Tracking Atoms in Two-Dimensional Materials Using Scanning Transmission Electron Microscopy

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Abstract:

Layered transition metal dichalcogenides (TMDs) possess unusual physical properties, such as superconductivity and metal-to-insulator transitions, that strongly depend on the structure of the atomic lattice. In tantalum ditelluride ($TaTe_2$), a structural phase transition occurs below 150 K, as indicated by x-ray diffraction and bulk resistivity measurements, but the precise microscopic structure is not well understood. Here, we use scanning transmission electron microscopy (STEM) to map the structure of $TaTe_2$ with atomic resolution. We perform measurements at room and liquid nitrogen temperatures, and at each of these temperatures the material is imaged from two different lattice orientations. We use fast Fourier transforms (FFTs) of the lattice images to extract periodic lattice information, and compare the high and low temperatures to determine the structural phase change. Atomic mapping of this structural phase change can help to understand and utilize the exotic properties of $TaTe_2$.

Introduction:

A Titan Themis CryoS/TEM is used at 300 kV to scan a narrowly focused electron beam across a thin sample of material. Electrons scattered from the sample are recorded on an annual detector at each scan position to produce a two-dimensional (2D) image. The STEM can image materials with atomic resolution, and the intensity of the image will scale with atomic number. Using this, a structural phase change in $TaTe_2$ at 150 K can be investigated.

These projection images will only give 2D information about the material, so two different lattice orientations are chosen to give a more complete picture of the structural changes. The plan-view orientation is a top-down view of the material 90-degrees rotated from the b-axis, and the cross-sectional orientation is along the b-axis where the gaps between the individual layers of the material are visible. The structural change in $TaTe_2$ is subtle, and requires high precision STEM imaging. Averaging over the entire field of view, the FFT of an image will have intensity peaks that correspond to its periodic structure. Because of this, FFTs are used to determine structural changes between two images; a more complex FFT implies a more complex physical structure.

Sample Preparation:

In preparation for STEM imaging the samples have to be thinned to electron transparency. To view the desired orientations, the $TaTe_2$ must be prepared for the microscope such that the imaging plane is orthogonal to the electron beam (Figure 1). The plan-view orientation is obtained through a standard mechanical exfoliation method, where flakes of $TaTe_2$ are exfoliated onto PDMS. A thin flake is selected using an optical microscope, and is then stamped down onto a Si_3N_4 electron microscope grid. The system is then heated to around 76°C and the PDMS is peeled away slowly, leaving the flake on the grid. The cross-sectional orientation requires the use of a focused ion beam. Gallium ions are used to mill trenches through a thin film of $TaTe_2$, which is then removed using a needle and then transferred to a microscope grid.

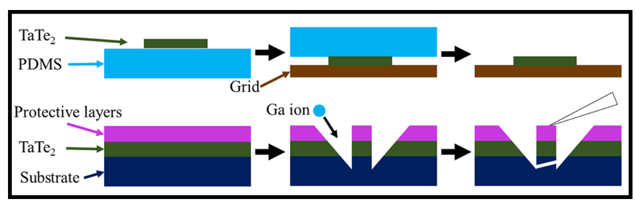

Figure 1: Graphics of sample preparation for plan-view (top), and cross-sections (bottom).

STEM Imaging:

$TaTe_2$ is imaged in plan-view at room temperature and cryogenic temperature (Figure 2). The tantalum atoms are slightly misaligned in their stacking, leading

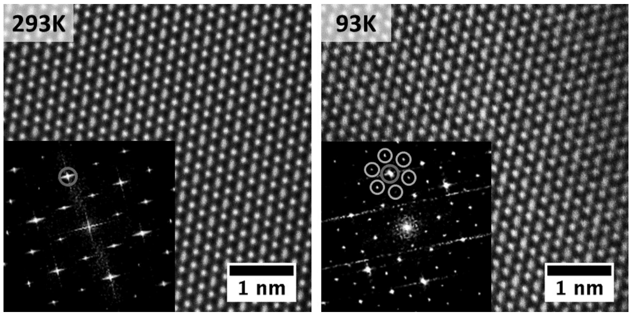

Figure 2: Plan-view STEM images of $TaTe_2$ with FFTs at room and cryogenic temperatures.

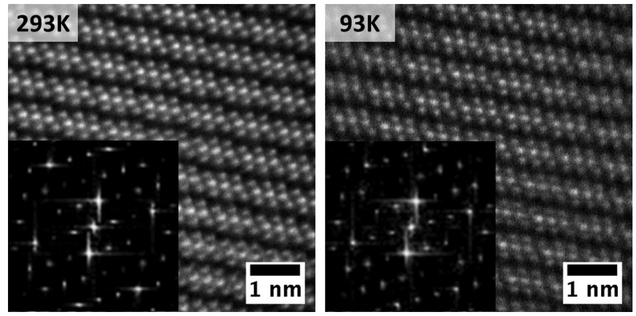

Figure 3: Cross-sectional STEM images of $TaTe_2$ with FFTs at room and cryogenic temperatures.

to an elongated shape, and the tellurium atoms form a hexagonal ring around the tantalum. Imaging at cryogenic temperatures is difficult, due to increased sample drift and the occasional bubbling of the liquid nitrogen. This causes the low temperature images to appear slightly noisier, but atomic resolution is still achieved. Looking at these images, it is not immediately evident that there is a structural change in the material. When comparing the FFTs, each has strong peaks which correspond to the symmetry of the structure, but the cryogenic image shows additional satellite peaks, indicating a structural phase change not easily seen in the real-space images.

The cross-sectional prepared $TaTe_2$ is also imaged at room and cryogenic temperature (Figure 3). The individual layers of the material are distinct and stacked on top of each other. The layers themselves are comprised of rows of tantalum with tellurium on top and bottom. Like the plan-view orientation, the structural change is not clear from the images. Additionally, the structural change is also not visible in the FFTs. This could imply that the structural change is not visible from this orientation, meaning the modulation is occurring along the axis of the electron beam. Looking closely, the tantalum in the center of the layers have larger spacings between every three atoms, which appear to decrease in intensity from left to right. By taking line profiles of several groups of three in each image, the intensity can be averaged and normalized to the far-left peak (Figure 4). The average intensity of the far-right atom is 96% of the far-left atom at room temperature, compared to 88% at cryogenic temperature. This subtle change might be due to a structural change that is not visible in the real-space image or its FFT.

Conclusions and Future Work:

STEM enables atomic resolution imaging of 2D materials to better understand their structure. These experiments show a structural phase transition of $TaTe_2$ in two different orientations at cryogenic temperatures. In the future, $TaTe_2$ will be imaged in different orientations so that the modulation can be extracted from the real-space images. These results will help to build a complete picture of this structural phase, allowing for better utilization of $TaTe_2$ properties.

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References:

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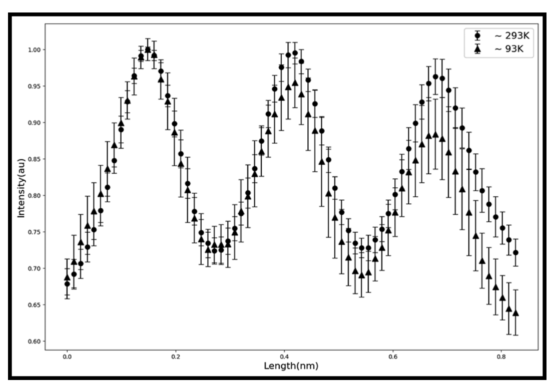

Figure 4: Average intensity across tantalum atoms in groups of three.