ESRF	**Experiment title:** IN-SITU STRAIN MAPPING OF 2D MATERIALS FOR NANOSCALE OPTICAL APPLICATIONS	**Experiment number**: MA-5167
Beamline: BM32	**Date of experiment**: from: 06.07.2022 to: 11.07.2022	**Date of report**: 28.02.2023
Shifts: 9	**Local contact(s)**: Ravi raj purohit Purushottam raj purohit (email: purushot@esrf.fr) Odile Robach (email: odile.robach@cea.fr)	*Received at ESRF: 28.02.2023*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Javier Martín-Sánchez* (University of Oviedo)

Dr. Carlos Quirós* (University of Oviedo)

Mr. Abel Martínez-Suárez* (University of Oviedo)

Dr. Odile Robach* (ESRF)

Report:

This proposal aimed a systematic study about the possibility to introduce strain fields on MoO_3 2D flakes by employing a novel hybrid 2D-piezoelectric device. Specifically, a pizoelectric $Pb(Mg_{1/3}Nb_{2/3})O_3]_{0.72}$-$[PbTiO_3]_{0.28}$ (PMN-PT) plate was gold coated on both side for electrical contacts and the 2D flake was transferred on top. The application of an electric field (i.e., voltage) along the piezoelectric plate will produce the out-of-plane deformation of the piezoelectric plate, which introduces an in-plane deformation due to the elastic response of the plate, that will be eventually transferred to the attached 2D flakes.

We note that the amount of collected data is very large and, therefore, its analysis is time consuming. Nevertheless, although the detailed study of the results is still under progress, we can already obtain some conclusions, as shown below, that allow us to address future research directions.

In the following, we comment on the conclusions obtained on each of the main aims for the suggested experiments in this proposal based on our preliminary analysis of the collected data by micro-Laue diffraction.

<u>1. Successful integration of the fabricated piezoelectric devices in the experimental setup at beamline BM32.</u>

- The installation of the hybrid devices in the BM32 beamline, including wiring, assembling and setup configuration was successfully accomplished. This is a very important milestone towards the development of future experiments employing these type of devices to optimize the already obtained results and extend them to other interesting nanomaterials, where in-situ studies of the introduced strain fields can be performed.

<u>2. Direct measurement of the piezoelectric device response as a function of the applied voltages: (001) and (110) piezoelectric plate orientation.</u>

The characterization of the commercial piezoelectric plates used in these experiments (purchased from TRS Technologies) is fundamental to fully understand the expected strain fields transferred to the attached 2D MoO_3 crystals. The response of the piezoelectric plates can be estimated from the diffraction spots related to the piezo crystal in the micro-Laue pattern. Note that any deviation between the strain field introduced by the piezoelectric plate and the eventual strain field measured in the 2D crystals will indicate the quality of the attachment of the bonding between the crystals and the piezoelectric substrate. The obtained results and conclusions are shown in the following.

- The functionality of the piezoelectric plates was tested on both type of devices with (001) and (110) orientations. Although the detailed study of the results to obtain the strain fields is currently under progress, we can extract some interesting partial conclusions. Specifically, we can measure the relative displacement of two diffraction spots in the micro-Laue diffraction pattern related to the PMN-PT piezoelectric plate, which can be directly related to the strain introduced by the piezoelectric plates as shown in Figure 1. Interestingly, a relatively good reversible behaviour is observed in the piezoelectric plate response as expected. The small hysteresis is attributed to a quick voltage ramp during the measurements (a waiting time of 5 minutes was performed to stabilize the piezoelectric drift, which is clearly insufficient for an optimum performance).

- A very large out-of-plane piezoelectric plate deformation was observed (up to 30 microns). We note that this gives an impressive out-of-plane strain value of about 15%.This is a very interesting result unknown until these measurements that has boosted the design of a new piezoelectric device to introduce extremely large uniaxial strain fields by rotating 90º the piezoelectric plate. We intend to use this type of devices in our next experiments.

