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Mapping the 3D Structure of Corrugated “Cardboard” MoS_2

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Building 3D structures from 2D materials through folding opens new possibilities in nanodevice fabrication [1]. Origami scales well from folding regular pieces of paper down to folding atomically thin sheets. The use of transition metal dichalcogenides (TMD) like MoS_2 bring additional functionality through their material properties beyond the geometry of the folded 3D object. This creates the possibility of cell-sized devices that can transform, move, and communicate. However, it is technically challenging to construct functional 3D structures merely with flat sheets of a 2D material owing to its low bending stiffness. Thus, we have grown atomically-thin corrugated MoS_2 with bumps around 50 nm in diameter and 20 nm in height. Since single-crystal 2D materials are intrinsically flat, the key question is to understand how curvature is accommodated and achieved at a microscopic level.

Characterization of a bumpy 2D material presents an interesting challenge in electron microscopy. A typical HAADF image at zero-degree tilt is unable to see the corrugations in projection (Fig. 1b). TEM tomography provides a simple start and a general idea of the 3D structure of the material (Fig. 1a). However, such a corrugated structure must have many different strains and crystal orientations as well as topological defects. The ability to map these features over a large field of view is difficult for traditional STEM imaging.

Instead, we perform quantitative analysis by collecting the full diffraction pattern at each scanning point using the Electron Microscope Pixel Array Detector (EMPAD) developed at Cornell [2]. It has a readout time of 0.86 ms/diffraction pattern, allowing a full 4D dataset to be recorded in minutes, minimizing the effects of drift and sample damage. Its large dynamic range of 1 to 1,000,000 electrons per pixel allows us to capture all the details in the central bright disk while also capturing the significantly weaker diffraction spots, allowing for sub-pm precision in lattice constant maps [3]. Figures 1c-f show that a variety of different diffraction patterns are present in the sample indicating different strains, tilt, and rotations.

A combination of peak-finding and center-of-mass algorithms allows automatic mapping of the location of the diffracted disks [3]. Measuring the distance of the diffracted disks from the central beam creates a projected lattice constant map (Fig. 2a), noting that the tilt of the corrugated surface dominates the disk displacement rather than a strain in the lattice. This map makes the corrugations visible even at zero-degree tilt. Furthermore, measuring the rotation of the diffracted disks maps the grain orientations (Fig. 2b). Overlaying these two maps (Fig. 2c) shows how the corrugated structure creates frustrations in the material, with grains often assembled in patches around a corrugation. This results in much smaller grains on the curved surfaces compared to the flat substrate [4].

References:

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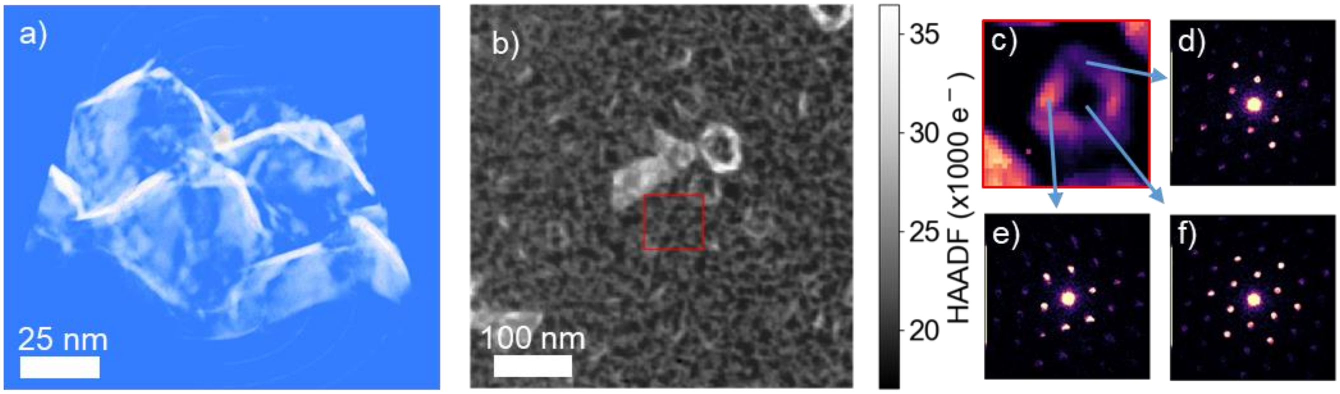

Figure 1. a) TEM Tomographic reconstruction shows the three-dimensional structure of the MoS_2 corrugated monolayer. Tomography data set was taken at 80 keV to avoid knock-on damage. Corrugations are around 50 nm in diameter and 20 nm in height. b) HAADF image of corrugated MoS_2 material at zero-degree tilt is not sensitive to the corrugated bumps. c) EMPAD projected lattice map of one particular corrugation taken from the region in the HAADF image boxed in red. The projected lattice map is sensitive to the tilt of the corrugated surface, which allows us to see the bumps. Blue arrows indicate approximate areas where d,e,f) diffraction patterns were collected. The diffraction patterns show an array of crystal tilts and orientations are present in just one corrugated feature.

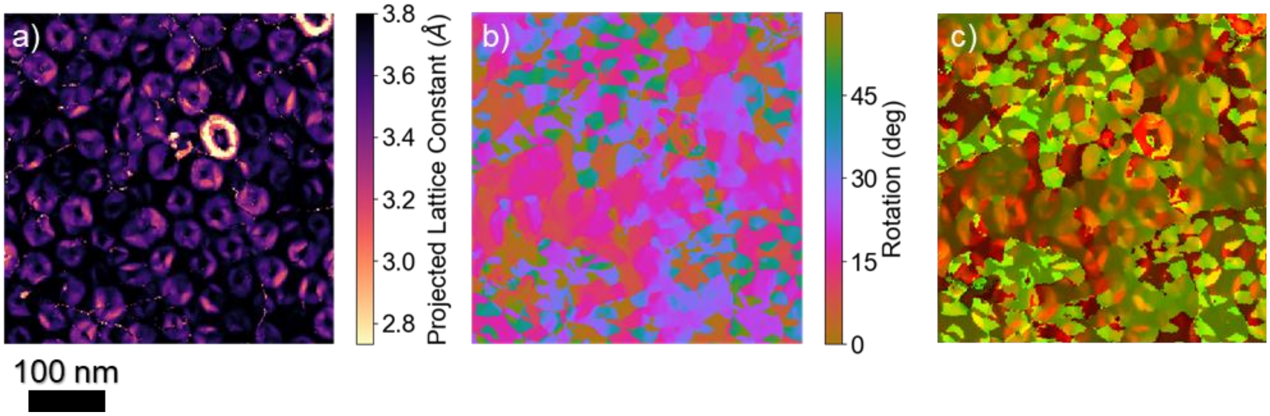

Figure 2. Mapping the location of the six primary diffraction spots from the diffraction patterns as shown in Fig. 1d-f generates projected lattice constant and rotation maps. a) Map of the projected lattice constant made by measuring the distance of the diffracted disk clearly shows corrugations not visible in the HAADF. Most of the change in lattice constant is dominated by the tilt of the corrugated surface. b) Measuring the rotation of the diffracted disks also creates a rotation map showing the variety of crystal orientations present in the material. c) Overlay of Fig. 2a in red and Fig. 2b in green shows that many of the changes in crystal rotation are centered about the corrugations. The HAADF, projected lattice constant, and rotation maps were all made in post-process from one 4D diffraction dataset collected on the EMPAD at 120 keV with a 1.35 mrad probe forming aperture.