ESRF	**Experiment title:** Nano-mechanical properties of ZnO piezoelectric semiconductor nanowire	**Experiment number**: A32-2-850
Beamline: BM32	**Date of experiment**: from: 28/10/2022 to: 01/11/2022	**Date of report**: 13/12/2022
Shifts: 12	**Local contact(s)**: MICHA Jean-Sebastien, ROBACH Odile, PURUSHOTTAM RAJ PUROHIT Ravi Raj Purohit	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

CORNELIUS, Thomas*; SAIDI, Soufiane*; TEXIER, Michael; ESCOUBAS, Stéphanie **(Aix Marseille Univ, CNRS, IM2NP UMR 7334, France)**

ARDILA-RODRIGUES, Gustavo **(IMEP-LaHC, INPG, Grenoble, France)**

SHARMA, Shruti*; TERNON, Céline **(LMGP, INPG, Grenoble, France)**

Report:

In this experiment we aimed to study the mechanical properties of single ZnO nanowires by *in situ* Laue microdiffraction during three-point bending tests using a custom-built AFM. Due to the size effect, it is expected for the nanowires to approach the limits of the yield strengths which allows for large elastic deformations.

ZnO nanowires with a diameter of 200 nm and a length of up to 10 µm were grown by hydrothermal growth method. The ZnO nanowires were detached from their growth substrate by ultrasonication that lead to a breaking of the nanowires into sections of 3 to 4 µm in length. The nanowires were then dispersed on a silicon substrate with 2 µm wide trenches. Some of the nanowires crossed the micro-trenches resulting in self-suspended nanowires that were thoroughly fixed by electron beam induced deposition of Pt at the nanowire ends (see Fig. 1(a)). For *in situ* three-point bending tests in combination with Laue microdiffraction, the custom-built AFM "SFINX" was installed on the sample stage at the BM32 beamline. The incident polychromatic X-ray beam was focused down to 300 nm x 500 nm using a pair of Kirckpatrick-Baez mirrors. The nanowires were located by measuring the Zn-K_α and Pt-K_α fluorescence yield as illustrated by the fluorescence maps displayed in Fig.1(b) showing the same nanowire as in Fig. 1(a).

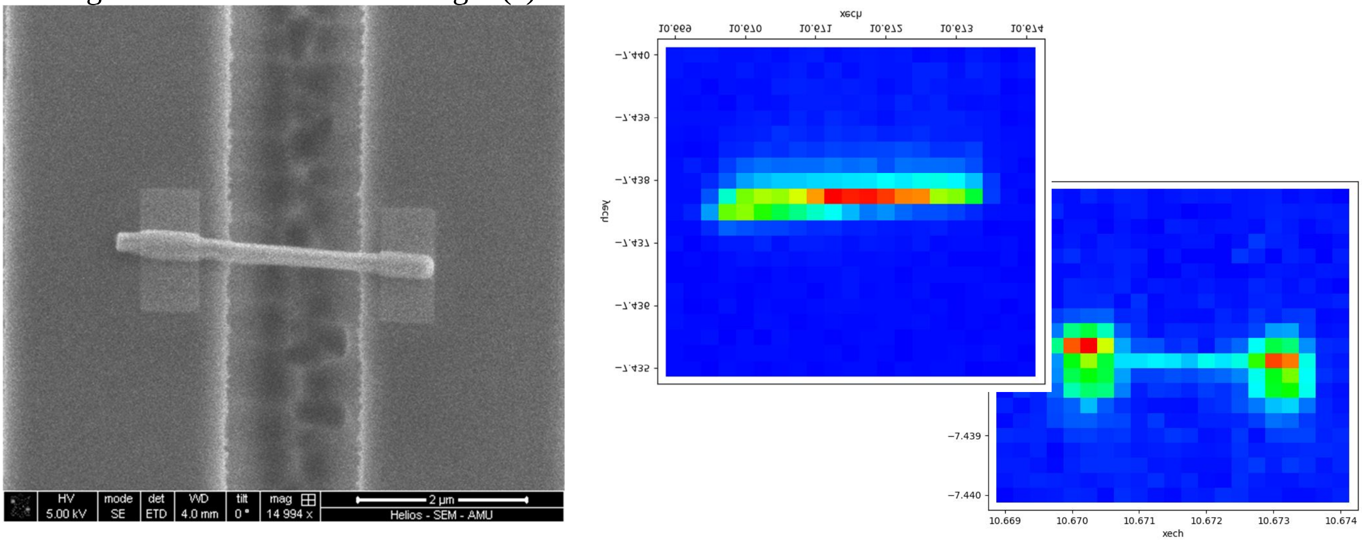

Fig. 1: a) Scanning electron microscopy image and b) Zn/Pt-K_α fluorescence yield map of a suspended ZnO nanowire.

