Nanomechanical visualisation of subsurface defects in WS_2/WSe_2 CVD flakes via Ultrasonic Force Microscopies

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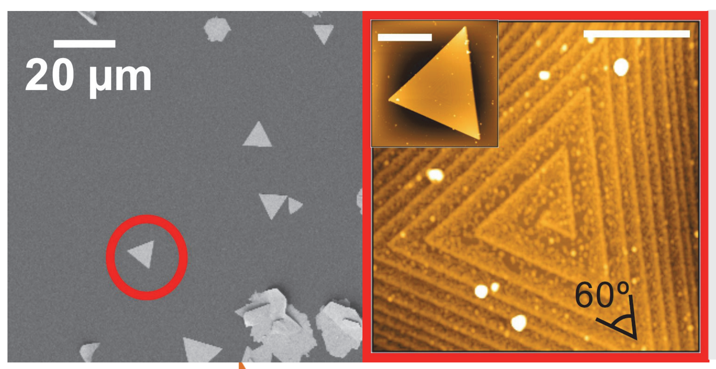
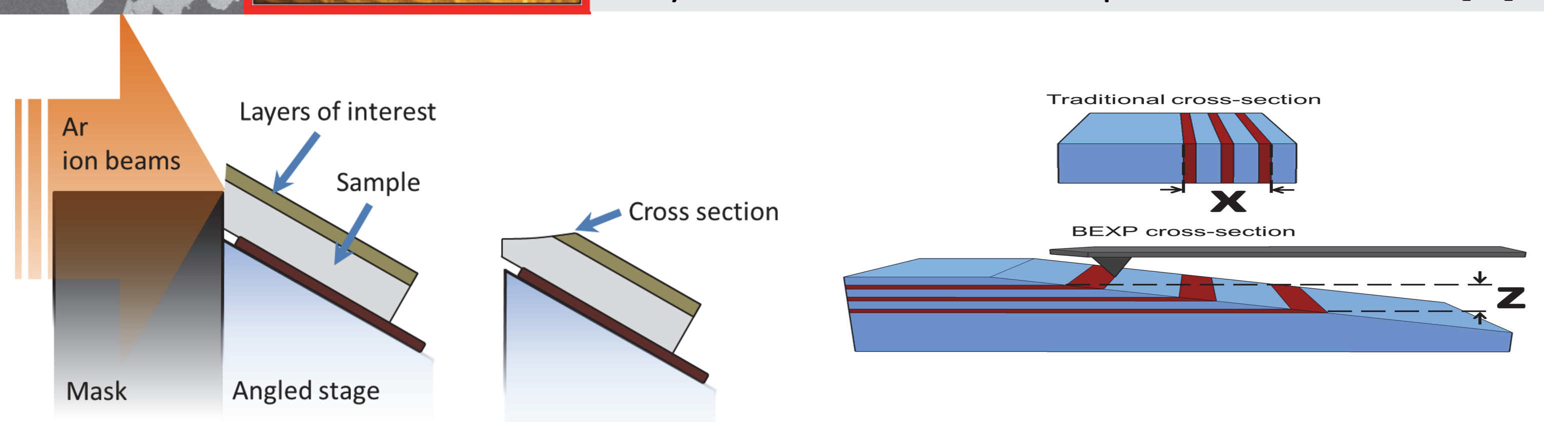
The implementation of **two-dimensional (2D) materials** in the development of electrical and optoelectronic devices has boosted the technological advances of the last years. In particular, the layered **transition metal dichalcogenides (TMD)** tungsten disulphide (WS_2) and tungsten diselenide (WSe_2) that are currently widely used in the aerospace, automotive, medical as well as military applications, have attractive optoelectronic properties as a photodetectors and light emitters in demanding environment conditions [1]. This work shows the visualization of hidden defects in the CVD WS_2 grown layers via different **Scanning Probe Microscopies (SPM)**. Moreover, the study has been complemented with the nano-cross-sectioning via Beam-Exit-Cross-section Polishing (BEXP) of WS_2+WSe_2 co-grown heterostructures.

Sample preparation



The chemical vapor deposition (**CVD**) growth of TMD materials allows to achieve complex few-layer structures, usually linked via the screw dislocations connecting different layers of 2D crystal structures in spiral structures [2].



