Probing Interfaces between Two-dimensional Van Der Waals Materials with Scanning Probe Microscopy

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1 Introduction

Atomically thin materials have been studied for decades despite a small amount of information known about them. These materials include graphene, hexagonal boron nitride (hBN), black phosphorous, and many more. Graphene has free moving electrons in the z axis, allowing for unconventional electronic properties that can revolutionize nanoelectronics, Many times, a material, such as gold, will be placed in-between the two van der Waal materials as an additional metal to further characterize these unconventional materials. When the two materials lay on top one another, the top layer can be rotated to cause Moiré lattices to occur. These lattices take place due to the mismatch of the materials' lattice structure. The Moiré lattices creates an alternate connection between the two van der Waal materials being observed. If the interaction between van der Waal materials can be better understood, it could revolutionize nanoeletronics. [1]

2 Procedure

AFM, or Atomic Force Microscope, is a type of scanning probe microscopy that allows one to image surface topography of the van der Waal materials. An additional probing method is EFM, Electrostatic Force Microscope, which allows for the probing of surface potentials and electrostatic forces of synthesized samples. These materials take place on the nanoscale, therefore human interaction tend to interfere with the data. In this experiment, I will attempt to counter this roadblock by using the scanning probe microscopy techniques mentioned above. Throughout the summer, I have built 2D layered materials from van der Waal materials and have used AFM and EFM to image the surface topography as well as characterize their surface potentials, capacitance, and charge coupling.

2.1 Creating Heterostructures

A heterostructure forms when van der Waal materials lay horizontally on each other. AFM allows the surface of the formed heterostructure to be analyzed and its surface potentials, capacitance, and charge coupling to be measured via EFM. For this experiment, a tri-layer heterostructure consisting of boron nitride (hBN) as the top and bottom layers and gold (Au) as the middle layer will be synthesized.

2.2 Materials

Listed will be all the materials needed to create a heterostructure: scotch tape, microscope, glass microscope slides, adhesive tape, tweezers, razor blade, silicon dioxide (SiO_2) wafers, boron nitride, and gold pellets.

2.3 Van Der Waal Transferring

Cut off a piece of scotch tape and place boron nitride onto it. Fold the tape in half onto itself. This spreads the material around the tape, allowing thin layers to be placed on the wafer. After the compound is thinned onto the tape, place the tape on the wafer and apply enough pressure to ensure the hBN flakes are transferred to the SiO_2's surface. This process needs to be repeated for addition silicon wafers. At least two wafers with hBN need to be used.

After all the wafers have been coated with hBN, gold needs to be added to a select amount of wafers. The E-Beam is used to evaporate the gold onto the surface of the wafer. A tray of SiO_2 wafers coated with hBN are placed upside down on the circular tray at the top inside the instrument. The gold pellets is then placed at the bottom inside the instrument and evaporated onto the surface of the wafers. This process takes an entire day to complete.

2.4 Creating Polydimethylsiloxane Stamps

Stamps are used to capture the the flake of interest, making it the top layer of the heterostructure. This process can be done by using Polydimethylsiloxane (PDMS). This is a silicon-based organic polymer that is heated to capture the flake of interest, as well as the flakes surrounding it, then cooled to ensure the flakes stay stuck on the surface of the polymer.

To create a stamp, two strips of orange tape are placed vertically on the glass microscope slide. Apply pressure via tweezers to ensure the adhesion between the glass and tape is secure. Use the tweezers to peel the plastic off the tape, leaving the adhesion substance exposed on the glass slide. Next, a three-inch piece of scotch tape is cut off and a small square section is cut from the tape. Place the tape vertically over-top the adhesion substance along the glass slide, having the square section of tape near the edge of the slide. Lastly, a small piece of polymer is placed inside the square hole on the tape, completing the stamp.

2.5 Finding Flakes of Interest and Stamping

After the above preparations have been complete, all SiO_2 wafers and PDMS stamps can be taken to the microscope to create the hBN-AU-hBN heterostructure. The hBN wafer will be the first under the microscope since it is the top layer. Once at the microscope station, a vacuum tube and heating pad are used in coordination with the wafer to create a seal to stabilize the wafer. The wafer is placed over-top the suction area with the ceramic heating pad under the wafer. Once complete, focus the lens on the surface of the wafer. If done correctly, boron nitride flakes are seen with many having an orange or yellow color. Thin flakes need to be found. They are very feint and have a blue tint to them. After a thin flake is found and pictures of the flake are taken, the stamp is mounted to the aligner instrument and arranged with the light shining through the polymer and onto the sample. The microscope is refocused and the stamp is lowered on the silicon wafer's surface.

