Artifacts in AFM topography images of TMD monolayers

Initially motivated by work with graphene, the broad class of two dimensional (2D) materials has generated enormous interest. Monolayers and heterostructures of transition metal dichalcogenides (TMDs) have attracted intense investigation as the long awaited 2D semiconductor analogs of graphene. Due to their direct bandgap, TMD monolayers are extremely promising candidates for atomically thin electronic, optical, and photovoltaic applications.

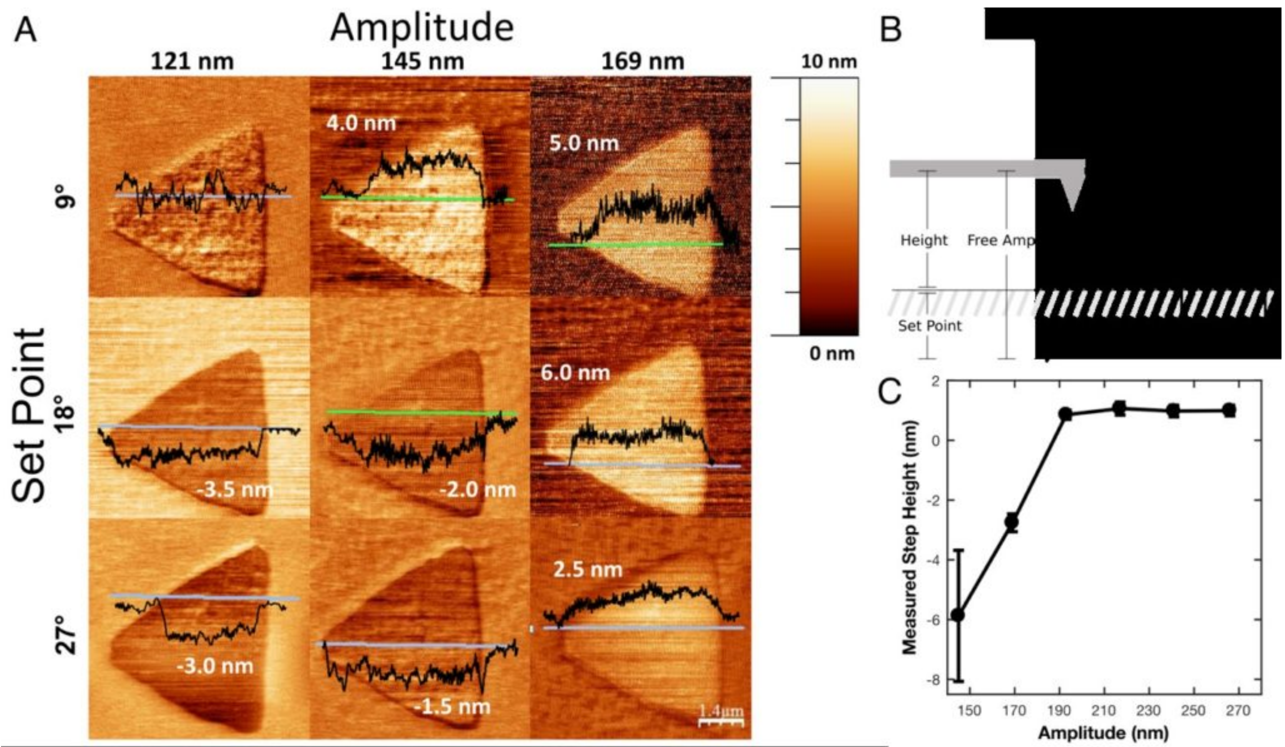

Fig. 1. Observed step heights for single crystalline WS2 monolayers grown on SiO2 using phase feedback based on set point and tip amplitude. (a) AFM topography scans of a single crystalline WS2 monolayer triangle grown on SiO2 with step heights. (b) The left side illustrates the AFM tip. The right side is an illustration of how the amplitude and set point change with height. (c) Step height vs. tip amplitude for phase feedback.

Before the possibility of using TMD monolayers in various applications, a comprehensive characterization of these materials is required. Atomic force microscopy (AFM) is able to characterize topography including loosely bound contaminants and adlayers through amplitude-phase-distance and force-distance curves, and has the additional potential for probing local electronic and magnetic properties. However, unwanted interactions between the tip and sample result in forces on the same order as the force from the property under investigation. While existing reports contain a range of results, anomalies in AFM scans have not been fully explored. The interaction forces between TMD and substrate have been known to affect the measured step height (or thickness) of TMD monolayers in AFM. Decoupling various forces for analysis requires a full understanding of the tip-sample interactions. Such a description of tip-sample forces and step height anomalies relating to TMDs has not yet been reported.

In our work, we provide a detailed look into the analysis of artifacts in AFM topography images of WS_2 monolayers including varied step height (WS_2 showing different thicknesses depending on the AFM settings) and contrast inversion (WS_2 appearing below the substrate). We describe our findings in terms of varying tip-sample interactions and capillary forces, and find that the step height depends strongly on the tip amplitude and set point. Furthermore, we prescribe tool settings to prevent contrast inversion and discuss “correct” TMD step height measurements, as we find our results applicable to other TMDs with similar wetting properties such as WSe_2 and $MoSe_2$.

Our key findings include 1) When present, capillary forces dominate measurement errors; 2) Removal of substrate water layers reduces the variation in height measurement from +/-6 nm to +/-0.5 nm; 3) When substrate water layers are present, contrast inversion is corrected by setting the AFM with high tip amplitudes and low set points; we show that high tip amplitudes reduce capillary forces; 4) When substrate water layers are absent, contrast inversion is corrected by setting the AFM with low amplitudes and high set points, which drive operation into the repulsive regime; and 5) Contaminants must be characterized through amplitude-phase-distance and force-distance curves, considering presence of surface contaminants and water layers.

This work represents a milestone in determining the “correct” height measurement in AFM, which cannot be determined from AFM topography images alone, but must be found through techniques such as force-distance characterization. Mr. Christian Cupo and Mr. Kyle Godin have experimentally led this project.

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