## Visualisation of subsurface defects in van-der-Waals heterostructures via 3D SPM mapping

<sup>1</sup>Marta Mucientes, <sup>2</sup>Melinda J. Shearer, <sup>2</sup>Bob Hamers, <sup>2</sup>Yuzhou Zhao, <sup>2</sup>Song Jin and <sup>1</sup>Oleg V. Kolosov.

<sup>1</sup>Physics Department, Lancaster University, LA1 4YB, UK <sup>2</sup>Department of Chemistry, University of Wisconsin-Madison, WI 53706, EE. UU <u>m.sanjuanmucientes@lancaster.ac.uk</u> <u>o.kolosov@lancaster.ac.uk</u> <u>www.nano-science.com</u>

The two-dimensional (2D) materials and their multi-layered heterostructures are being actively explored as new materials for multiple applications in optoelectronics, energy storage, gas- and biosensors, and photocatalysis and solar energy conversion, to mention a few. In particular, the transition metal dichalcogenides (TMDs), such as tungsten disulfide (WS<sub>2</sub>) and tungsten diselenide (WSe<sub>2</sub>), are promising materials for optoelectronic applications thanks to their unique optical, electrical and mechanical properties [1]. While these materials and their complex WS<sub>2</sub>-WSe<sub>2</sub> heterostructures can be effectively grown by chemical vapour deposition, the resulting structures and their surface and interfacial defects may significantly affect the performance of resulting devices.

We therefore studied the hidden subsurface features of the samples, such as dislocations and stacking faults. They have been identified by nanomechanical mapping via the well-known methods Ultrasonic Force Microscopy (UFM) and Heterodyne Force Microscopy (HFM). These techniques combine Atomic Force Microscopy (AFM) with ultrasonic excitation of the tip, sample or both, to probe changes in the sample stiffness due to different materials, buried defects, and crystal quality or layer thickness variations [2]. This work has been also completed by the cross-sectioning of the TMD heterostructures with Beam Exit Cross-sectional Polishing (BEXP<sup>TM</sup>), revealing the inner structure by cutting the material with Ar-ion beam resulting in near-atomically flat sections. This methodology creates an oblique damage-free section, with a very shallow angle from the surface, which allows easy access via SPM methods to the near-surface and deep subsurface regions [3].

The nanomechanical mapping of the UFM and HFM images shows clear contrast in areas that can be either linked or independent from the topographical features regions (Fig 1.a and 1.b), both for intact and BEXP<sup>™</sup> sectioned heterostructures (Fig 1.c and 1.d). The obtained subsurface structures indicate that these are linked to dislocations and stacking faults, as well as misorientation of the crystallographic axis of the layers.



- [1] Eftekhari A, J. Mater. Chem. A, 5 (2017) 18299-18325.
- [2] Cuberes MT et al., Journal of Physics D-Applied Physics, 33 (2000) 2347-2355.
- [3] Kolosov, O. V. et al. Nanotechnology 22, (2011), 185702.