Laue microdiffraction patterns were recorded *in-situ* while bending the nanowire. Figure 2 presents a mosaic of the displacement of the ZnO (0 $\bar{2}$ 2 0) Laue spot during the application of a force on the nanowire. The Laue spot displacement on the detector indicates a rotation of the crystalline lattice by bending and/or torsion of the nanowire. Besides the displacement of the spot, it also enlarges, in particular, in the vertical but also in the horizontal direction as illustrated in Fig. 3, indicating the local deformation of the ZnO crystal.

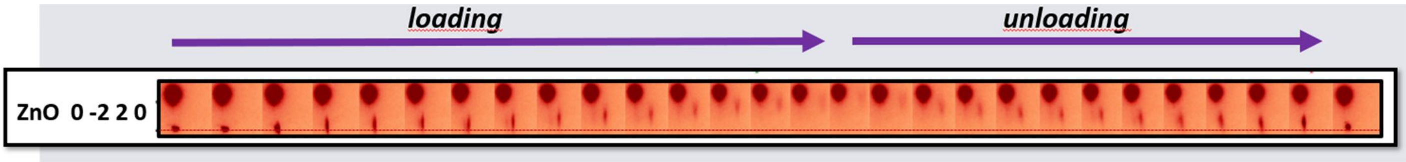

Fig. 2: Mosaic of the ZnO (0 $\bar{2}$ 2 0) Laue spot during bending test

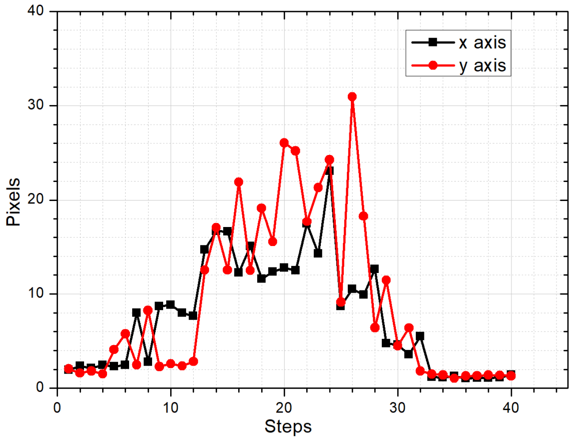

Fig. 3: Enlargement of the ZnO (0 $\bar{2}$ 2 0) Laue spot size during the bending test in reference to the original size

The ZnO nanowire microdiffraction patterns were indexed using the LaueTools software providing the UB orientation matrices. The rotation of the crystal was calculated with help of these matrices. Fig. 4 shows the rotation angle as a function of the measurement steps during loading and unloading of the nanowire with the AFM tip for the three orthogonal lattice planes (0 0 0 1), (0 $\bar{1}$ 1 0) and the (2 $\bar{1}$ $\bar{1}$ 0). The rotation of the (0 $\bar{1}$ 1 0) and the (0 0 0 1) crystal planes indicate a bending of the nanowire. The additional rotation of the (2 $\bar{1}$ $\bar{1}$ 0) crystal plane, however, shows a more complex behavior, which may indicate either a torsion or a non-vertical bending due to a non-vertical force application.

