103
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Characterization of Layer Thickness and Orientation of 2D WSe_2/MoS_2 Heterostructures using EDS, EBSD and AFM

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2D transition metal chalcogenides enable exciting new applications in electronic devices and show great promise to replace traditional silicon technology as functional building blocks [1]. However, in order to realize this potential there is a range of fabrication and integration challenges that have to be overcome and suitable, non-destructive characterization techniques are needed. Due to their high resolution, electron optical characterization in scanning electron microscopes (SEMs) and atomic force microscope is ideally suited. We show how a full structural and compositional characterization can be obtained by combining EDS, EBSD and AFM analysis.

The number of layers present in a 2D material is critical to its performance. As figures 1 and 2 show, we can obtain data of sufficiently high quality to non-destructively measure the number of layers in 2D MoS_2 and WSe_2 as well as from heterostructures containing both materials by processing EDS data obtained in the SEM. Figure 1 shows an SEM image of a flake of MoS_2 with two regions, one with two layers of MoS_2 and one with one layer as verified by step height measurements in the AFM and by Raman spectroscopy. EDS spectra acquired from the two different regions show a clear difference in the peak height of the overlapping Mo L-lines and S-K lines. The difference can be quantified by processing the data in a special software designed to calculate the thickness of thin films on substrates (AZtec LayerProbe) [2]. For the calculation, a density of 5.06 g/cm^3 was assumes for MoS_2. The resulting values shown in figure 1 correspond well to a theoretical interlayer distance of 0.65nm.

In order to test whether this method is also suitable for heterostructures of 2D materials, we obtained measurements from a sample where a flake of MoS_2 had been transferred onto a flake of WSe_2. In the region of where the two flakes overlap, Raman spectroscopy showed that while there is only a single layer of WSe_2 present, MoS_2 occurs in one layer and two layers. Figure 2 indicates the different regions of interest on the sample. As the W-M line overlaps closely with the Si-K line, the Se-K line was used for the layer thickness measurement. The results in figure 2b show that both the WSe_2 layer and the MoS_2 layer thickness can be accurately determined.

We also show that Kelvin Probe force measurements (KPFM) can be used to image the contrast between different layer thicknesses in both single layers and heterostructures (figure 2c). Further work is necessary to determine whether the work function measured by KPFM can be quantified.

In order to add crystallographic data revealing misalignment between flakes, we can use EBSD. IPF maps of an area that contains several flakes of exfoliated MoS_2 clearly indicate significant misalignment between some of the flakes (figure 3). This may aid the understanding of the exfoliation process which is still widely used to produce 2D materials for research purposes.

Our results indicate the great potential of SEM and AFM for the characterization of devices based on 2D materials and indicate avenues of further work to establish them as means for failure analysis and production quality control.

References:

[1] S.Z. Butler *et al*., ACS Nano **7** (2013), p. 2898.
[2] C. Lang *et al*., Microscopy and Microanalysis **19** (2013), p. 1872.

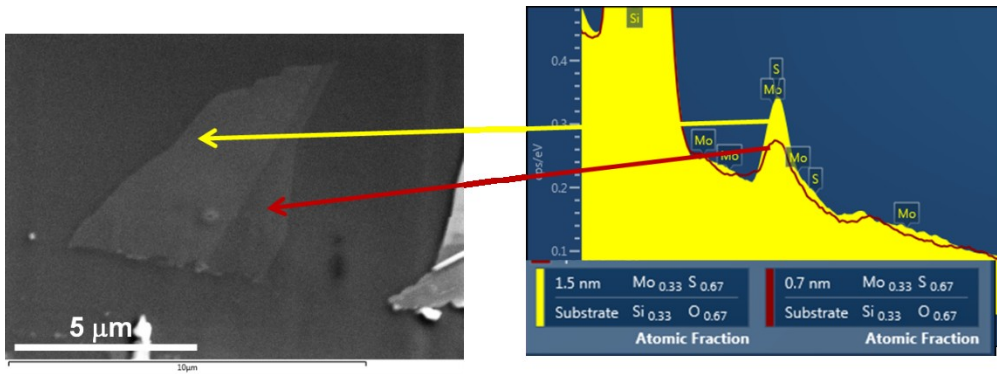

Figure 1. A flake of MoS_2 on an SiO_2 substrate with EDS spectra and resulting layer thicknesses.

