Scanning Tunneling Microscopy

Elise Jortberg, Lab Partner: Dena Guo
Northeastern Department of Physics
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I. INTRODUCTION

Scanning tunneling microscopy (STM) images surfaces of materials at sub-angstrom level resolution by applying a bias voltage to a sharp tip very close to the sample. The bias voltage allows an electron to tunnel across this potential difference between tip and surface without the two ever making direct contact. The tip experiences a current as a result of the electron transfer. This current changes in response to the local density of electron states as the tip moves across the material. The resulting image from tracking tunneling current as a function of position can measure atomic scale features of a surface, such as geometric patterns, lattice constants, and bond lengths. Binnig and Rohrer developed this technology while at IBM Labs (Physical Review Letters, 1982) and earned the Nobel Prize in Physics in 1986.

We used a NanoSurf NaioSTM to capture STM images of the surface of graphite. We prepared the STM by sterilely making the scanning tip, cleaning the graphite, and aligning the sample. We carefully approached the tip the the sample, making sure that the tip never came into contact with the surface of the graphite. Once we had a sufficiently sharp tip and found a good section of the graphite to scan, we routinely zoomed in until we reached a nanometer sized image. From this high resolution image, we observed the hexagonal pattern of graphite and a lattice constant of 2.10 ± 0.15 angstrom. We then captured the I-V curve of our apparatus using the scanning tunneling spectroscopy mode to ensure the system was well calibrated for sub-angstrom resolution.

II. BACKGROUND THEORY

STM images measure tunneling currents because of the quantum mechanical phenomenon of electrons tunneling between surface and tip. Classically, electrons need a connected medium to travel across a given barrier (in this case space between sample surface and tip). Electrons are subject to wave-particle duality. The probability of the electron's position is described by Schrodinger's wave equation.

$$\frac{-\hbar^2}{2m}\frac{d^2}{dx^2}\psi(x) + V(x)\psi(x) = E\psi(x) \tag{1}$$

Heisenburg's uncertainty principle states that a particle's definite momentum and position can not simultaneously be known exactly.

$$\sigma_x \sigma_p = \frac{\hbar}{2} \tag{2}$$

Solving for the wave equation with Heisenburg's limitation allows for the probability of the electron existing on the other side of the barrier. When this occurs, some of the energy reflects and some tunnels through, creating a measurable potential (illustrated in Figure 1).

$$I\ \alpha\ \psi^2 = \psi_o^2 e^{-2kz} \tag{3}$$

where

$$k = \frac{\sqrt{2m\Phi}}{\hbar} \tag{4}$$

and work function Φ is

$$\Phi = U - E \tag{5}$$

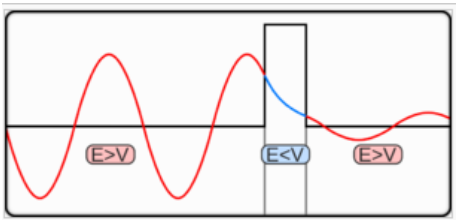

FIG. 1. Quantum Tunneling

This measured potential from electron tunneling changes as the tip scans across the material. At each point, the material has a local density of electron states varying in height, affecting the potential that provides the information for the final image. By keeping a constant tunneling current, a piezoelectric crystal detects changes in potential and adjusts the tip's height accordingly. The profile of electron density states on a surface

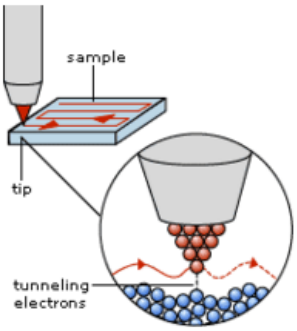

FIG. 2. STM tip-surface interaction

of a material (a property known as corrugation) depends on the local electron position and energy.

Scanning tunneling spectroscopy measures the local density of states with respect to electron energy. A range of voltages is applied to the tip. This changes the electron's energy. Keeping the tip fixed, the change in current from the electrons as the applied voltage changes is measured. The I-V curve reflects the electron configuration, local quality of the sample, and the curve's derivative ($\frac{dI}{dV}$) is the local density of states at that particular point.

