

Probing 2-Dimensional Quantum Materials at the Atomic Scale with Scanning Transmission Electron Microscopy

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Abstract:

Scanning Transmission Electron Microscopy (STEM) allows us to image materials at the atomic scale to study their structure and properties. Our goal is to create a platform with which we can tune materials by applying electrical bias to manipulate their properties. In this study we focus on the 2D material 1T-TaS₂, which was exfoliated to create a thin sample which was imaged using scanning transmission electron microscopy and can be tuned *in situ* with an electrical bias to drive a charge density wave phase transition.

Introduction

Scanning Transmission Electron Microscopy (STEM) is a technique that uses an electron beam to shoot electrons across a sample point by point in a raster pattern which are collected by a detector and formed into an image. This technique allows for properties and structures of materials to be studied. Properties of certain materials can be tuned and manipulated when biasing is applied. Atomic resolution imaging has been improved to be able to see atoms at a resolution of 0.39 Å [1]. 1T-TaS₂ properties have been studied previously. 1T-TaS₂ is a 2D material held together by Van der Waals forces vertically and by covalent bonds horizontally. It is known that temperature can be applied to drive charge density wave (CDW) transitions of 1T-TaS₂. At room temperature, 293 K, 1T-TaS₂ is in the nearly commensurate phase, but when cooled with liquid nitrogen to ~ 95 K it transitions to the commensurate phase [2]. With an applied bias range of an electric field a constant temperature ~ 90 K, many different CDW transitions can occur. With an applied electric field between 3 and 15 kV/cm, a commensurate to near commensurate transition occurs. With higher applied electric fields at the same temperature, a nearly commensurate to incommensurate transition and incommensurate to a metallic phase transition occurs [3].

Methods:

To be able to image and study 1T-TaS₂ a thin sample must be prepared. To get a thin flake, the material is exfoliated using scotch tape. The samples are transferred from the scotch tape to the gel polymer PDMS which is inspected under an optical microscope. A thin flake is then selected and stamped down from the PDMS to a Si₃N₄ TEM grid. The grid is then inserted into the STEM microscope to be further imaged.

Results:

Images using high angle annular dark field (HAADF) STEM were taken of a thin flake of 1T-TaS₂. Figure 1a shows a photo taken of the flake using the optical microscope. The thin flake was stamped down over a hole on the Si₃N₄ TEM grid to allow electrons from the electron beam to pass through the sample in the STEM microscope. Figure 1b shows an overview image of the hole with the thin sample focused on to be further imaged at the atomic scale. Figure 2 shows this imaging

of 1T-TaS₂ where there is a visible edge of thin

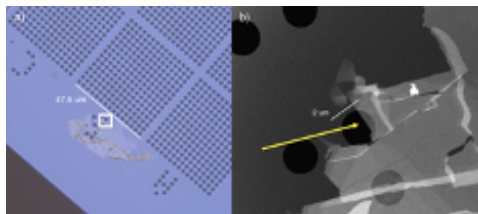


Figure 1: a) A photo of a thin flake of 1T-TaS₂, taken from an optical microscope. b) Closer overview image of thin sample taken using STEM.

material. This image shows individual atoms of 1T-TaS₂. These images were taken at room temperature and with no applied bias. Figure 3 shows the diffraction

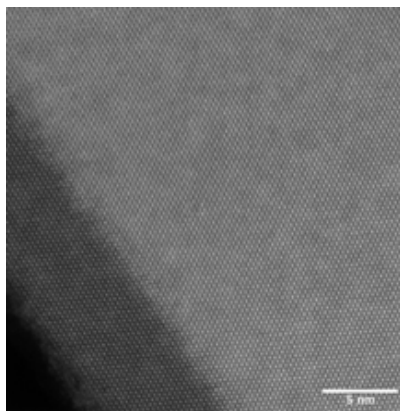


Figure 2: Atomic resolution STEM image of 1T-TaS₂ at room temperature.

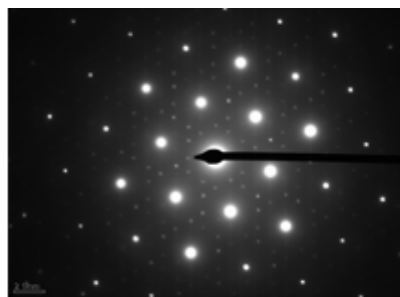


Figure 3: Diffraction pattern of 1T-TaS₂ at room temperature

pattern of the material at room temperature. The first order peaks are slightly visible while the second order peaks are much brighter. This image is also in the nearly commensurate phase. In order to characterize the thinness of the flake, a position averaged convergent beam electron diffraction (PACBED) pattern was acquired, shown in figure 4a, and compared to PACBED simulations, for different thicknesses of 1T-TaS₂ (figure 4b) [4]. Because the PACBED simulations used a slightly different convergence angle, the overlap in diffraction disks differs. However, we still can use the simulations to make a rough comparison that the sample

flake imaged is about 4.72 nm or 5.32 nm thick.

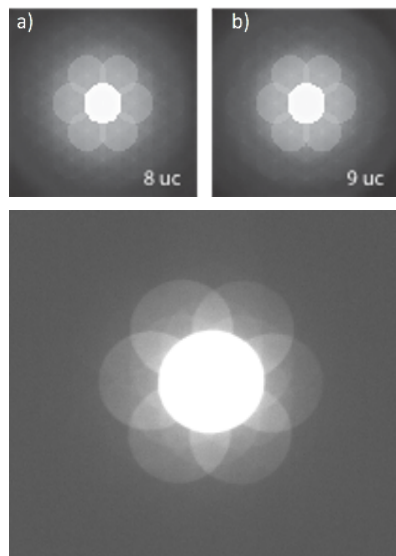


Figure 4: a) PACBED pattern from 1T-TaS₂ flake, same region as Figure 2. b) PACBED simulations used to compare sample thickness [4].

Future Work/Conclusion:

1T-TaS₂ was imaged on a Si₃N₄ TEM grid but has not yet been successfully stamped onto a four contact biasing chip. Once a thin flake is able to be stamped onto a chip, it can then be inserted into the STEM microscope with an applied bias to physically see how 1T-TaS₂ properties change under these conditions.

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References:

- [1] Courtland, R. *Nature* **563**, 462-464 (2018)
- [2] Hovden, R., Tsen, A,... Proceedings of the National Academy of Sciences Oct 2016, 113 (41) 11420-11424
- [3] Geremew, A., Rumyantsev, S,. ... ACS Nano **2019** 13 (6), 7231-7240
- [4] Hovden, R., Liu, P., . . . (2018). Thickness and Stacking Sequence Determination of Exfoliated Dichalcogenides (1T-TaS₂, 2H-MoS₂) Using Scanning Transmission Electron Microscopy. *Microscopy and Microanalysis*, 24(4), 387-395.

