Subsurface imaging of stacking faults and dislocations in WS_2 CVD grown flakes via Ultrasonic and Heterodyne Force Microscopy

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The two-dimensional (2D) materials hold multiple promise in modern day technology, from electronics and optoelectronics [1], to fabrication of micro and nanoelectromechanical systems (MEMS and NEMS) and flexible displays. In particular, the layered transition metal dichalcogenide (TMD) tungsten disulphide (WS_2) that is currently widely used in the aerospace, automotive, medical as well as military applications as a lubricant, has attractive optoelectronic properties as a photodetector and light emitter in demanding environment conditions. This work opens the possibility to visualise hidden defects in the CVD grown layers of TMD's via ultrasound assisted SPM, guiding the development of super-high quality TMD nanostructures.

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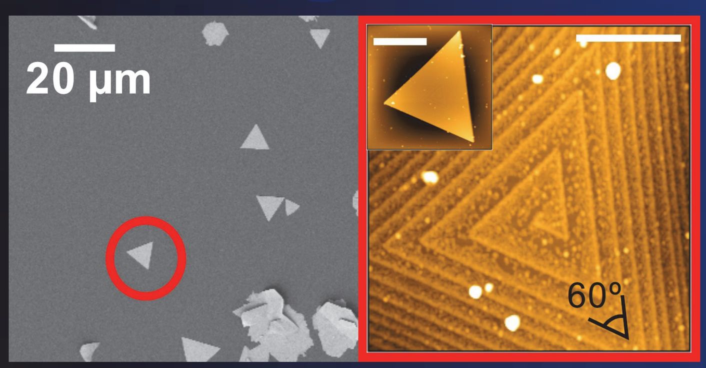
CVD growth

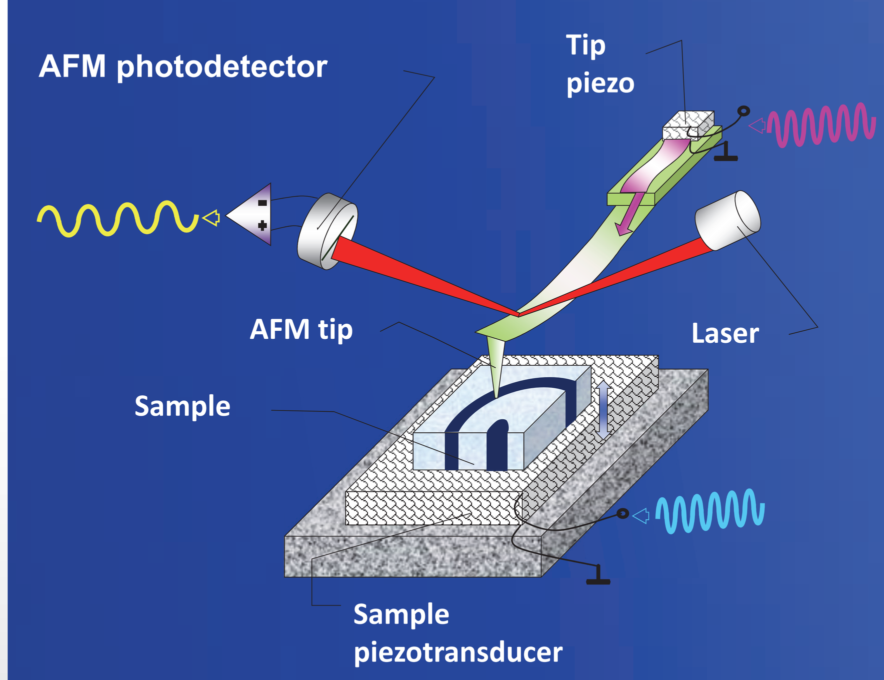


The chemical vapor deposition (CVD) growth of transition metal dichalcogenide (TMD) materials allows to achieve complex few-layer structures by manipulating the orientation of individual layers. These are usually linked via the screw dislocations connecting different layers of 2D crystal structures in spiral structures [1].

SPM methods

In order to sense the defects in the TMD growth we compare several SPM techniques combined with ultrasound - the Ultrasonic Force Microscopy (UFM), Waveguide) and the Heterodyne Force Microscopy (W-UFM and HFM) to identify the dislocations and faults between several stacked WS_2 layers [2].