Once the stamp is lowered on the silicon wafer's surface, the ceramic heater is set to 150 degrees Celsius. After it has reached approximately 150 degrees Celsius, the stamp is lifted upward and the heater is disabled, cooling the polymer, encasing the flake of interest and the surrounding flakes inside the PDMS.

Now that the soon-to-be top layer of the heterostructure is captured in polymer, the Au-hBN wafer can be inspected. The wafer-heater-suction coordination, lens focus, and finding a thin flake are all repeated. When looking for a flake of interest on this sample, the flake must be thin and have gold on its surface. When the flake is found, the stamp is lowered on the surface of the sample with the top layer aligned on the newfound flake. Once aligned, the heating pad is heated to 170 degrees Celsius and lowered onto the wafer's surface. Once contact is made, the stamp is lifted and the heater is turned off to cool. The top layer of the stamp transferred to the wafer's surface.

The new wafer should look similar to this image:

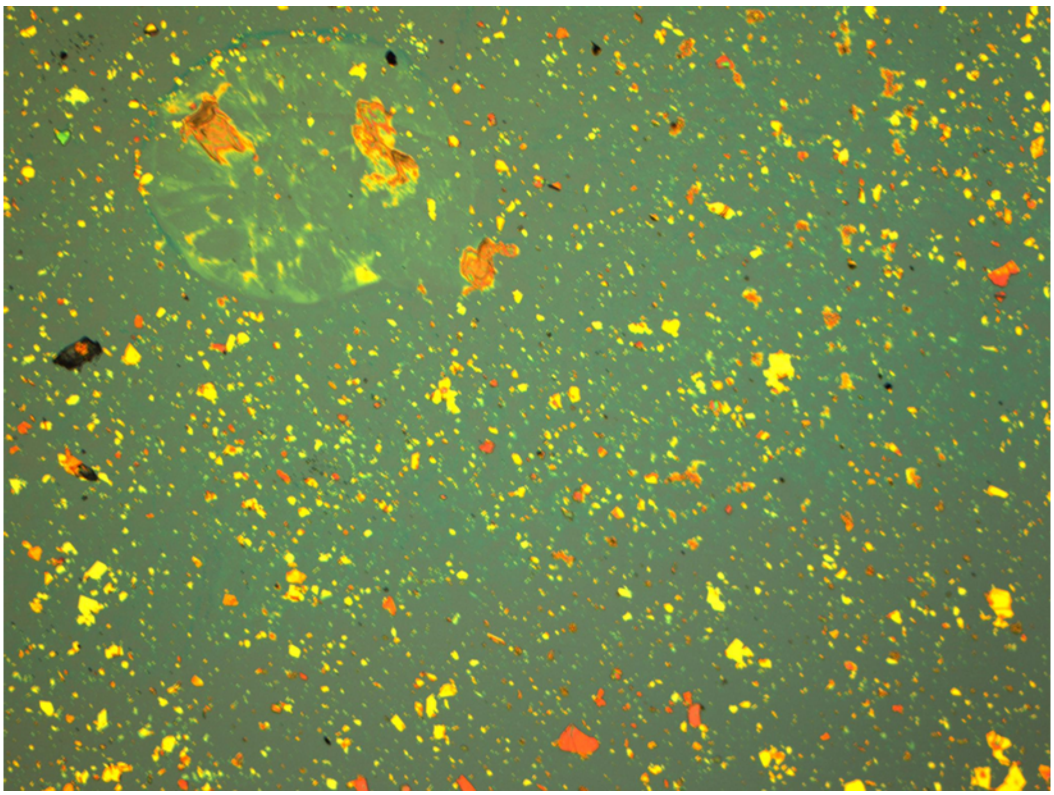

Figure 1: End Result

To ensure the creation of the heterostructure, focus the microscope on the surface and identify the two flakes of interest have intersected each other. Once confirmed, the wafer sits in chloroform over night to remove the PC layer. Once removed, it is taken to the Atomic Force Microscopy instrument for further evaluation.

2.6 Probing Heterostructure

The hood of the AFM instrument is opened and the top, metallic head is removed. Under the head houses the cantilever and tip. Replace the tip if needed. Place the wafer on the sample board and place the head over-top the wafer, fitting in the three notches. Open the AFM software and configure the settings. The head emits a laser which needs to be aligned with the tip on the cantilever. If done correctly, the deflection should be close to zero. Zoom in on the wafer and adjust the focus until the flakes are visible. The area of interest contains the heterostructure. Adjust the X and Y plane until the heterostructure is located. Once located, fit the area of interest over the heterostructure and begin probing. This process takes approximately thirteen minutes to complete.

Once the topographic image is complete, the heterostructure's surface potentials, capacitance, and charge coupling are measured. The tip is induced with several different voltages, making this process several hours long. These voltages include: 250mV, 500mV, 750mV, 1.00V, 1.50 V, 2.00V, and each of their respective negative values. Once the data from each source is collected, it can be analyzed.

