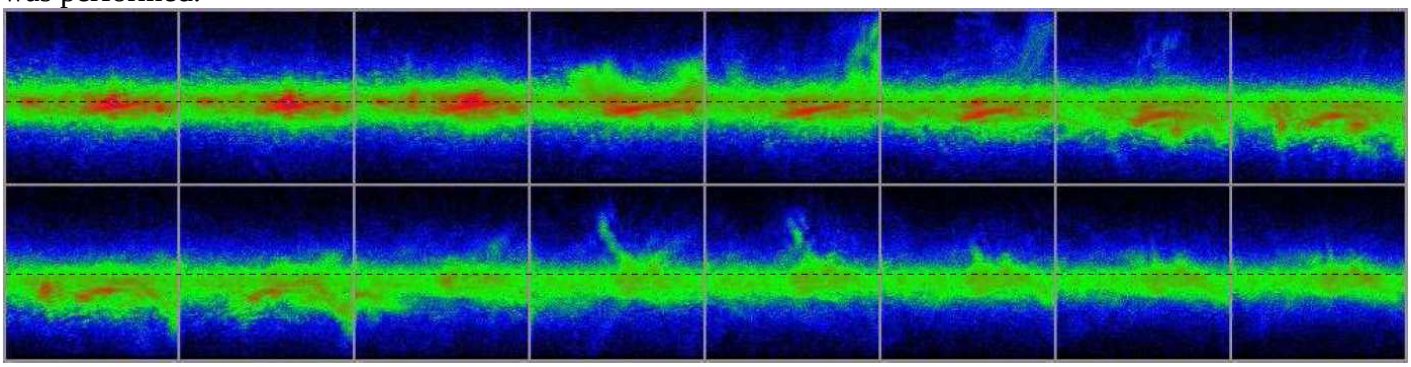

Fig. 2: Sequence of XRD images during pressure application.

For further data evaluation we analyzed the center of mass of the intensity distribution in q_x and in q_z-direction of the diffraction images as a function of both the resonance frequency and the applied pressure. The two centers of mass give access to the mean change of the lattice parameter. Abrupt changes are apparent in the graph where compressions in the vertical center of mass is accompanied with a change in the horizontal center of mass. These sudden changes may be attributed to defect creations which occur at very similar compression states, namely 0.03 % lattice compression. Moreover, they occur at 0.6, 0.87, 1.2, 1.6, and 1.95 GPa. Thus, an additional pressure of 0.3 – 0.4 GPa was necessary to induce a new burst of defects. Note that these changes are convoluted with the overall expansion of the illuminated sample.

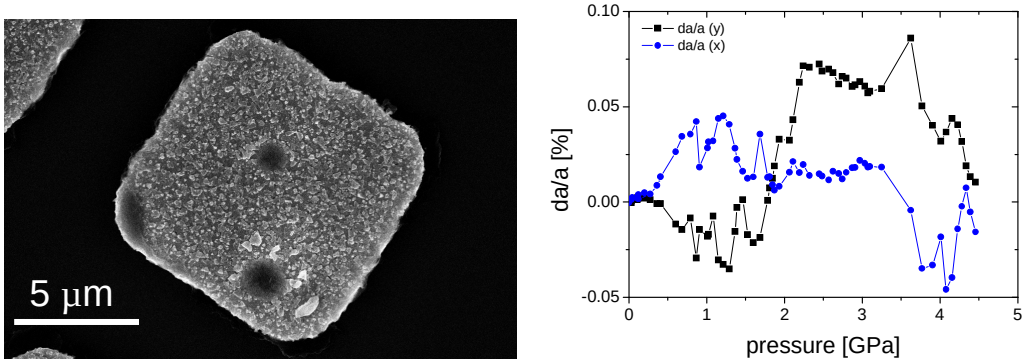

Fig. 3: a) SEM image after two indentation tests showing the plastic deformation of the structure. b) Lattice parameter change as a function of the applied pressure.

In conclusion, we could follow the plastic deformation of bismuth during compression tests *in-situ*. However, no pressure induced phase changes could be recorded, most probably, due to the fact that only an uniaxial pressure was applied and, thus, the structures could relax laterally.