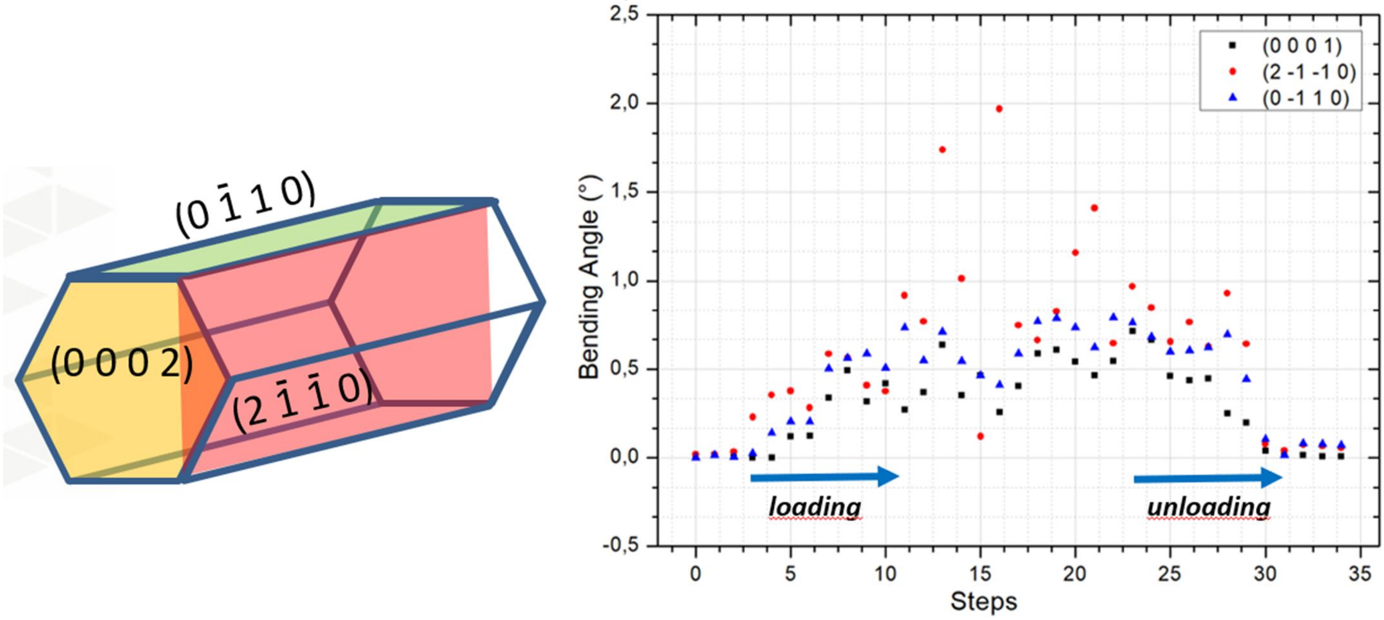

Fig. 4: Rotation of the three orthogonal lattice planes (0 0 0 1), (0 $\bar{1}$ 1 0), and (2 $\bar{1}$ $\bar{1}$ 0) of a ZnO nanowire as a function of an applied voltage inferred from in situ Laue microdiffraction

During this beamtime, we tested 10 ZnO nanowires by *in situ* three-point bending up to their fracture as illustrated by the SEM images taken before and after the experiment. The data is currently under analysis.

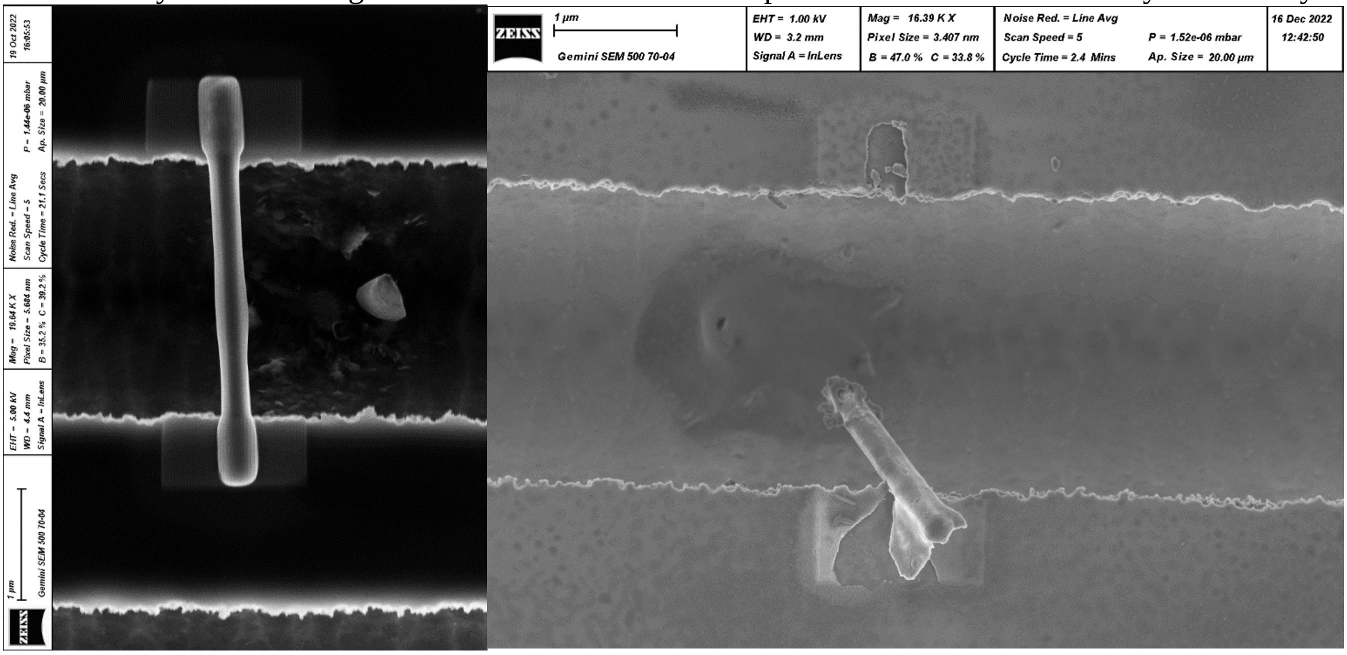

Fig. 5: SEM image of a suspended ZnO nanowire before and after mechanical testing

<u>- Justification and comments about the use of beam time</u> <u>(5 lines max.):</u>

This *in-situ* Laue microdiffraction experiment during the three-point bending of single suspended ZnO nanowires focused on the mechanical behavior of the nanowires such as elasticity, plasticity, and fracture toughness. While this beamtime focused on ZnO nanowires with a diameter of ~200 nm, future experiments will focus on thinner nanowires to study the fracture toughness.

<u>- Publication(s)</u><u>:</u>

S. Saïdi, M. Texier, S. Escoubas, O. Thomas, S.Sharma, G. Ardila, C. Ternon, T.W. Cornelius, *In-situ evaluation of the electromechanical behavior of ZnO nanowires,* **ESRF User meeting 2023**, Grenoble (France) Feb 2023, *poster*