3 Analyzing and Visualizing Data

Phase shifts of heterostructures are the most important data points when collecting EFM measurements. Surface potentials, capacitance, and charge coupling are all measured based on phase shifts. The phase shift, represented by φ, is already calculated through the software, however the phase shifts from the sample and background, denoted φ_S and φ_B respectively, need to be subtracted from each other to insure correct data. φ is calculated by using Equation 1.

$$\tan(\varphi) = \frac{\omega\gamma}{\omega_0^2 - \frac{F_P'(x_0)}{m} - \omega^2} \tag{1}$$

Where $F_P'(x_0)$ represents the interacting force between the AFM tip and the sample and substrate, ω_0 is the oscillator's resonate frequency, and m is the mass of the oscillator.

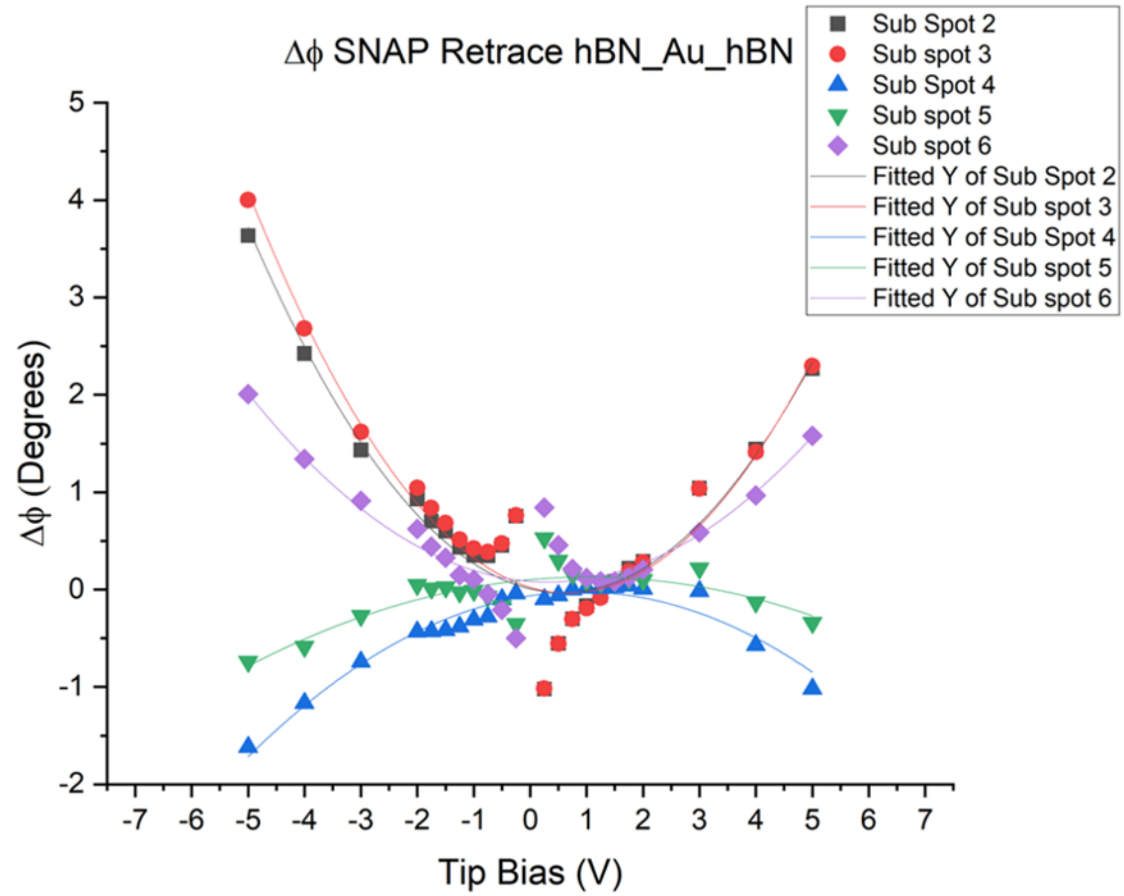

Figure 2: $\Delta\varphi$ SNAP Retrace hBN-Au-hBN

The resulting data should form a positive or negative parabola depending on the materials being used, however, the resulting graph from this heterostructure is reciprocal. At this moment, what is causing this disruption in data is unclear. A leading idea is the numerator and denominator need to flipped, causing the function to be cotangent in Equation 1.

Acknowledgements

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References

[1] Kaustav Banerjee Pulickel Ajayan Phillip Kim. “Two-dimensional van der Waals materials”. In: *Physics Today* 69 (Sept. 2016).