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Determining the Thickness of Atomically Thin MoS_2 and WS_2 in the TEM

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Transition metal dichalcogenides (TMDs) with formula MX_2, where M is a group 4-6 transition metal and X is a chalcogen, have captured immense research interest as a unique class of 2D materials with electronic and optical properties distinct from that of graphene and hexagonal boron nitride (h-BN) [1]. TEM has proven to be an excellent tool for atomic level characterization of TMDs using bright-field conventional TEM (BF-CTEM) [2], selected area electron diffraction (SAED) [3] and annular-dark-field scanning TEM (ADF-STEM) [4].

Most TEM studies of TMDs are supplemented by atomic force microscopy (AFM) to provide direct thickness measurements. However, the low throughput of AFM combined with the potentially small lateral size of TMD flakes makes this a time-consuming task. Thus, it is advantageous to determine the number of layers present only using TEM. One method is to observe the intensity variations during SAED tilting, as experimentally demonstrated on MoS_2 by Brivio et al. [3] for differentiating monolayers from multilayers. Another method analyzes the column-to-column intensity ratio of metal and chalcogen sites in ADF-STEM images [4].

In this work, multislice simulations [5] were used to analyze the reliability of ADF-STEM imaging and (SAED) for determining the thickness of 2H and 1T polymorphs of MoS_2 and WS_2 layers in the aberration-corrected TEM. Simulated ADF-STEM images and diffraction patterns were generated from samples of 1 to 4 layers tilted up to 500 mrad relative to [0001] axis about the $[10\bar{1}0]$ and $[11\bar{2}0]$ crystallographic directions.

Unlike similar studies of graphene and h-BN, simulated SAED patterns of monolayer MoS_2 (figure 1(a)) and WS_2 showed strong variations in spot intensity with tilt. Figure 1(b) tracks the intensity changes with tilt of the $(01\bar{1}0)$ diffraction spot and suggests that samples of 1-4 layers may be distinguishable by tilt. Furthermore, simulated ADF-STEM images of MoS_2 (figure 2) and WS_2 also showed unique distortions with tilt at each thickness. Results ultimately conclude that the combination of SAED and ADF-STEM should allow unambiguous identification of layer count up to 4 layers.

References

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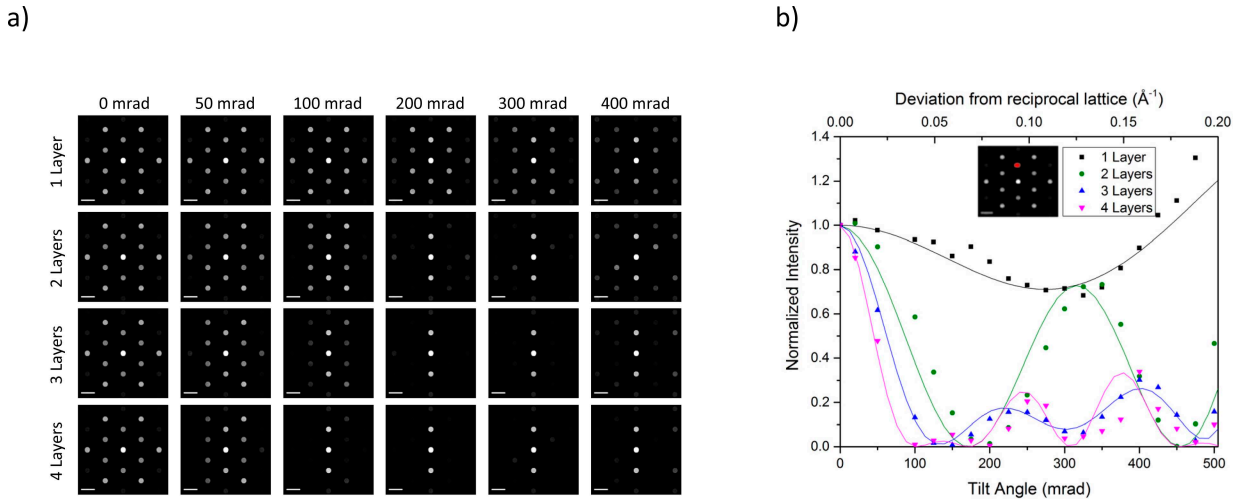

Figure 1. Simulated SAED patterns of 2H-M_0S_2 at various tilt angles. *(a)* x-tilt series. Linear intensity scaling; scale bars are 0.24 Å^{-1} long. **(b)** $(01\bar{1}0)$ diffraction spot intensity variations with tilt. Scattered points represent simulated multislice data; solid lines represent the simple kinematic model.

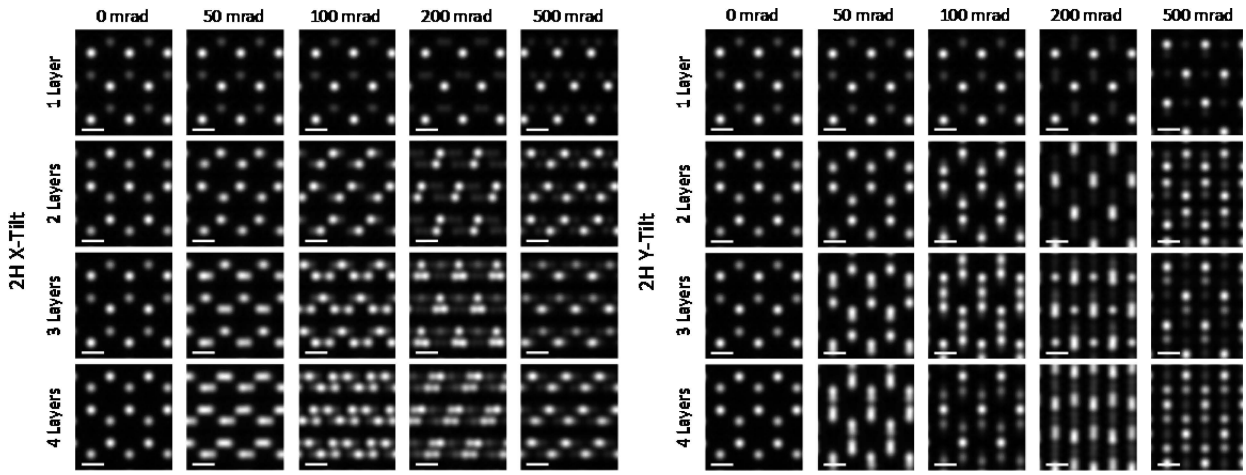

Figure 2. Simulated ADF-STEM images of 2H-MoS_2 at varying x and y-tilts. Both tilt directions produce unique intensity patterns at all thicknesses as a result of overlapping of Mo and S atoms. Linear intensity scaling; scale bars are 1.8 Å long.

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