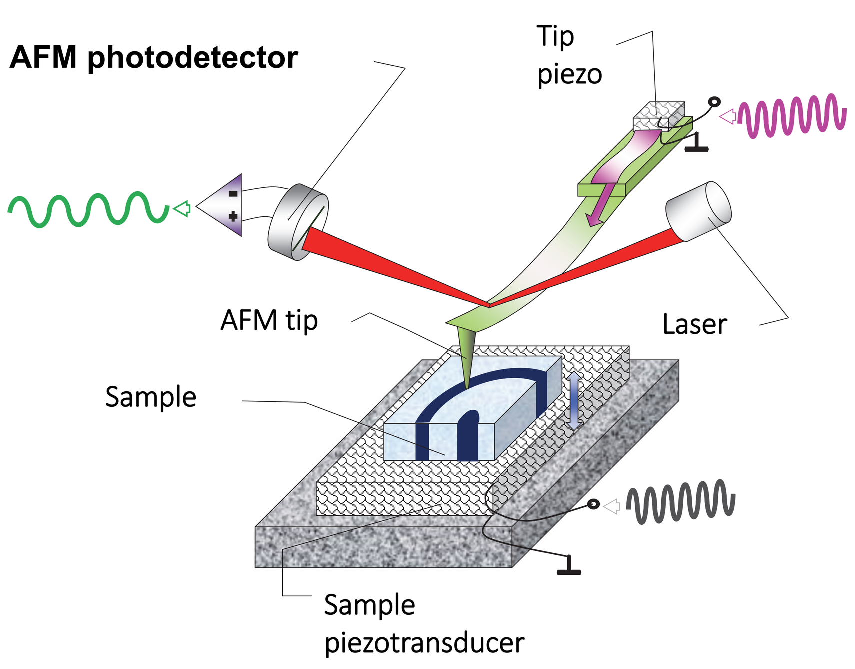
The Beam-Exit-X-Sectional Polishing (**BEXP**) is a novel ion-beam polishing method creating oblique sub-nm roughness flat section perfectly suitable for SPM studies. [3].

SPM methods

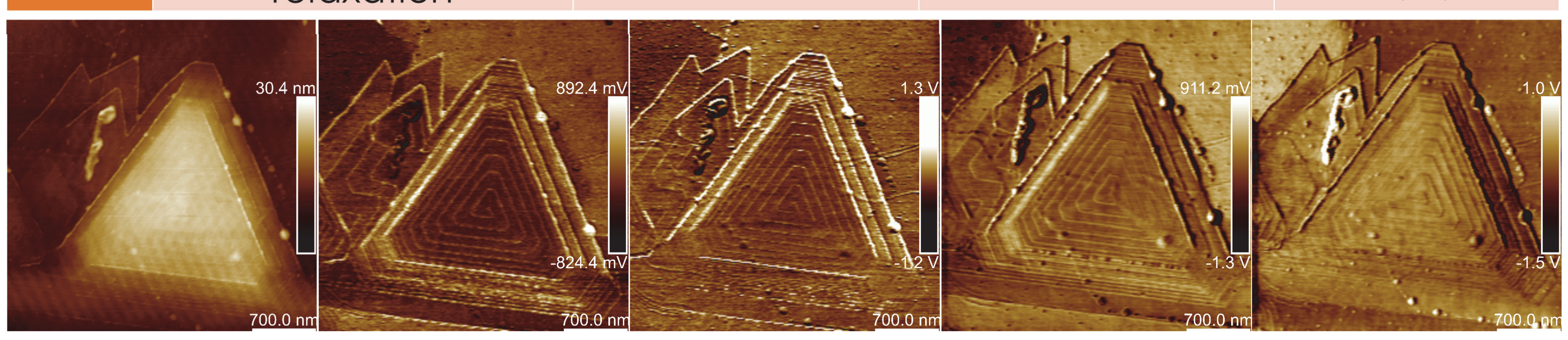
We use several SPM techniques combining Atomic Force Microscopy (AFM) with ultrasound to achieve nanomechanical mapping - Ultrasonic Force Microscopy (UFM), Waveguide and Heterodyne Force Microscopy (W-UFM and HFM) to identify the dislocations and faults between several stacked TMD layers [4].

The techniques are implemented in a modified AFM allowing to ultrasonically excite the sample/cantilever (table below) via piezoelectric drive at MHz frequencies.

Modulating the HF excitation produces cyclical nanoindentation of the tip into the sample and by monitoring the subsequent cantilever deflection at the modulation frequency, the resulting images show nanoscale maps of the sample stiffness [5].