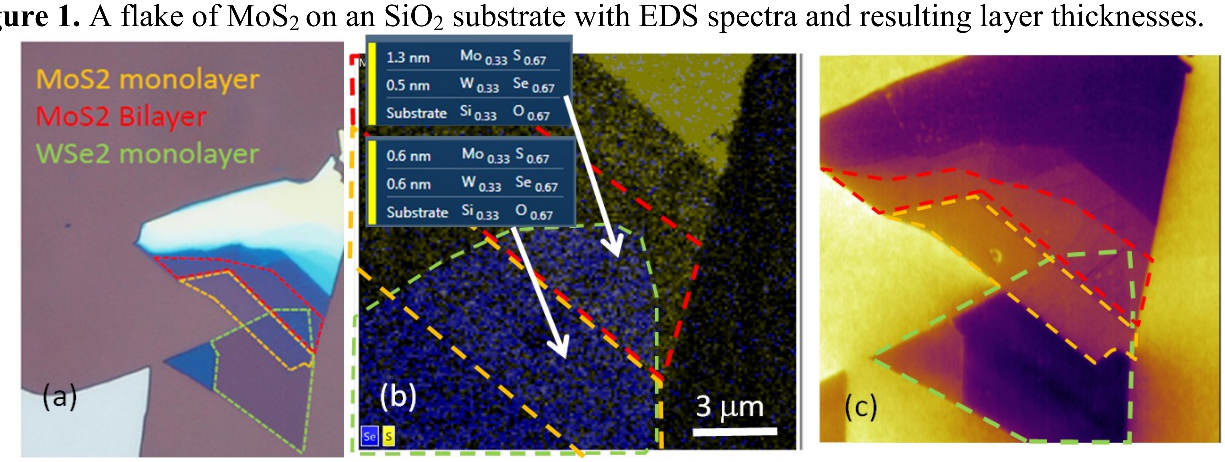

Figure 2. (a) optical micrograph of a MoS_2/WSe_2 heterostructure. (b) EDS maps at 4kV of the overlap regions indicated by dotted lines and (c) KPFM images of the overlap regions.

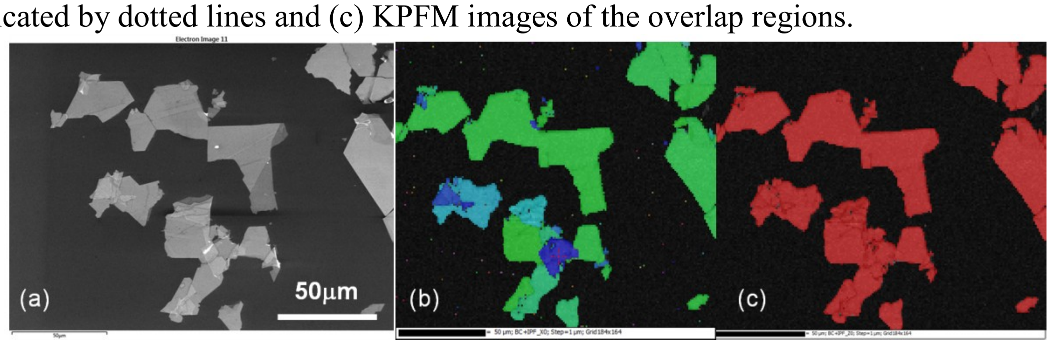

Figure 3. (a) Electron image and (b) IPF x map and (c) IPF z map of MoS_2 flakes on SiO2 indicating only in-plane rotational misalignment of the flakes.