III. MATERIALS AND METHODS

We used a Nanosurf Naio STM and the Nanosurf software to control the STM and view the captured images. In order to achieve sub-angstrom precision, the tunneling

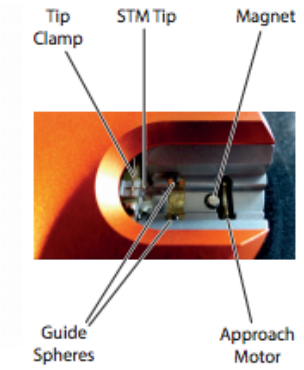

FIG. 3. Nanosurf Naio STM

current will flow through one electron at the end of the tip. Therefore the tip must be very sharp and sterile, requiring careful preparation. We then placed the tip into the tip clamp and placed the sample holder on the magnet. We initialized the image capture by inputting the feature dimensions into the software. The STM tip requires a very precise approach to the sample. The closer the tip is to the sample, the higher probability tunneling will occur; however, making contact with the sample damages the tip and a new one must be made.

For our images, we used STM to sample graphite. Graphite is a common calibration material for STM imaging due to its uniform hexagonal lattice, corrugation, and planar surface, all properties which make it relatively easy to resolve. We removed excess flakes from the surface and cleaned the sample before placing it into the apparatus.

Once our tip finished approaching the sample, we began imaging on a 400 to 500 nm scale. We selected a smooth/uniform section of the image and zoomed in on that section. We repeated this process until we began to see a pattern emerge at smaller areas. We routinely adjusted parameters to maximize image resolution at each scale. For example, a higher tunneling current caused the tip to be more sensitive which, though better for resolution, introduced noise. We adjusted to P, I, and D gain parameters (adjust gain according to error, the integral and derivative of error respectively). After obtaining a high resolution image, we altered the parameters of scan range, time per scan line, bias voltage, and tunneling current to observe their effect on image quality. Finally, to validate our STM system, we switched to spectroscopy mode to capture a smooth, steep I-V curve.

IV. RESULTS

We began by imaging a larger section (approx. 500x500 nm) of the sample with a bias voltage of 0.05V, so that the surface appeared smooth. Atoms in the graphite sample are not stationary; thermal energy permits these atoms to oscillate. When imaging at the angstrom scale, the tip must scan the sample faster than the atoms move, else the image will appear blurry. After routinely zooming in on a smooth section of the surface of the graphite, we were able to resolve the hexagonal lattice on a 2.3x2.3 nm image.

Figure 4 displays the final, high res image of graphite. Figure 4 has highlighted dots to illustrate the atoms' configuration. The atoms in the hexagon make different bonds (yellow lines) within the lattice. Some atoms appear white on the image (pink dot) while others appear grey (green dot), depending on the potential. The black space marks the center of the hexagon where no atoms exist and the tip feels more potential. The greyscale bar reflects how high/low the tip feels the current.

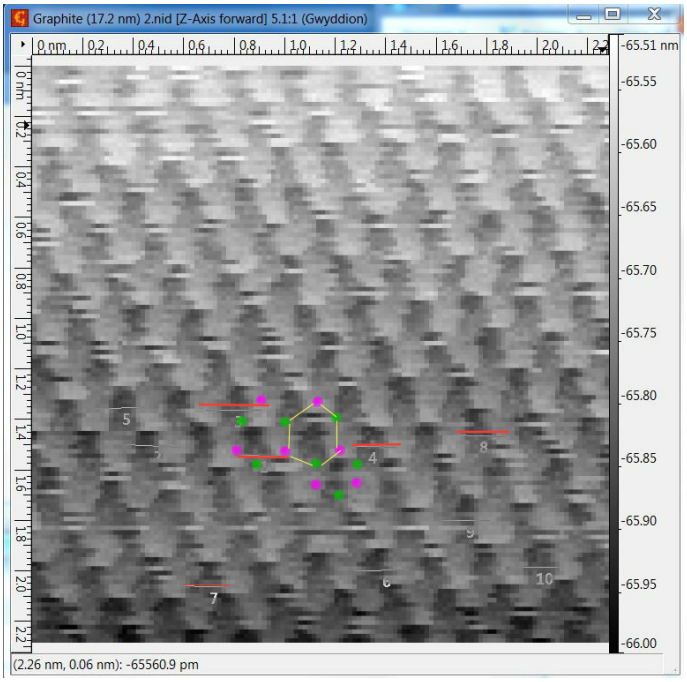

FIG. 4. Graphite

Graphite bonds in layers (Figure 5). From the imaged surface, we see one face of the unit cell. The 3 dimen-

FIG. 5. Graphite Bond Structure

sional shape of graphite is a rhombic prism with dimensions a, b, c. The red lines in Figure 4 are measurements of the lattice constant. From our STM image we can determine the 'a' lattice constant by taking measurements using Gwyddion (open source software for measurements on AFM and STM images). The measured lattice constant was calculated to be 2.10 ± 0.15 angstrom, close to the accepted value of 2.46 angstrom.