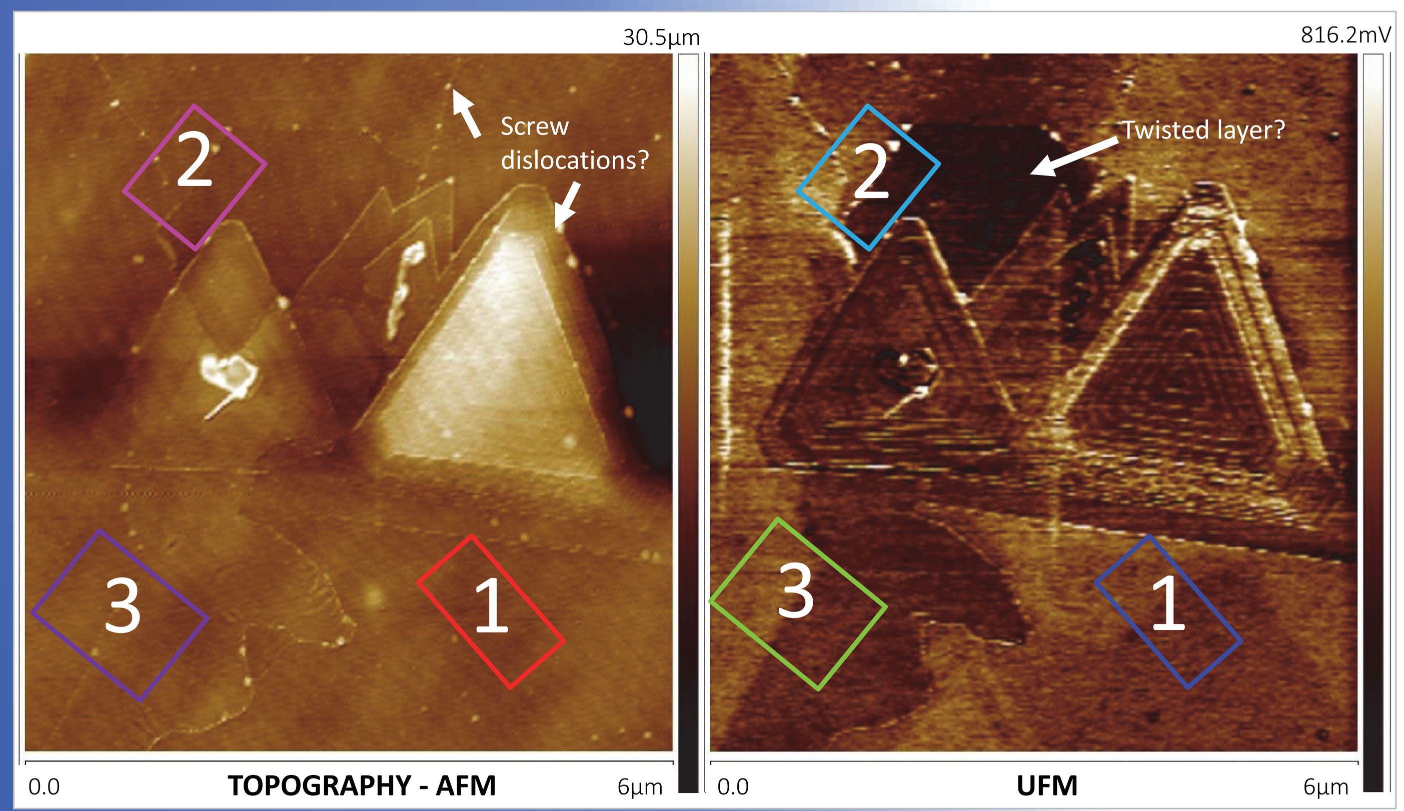
These techniques have been directly implemented in a modified Bruker Multimode microscope with modification allowing to excite the sample or the cantilever via piezoelectric drive at MHz frequencies much higher than the cantilever resonance, modulated at low kHz frequency. The cantilever inertia at the excitation frequency requires the tip to indent cyclically into the sample, resulting in the additional "ultrasonic" force due to tip-surface force–distance nonlinearity, detected at the modulation frequency resulting in images reflecting nanoscale maps of the sample stiffness [3].