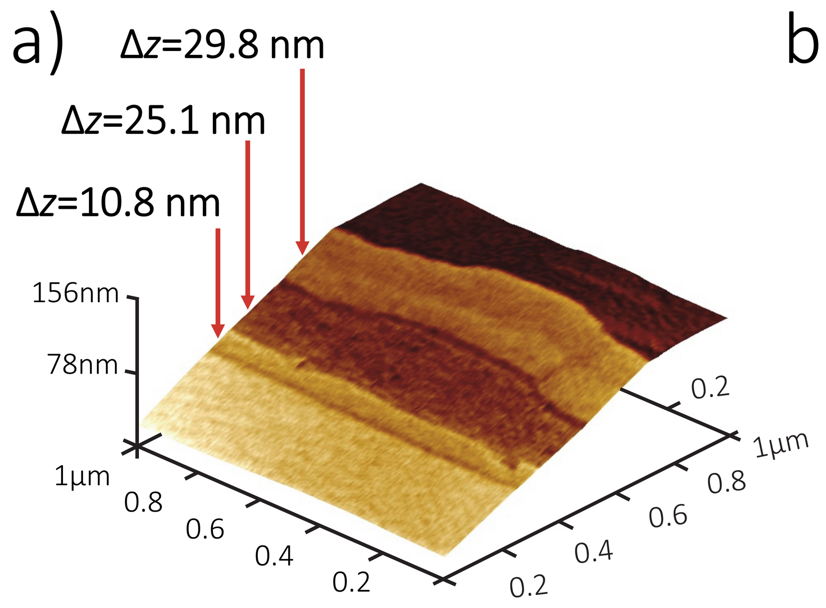
	INFO	SETUP: Tip piezo exc.	SETUP: Sample piezo exc.	MODULATION
AFM	Topography	✗	✗	✗
UFM	Stiffness	✗	✓	✓
W-UFM	Stiffness	✓	✗	✓
HFM	Stiffness and dynamic relaxation	✓	✓	✗



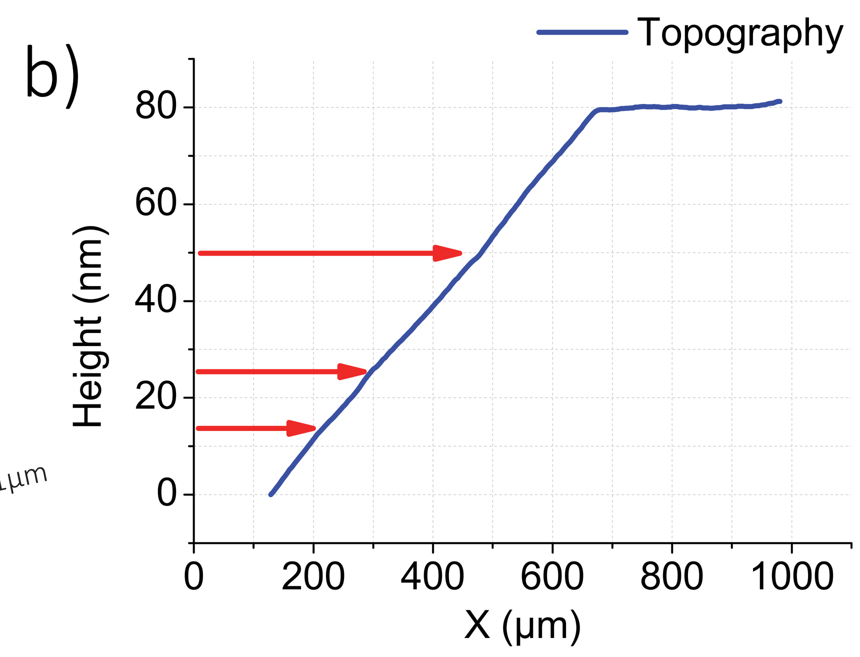
TOPOGRAPHY UFM W-UFM HFM-Ampli. HFM-Phase

Results

a)