We then retook the image multiple times at the same zoom level while alternating the following parameters one at a time: scan range, time per scan line, bias voltage, and tunneling current (see Figure 6).

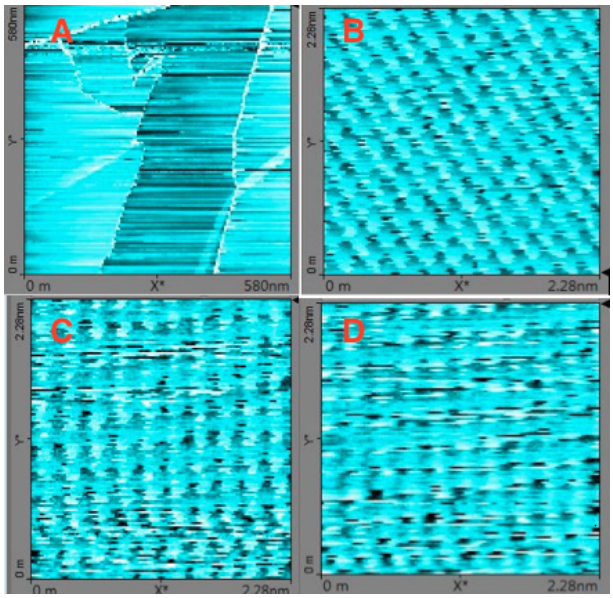

FIG. 6. Altering Parameters. A) Scan Range B) Time per line C) Bias Voltage D) Tunneling Current

We imaged the graphite sample at multiple scales. Zoomed out to 580x580nm reveals the gross structure of the sample. A) shows cracks in the sample. We continually zoomed in until we were able to resolve the hexagonal structure of graphite (Figure 4). When zooming in beyond the resolution in Figure 4, we could not resolve a distinguishable hexagonal pattern.

We then altered the time the tip spent imaging per scan line. We increased the time from 130ms to 250ms. 250ms is too slow to resolve the moving atoms. As a result, the image in B) appears blurry.

Our final high resolution image was captured at 50mV. We took a series of image with voltages ranging from 5 to 100mV. At 5 and 100mV the images appeared streaky and blurry as there was too little or too much voltage respectfully. C) shows the sample imaged at 100 mV. Too little voltage results in not enough potential for the electrons to tunnel and create the measurable potential whereas too much causes a noisy system.The images taken at 25 and 75mV could resolve the hexagonal pattern; however, were not as clear as the 50mV image.

We then increased the tunneling current from 2 to 5 nAmps, shown in D). Increasing the tunneling current results in a more sensitive tip. Similar to the bias voltage, increasing this parameter creates a noisy, streaky image. See Appendix for more referred images with altered parameters.

Finally, we switched the STM into spectroscopy mode to capture the I-V Curve. Figure 7 shows the sampled

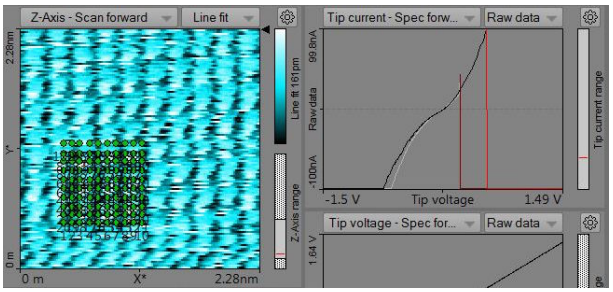

FIG. 7. IV Curve

area and the captured I-V curve from -1.5 to 1.5V. Our steep curve validates that we have found a good section of the sample to image and that our tip has not degraded. Once this I-V curve becomes noisier or broader, the experiment may require a different sampled area or a new tip.

V. DISCUSSION

We collected STM images of a graphite sample at angstrom level resolution. STM reads information about the sample by scanning a very sharp tip above the sample without making physical contact with the surface. The electrons from the single atom at the tip can spontaneously tunnel across the barrier between the tip and sample. This produces a potential that is precisely measured by a piezoelectric crystal. The change in potential is recorded as a function of the tip's position and mapped into a final image.

The STM is very sensitive due to the physical nature of the imaging apparatus. The tip has to be carefully prepared by shearing a piece of tip material (conductive, typically a metal such as platinum-iridium). Both tip and sample must be free of dust particles and oils (from

human hands). The tip is fragile and easily breaks upon making contact with another surface. Therefore, the tip should be moved with tweezers. Any vibrations from the table the STM rests on detract from image quality. To ensure the apparatus is working as intended, perform imaging in a quiet setting.