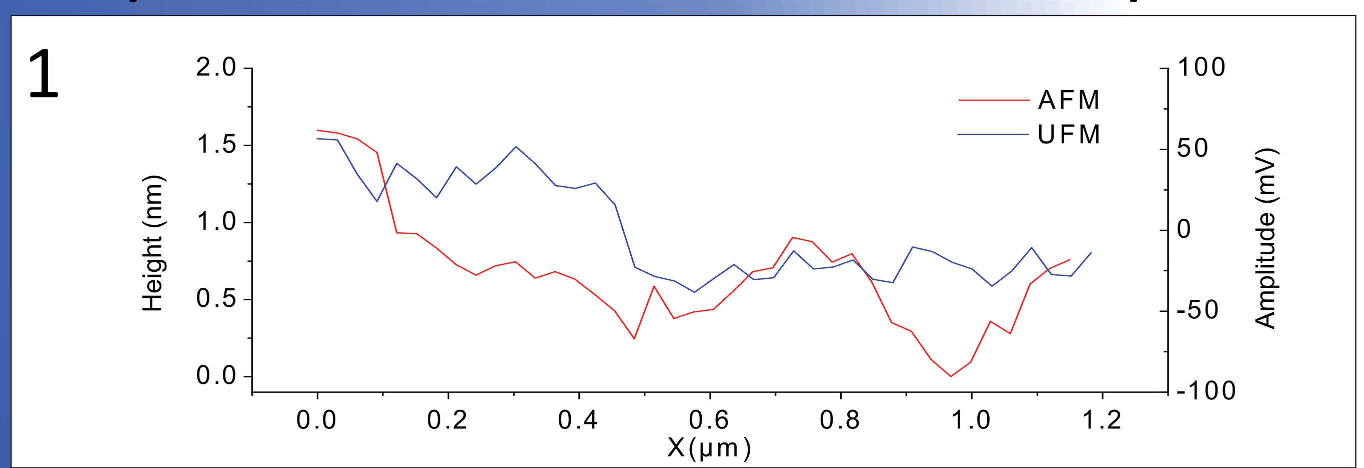
	INFO	SETUP		MODULATION
		Tip piezo exc.	Sample piezo exc.	
AFM	Topography	✗	✗	✗
UFM	Stiffness	✗	✓	✓
W-UFM	Stiffness	✓	✗	✓
HFM	Stiffness and dynamic relaxation	✓	✓	✗