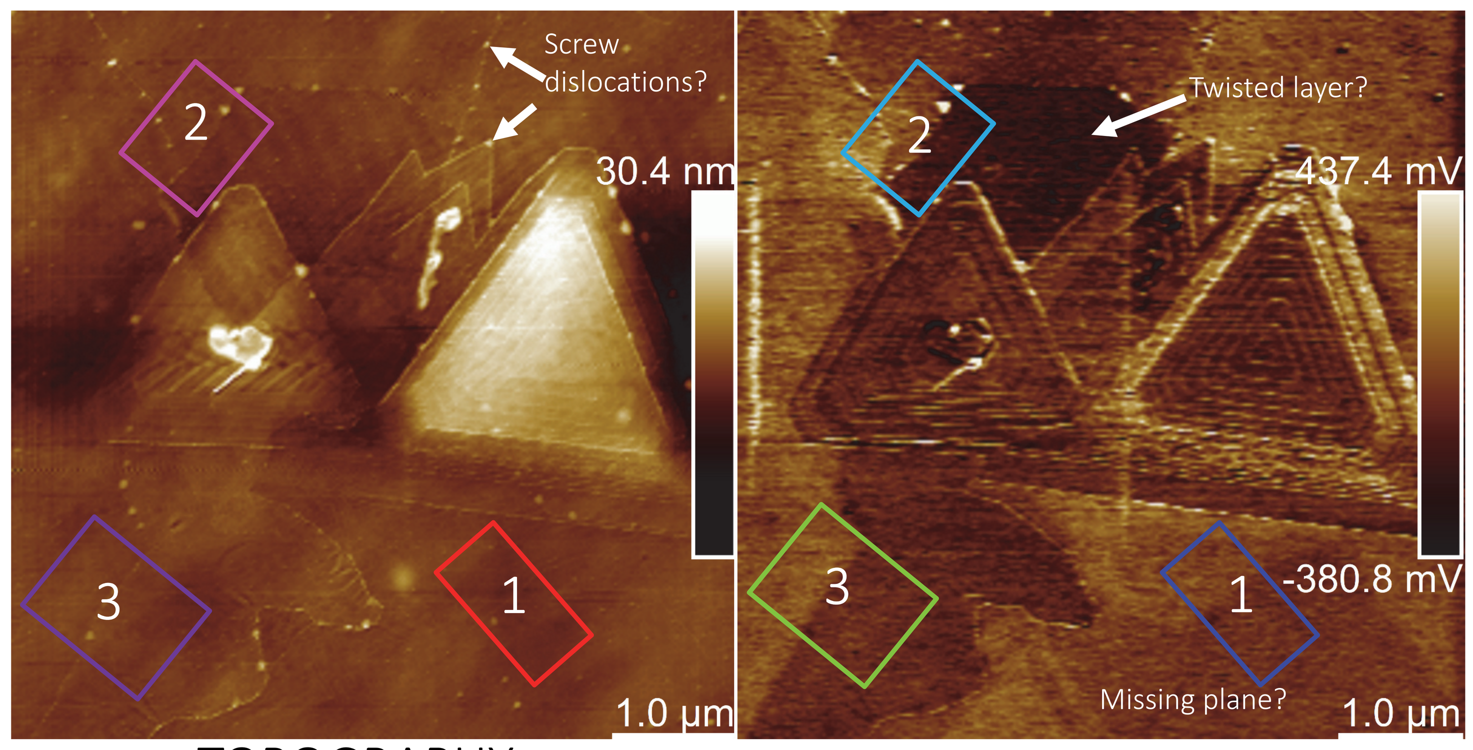


b)



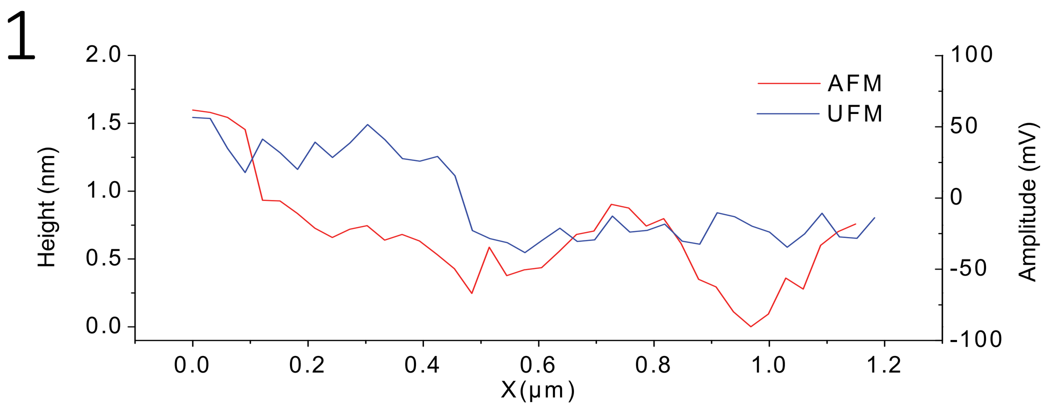
(a) 3D rendering of the cross-section topography, with superimposed contrast reflecting nanomechanical properties of the different layers via UFM (bright – more stiff).
(b) Profile of the topographical section showing slightly different slopes of sample layers.