To explore the effects of altering the STM configuration, we gradually changed the scan range, time per scan line, bias voltage, and tunneling current parameters. A large scan range shows bigger scale (100 nm order) features. The images appear to be uniform noise until you reach a below 10 nm range. The time per scan line needs to be fast enough that the atoms do not significantly deviate while the tip images them. Having a too high time per scan line yields blurry images. Both bias voltage and tunneling current should not be too high else the tip becomes too sensitive and the image becomes noisy. On the other end, if bias voltage and tunneling current are too low, there may not be enough potential difference for quantum tunneling of electrons and the image loses information. All of these alterations proved how sensitive the STM is to each parameter input. To ensure the highest resolution possible, scan the sample with a variety of configurations. These configurations will be different for each material being imaged as each unique material sampled will had different surface properties.

In this experiment, we imaged graphite because of its recognizable honeycomb lattice structure. In practice, scanning graphite is a calibration step for high resolution STM imaging. The STM apparatus is considered reliable upon reproducing the known structure of graphite. STM has been very useful in the study of atomic bonds in both chemistry and solid state physics. For analysis our image, we used Gwyddion to measure the angstrom scale features of the surface of graphite. We repeatedly measured the 'a' lattice constant and confirmed the value against the accepted length. To further ensure that our STM was well calibrated and we had a good subsection of the material to sample, we switched to scanning tunneling spectroscopy mode and recorded the I-V curve of the tip. The smooth, steep curve shows that the tip's quality was intact and that there was good tip-sample interaction and the time of imaging.

VI. CONCLUSION

We imaged a sample of graphite using STM to identify graphite's cell configuration and lattice constant. STM measures quantum tunneling potential interaction between the sharp tip and surface electrons. The final image maps the potential against the tips position to produce a topographical map of the surface with sub-angstrom level resolution.

STM can take images up to sub angstrom resolution with careful, sterile preparation of tip and sample. Once the tip successfully approached the surface, we configured the apparatus to optimal parameters using the proprietary software. Routinely zooming in on uniform regions of the image eventually gave us our final high res image of the surface of graphite (Figure 4). From this image we could identify the hexagonal unit cell configuration characteristic to graphite. Using Gwyddion, we measured the 'a' lattice constant to be 2.10 ± 0.15 angstrom which is on order of the accepted value of 2.46 angstrom.

We studied the effects of the apparatus parameters on the image by routinely altering the values of scan range, time per scan line, bias voltage, and tunneling current. Deviating each from the optimal parameters resulted in blurry or streaky images, proving that the STM apparatus has to be carefully configured for high resolution performance.

Finally, we switched the STM into spectroscopy mode to see the I-V curve of a selected section of our final, high-res image. We observed a steep curve, reflecting the local density of states of the surface electrons. This clean curve validated that we had a good imaging surface on the sample and a sharp tip.

VII. BIBLIOGRAPHY

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VIII. APPENDIX: FIGURES

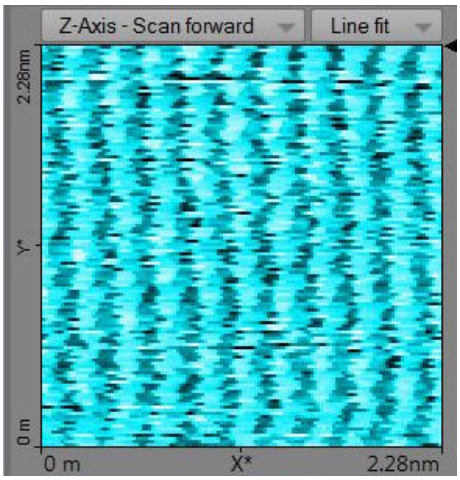

FIG. 8. Tunneling Current 2 nAmps

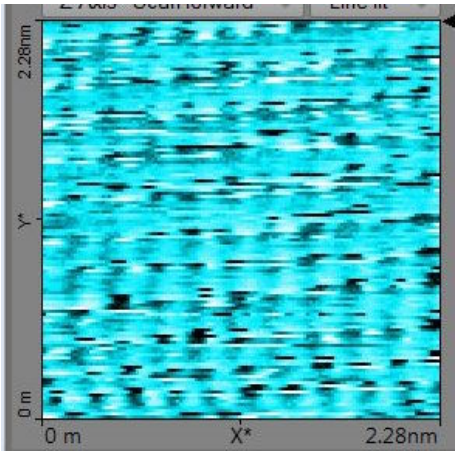

FIG. 9. Bias Voltage 5 mV