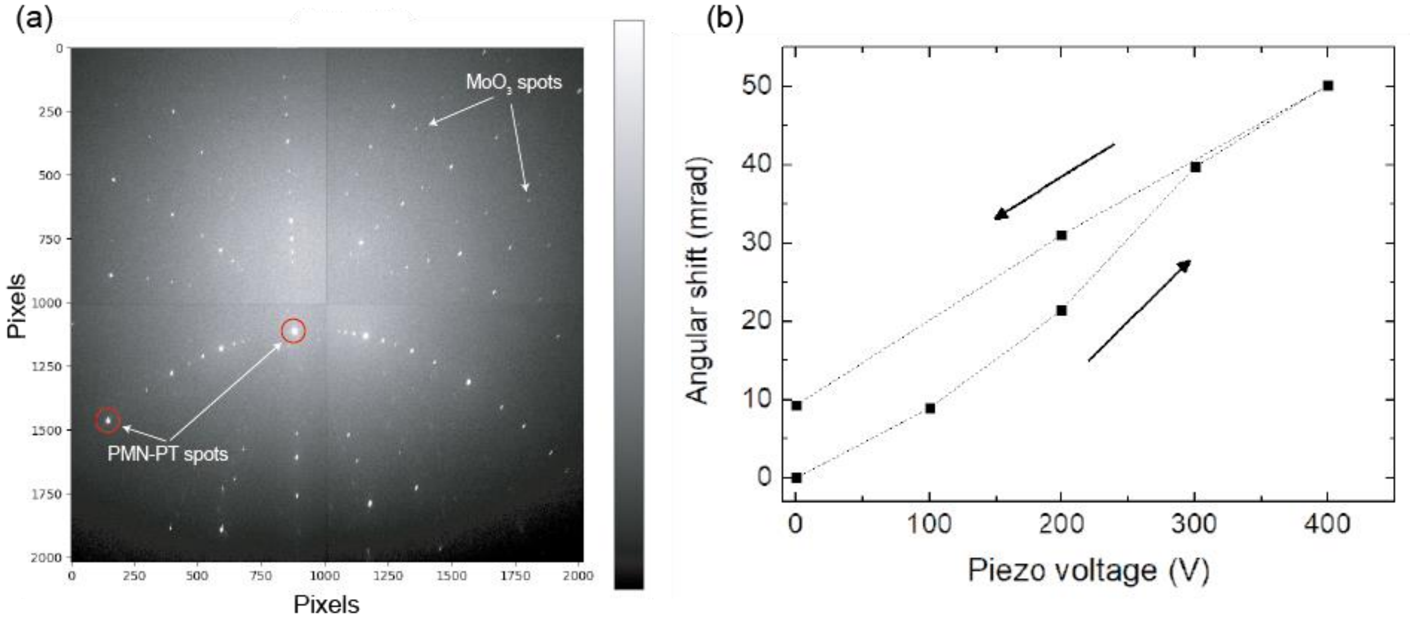

Figure 1.- Piezoelectric response of (001)-oriented PMN-PT plates. (a) Micro-Laue diffraction pattern where diffraction spots related to the piezoelectric plate and MoO_3 flakes are resolved. (b) Relative angular shift between the spots corresponding to the PMN-PT plate marked with red circles in panel (a). A relatively good reversible response is identified where the observed hysteresis is attributed to well-known drift effects of piezoelectric materials owing to a rapid voltage sweep.

- Our preliminary results reveal some indications demonstrating an anisotropic response of the (110)-oriented PMN-PT plates and an isotropic response of the (001)-oriented plates. This is closely following the expectations as given by TRS Technologies and as calculated by numerical simulations.

<u>3. Effect of the thickness of MoO_3 flakes on the measured strain fields.</u>

- The micro-Laue pattern shows faint diffraction spots related to the MoO_3 2D crystals owing to the limited thickness of the crystals employed in these experiments (maximum thicknesses of about 100 nm). Nevertheless, we were able to track some of the diffraction spots to study relative shifts trying to compare them with those observed for the PMN-PT plates shown in point 3 of this report. Although our preliminary results do not allow us to withdraw firm conclusions at the moment, we did observe relative changes between some of the diffraction spots related to the MoO_3 2D crystals as a function of the applied voltage. However, these changes are not so large as the ones observed on the piezoelectric plates. Based on our experimental findings during this beamtime, we believe that a better result is possible in terms of strain transfer by improving the adhesion between the MoO_3 2D crystals and the piezoelectric plates. This is somehow unexpected based on our previous studies using h-BN 2D crystals and that could be due to a different adhesion strength to the piezoelectric device. On the other hand, thicker MoO_3 crystals would help to improve the brightness of the diffraction spots.

- We also observed elongated diffraction spots related to the MoO3 crystals as the voltage was increased (i.e., applied strain). We attribute this result to irreversible morphological changes of the MoO_3 2D flakes likely due to flake bending induced by the poor adhesion of the flake to the piezoelectric plate.

We believe that the obtained results during this set of experiments are very valuable considering that this is the first time we measure this type of devices in a synchrotron facility. Moreover, the results will help to improve some aspects of the device design as mentioned above. Specifically, for a future proposal, we believe that both slightly increasing the thickness of the MoO_3 crystals and enhancing the bonding by employing an intermediate SU-8 bonding layer would allow help to improve our results as we demonstrated in GaAs nanomembranes in a separate work [1]. We highlight that the quantitative estimation of the angular shifts in the piezoelectric plate diffraction spots (shown in Figure 1b) is a very interesting and new result that will allow us to extract the strain tensor of the deformed piezoelectric plate at different voltages.

References:

[1] D. Ziss, J. Martín-Sánchez et al. J. Appl. Phys. 121, 135303 (2017)