Comparison of AFM and nanomechanical UFM profiles.



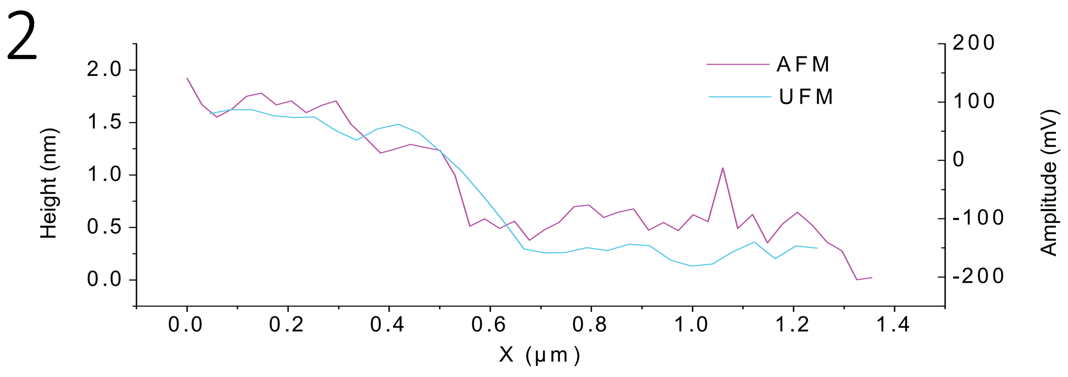
TOPOGRAPHY UFM

1



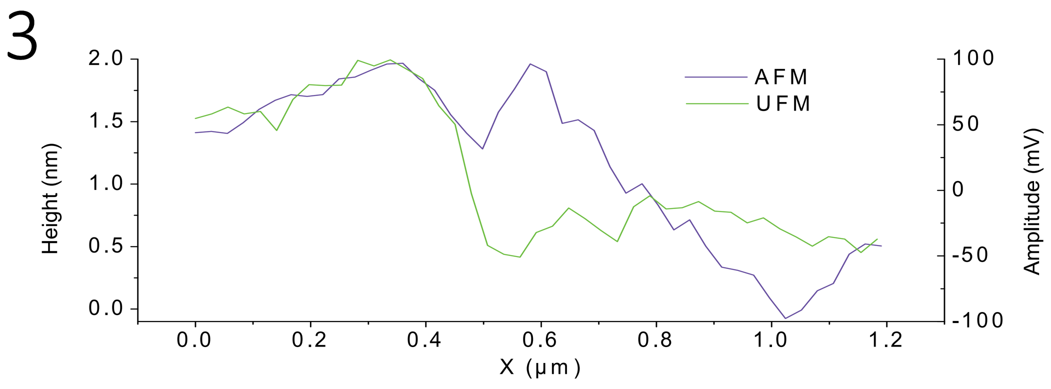
1. No clear topographic steps whereas the nano-mechanical contrast is present. We believe due to missing plane in the buried layers.

2



2. Topographical step and UFM contrast: few layers difference.

3



3. Topographical dip but no step-change in UFM: possible subsurface plane mis-orientation of the crystallographic axis and/or edge dislocation.

Conclusions

The nanomechanical contrast in UFM, W-UFM and HFM images of surface and nano-cross-sectioned layers show excellent contrast and details in areas in which may or may not have direct topographical features. There is a strong indication that these suggest subsurface features such as missing plane or plane misorentation of the buried layers. Variation of the contrast between different nanomechanical modes demands a more detailed study of the nature of such contrast currently underway.

References:

[1] A. Eftekhari, J. Mater. Chem. A, 5 (2017) 18299-18325.
[2] M.J. Shearer, et al., Journal of the American Chemical Society, 139 (2017) 3496-3504.
[3] A.J. Robsonet al., Acs Applied Materials & Interfaces, 5 (2013) 3241-3245.
[4] Yamanaka K et al., Jpn J Appl Phys Part 1. 1994;33(5B):3197-203.
[5] Bosse JL et al., Journal of Applied Physics, 2014, 115(14):144304.

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