AFM UFM W-UFM HFM Amplitude HFM Phase

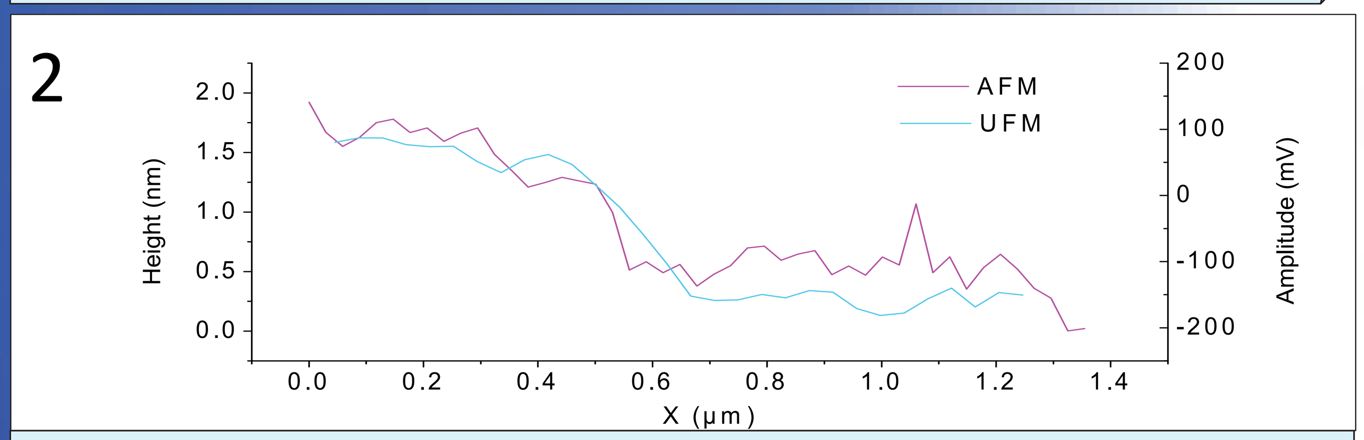
Results



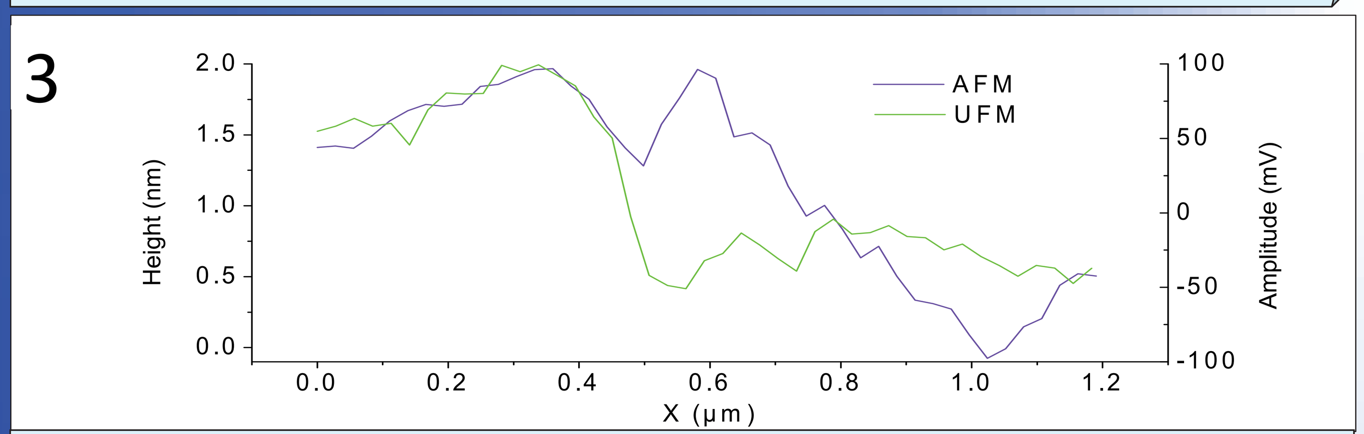
Comparison of AFM and nanomechanical UFM profiles.



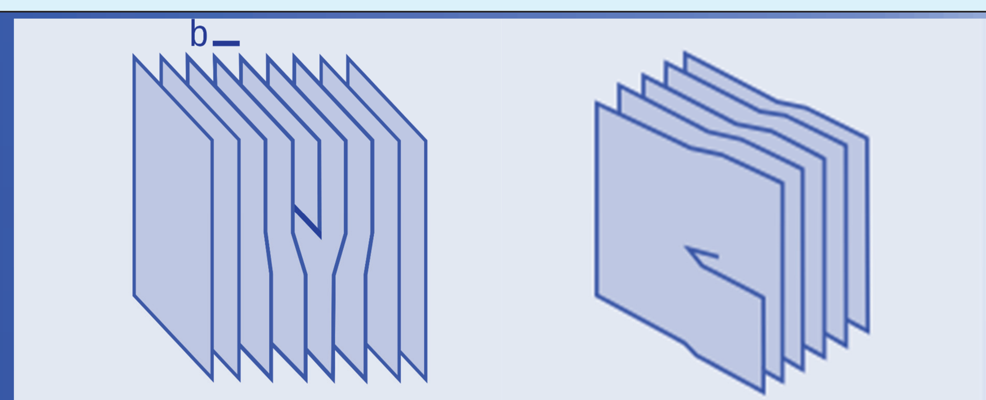
1. With no discernable topographic step, the nanomechanical contrast nevertheless reveals possible missing plane mis-orientation of the crystallographic axis of a hidden layers.



2. The topographical step corresponds to a few layers difference, and UFM also shows different stiffness in these areas.



3. There is a topographical dip, but no step with UFM revealing possible subsurface plane mis-orientation of the crystallographic axis and/or edge dislocation.



Edge (left) and screw (right) dislocations

Conclusions

- The nanomechanical contrast in UFM, W-UFM and HFM images 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.

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