A simple investigation of several samples using a scanning tunneling microscope

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A variety of samples, namely HOPG (highly ordered pyrolytic graphite), gold (111), a nanogrid and tantalum disulfide (TaS\textsubscript{2}) were scanned using a scanning tunneling microscope (STM) and analysed. The lattice constant of graphite was found to be \((0.141 \pm 0.038)\) nm which is in agreement with the literature value\textsuperscript{1}. Scans of the other samples provided good images which were analysed numerically in a variety of ways. A nanogrid (a manufactured sample with a known structure on the nanoscale) was scanned and used to investigate the calibration of the STM with some interesting results; the piezo actuators that move the tip suffer from hysteresis and thus only delivered accurate values at small scan ranges.

I. INTRODUCTION

Since its development in 1981\textsuperscript{2} the scanning tunneling microscope (STM) and its related “older brother” the atomic force microscope (AFM) have established themselves as key tools for research on the nanoscale\textsuperscript{3}. As such, our primary aim was to familiarize ourselves with the STM and gain some theoretical and practical knowledge of this exciting method of measurement.

STMs work by exploiting quantum tunneling – an effect where particles can move across a potential barrier whose energy is larger than the particles total energy. This effect is exponentially proportional to the width of the barrier, or in the case of the STM, the distance between the scanning tip and the sample. It can thus be used in a “constant-current mode” where the tip’s distance to the sample is constantly adjusted by a PID feedback loop to maintain a constant set current as the tip moves across the sample. The distance between the sample and the tip can then be recorded as a function of the tip’s lateral position and used to produce a topographic scan of the surface mapping the density of states of the sample.

Many sophisticated STMs scan samples in an ultra-high vacuum and at temperatures near zero Kelvin using tips sharpened by chemical etching. The two STMs employed here (the easyScan and the easyScan 2 manufactured by Nanosurf) where used to scan a variety of samples in air at ambient temperature using mechanically sheared tips.

II. METHOD

Tips for the STM were cut from Pt-Ir wire following the guidelines set out in the manual supplied with the STM\textsuperscript{4}. These were installed and the sample was manually brought fairly close to the tip (within a few mm) before starting the sample holder’s automatic approach to the tip. In practice only about a quarter of all attempts to take measurements were successful either due to crashing the tip into the sample during the approach or because the tip was not always sufficiently sharp to obtain atomic resolution.

All parameters of the scan were controlled using the EZSCAN software which also displayed any data collected from the apparatus.

III. HOPG

The following scan of HOPG at atomic resolution was obtained using the easyScan 2 microscope.

![Figure 1: A scan of HOPG using the following parameters: Tip Voltage = 50mV, Current set point = 1nA, P & I Gain = 1000 and 2000 respectively (as set in EZSCAN software).](image-url)
By using the EZSCAN software to measure the distance between bright hills in fig. 1 and using some simple trigonometry, the lattice constant was determined as \((0.141 \pm 0.038)\)nm which is in agreement with the literature value\(^1\). The error on this value was derived from the resolution of the scan.

IV. GOLD

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Fig. 2 shows a scan of gold (111). The area highlighted in red is approximately \(85\)nm\(^2\) and contains in the order of 1000 gold atoms. The height variation between it and the surrounding area is approximately \(0.3\)nm which is roughly equal to the diameter of a gold atom\(^5\). This suggests that this area is a small plateau of about 1000 gold atoms and of a thickness of one gold atom.

V. TANTALUM DISULFIDE

TaS\(_2\) exhibits an electronic phase transition from a normal into a condensed state which is called the Charge Density Wave (CDW) state. The transition is caused by an electron-phonon coupling.

Fig. 3 shows the CDW lattice of TaS\(_2\). It was measured to be \((1.035 \pm 0.067)\)nm which is of approximately the desired order of magnitude\(^6\).

VI. NANOGRID

The nanogrid is a sample which consists of a two-dimensional grid with a square pitch of 160nm and can be used to calibrate STMs. The polycarbonate grid is manufactured by injection molding using a special compact disk tool\(^7\) and is then sputtered with a thin layer of gold to ensure sufficient conductivity for STM measurements.

Fig. 4 shows a scan of the nanogrid. Several measurements of the distance between cells on the nanogrid repeatedly delivered values much lower than the 160nm stated by the manufacturer. The measured values were consistently closer to 110nm. After the manufacturer was
contacted and confirmed the pitch of the grid as \((160\pm5)\)nm the internal calibration of our STM was investigated.

The tip is moved by three (one each for the X-, Y-, and Z-direction) piezo actuators. When the voltage across the piezo transducer is changed, \(\Delta V\), it moves the tip by an amount, \(\Delta L\), proportional to \(\Delta V\). This relationship between displacement, \(\Delta L\), and voltage change, \(\Delta V\), is however, also depended on the rate at which this voltage change is applied.

\[
\frac{dL}{dV} = f\left(\frac{dV}{dt}\right)
\]

This effect is known as hysteresis. Unfortunately, as the size of the scan range was increased \(\frac{dV}{dt}\) increased too, as larger voltage changes were applied to the piezo per unit time in order to cover the larger distances. This caused the piezo actuators to overshoot and travel a distance larger than the controller “thought” they were actually travelling. This explains why the nanogrid was measured as smaller than it actually is.

In addition the amount of hysteresis increases with increasing voltages applied to the actuator. This should mean that, when scanning at full range, one would expect features further away from the scan origin to become smaller and smaller as the piezo’s will have a larger voltage applied to them and overshoot by a larger amount.

Indeed, the diameter of the “hills” in the nanogrid seems to have decreased as you move further away from the scan origin. Fig 5 shows a graph of the diameter of a given pit vs. its distance from the origin. As expected there is a negative correlation between the two.

![Graph of the diameter of a given pit vs. it's distance from the scan origin.](image)

VII. CONCLUSION

STMs are an extremely useful tool and, potentially, have a lot to offer. Their practical usage can, however, be frustrating and tiresome. Much of the time spent on this investigation involved cutting tips for the STM – the majority of which crashed sooner or later – or waiting for the automatic approach to bring the sample into tunneling range.

There is, however, much reward lurking behind the frustration. When it does works it can deliver excellent results and all negativity is quickly replaced by a buzz of success.

While a good value for the lattice constant of graphite was obtained, possibly the biggest achievement of this investigation was the discovery of hysteresis in the piezo actuators that control the tip. Knowing this effect is present allows for it to be accounted for in future investigations. A good way to minimize this effect would be to increase the time per scan line when scanning over large ranges and thus allow voltage changes to be applied to the piezos slower.

VIII. ACKNOWLEDGMENTS

The authors are grateful for the continuous assistance provided by the 3rd year laboratory staff at UCL and would like to extend special thanks to Dr. Cyrus Hirjibehedin from the London Centre for Nanotechnology. His questioning allowed us to reflect on our work and guided us into the right direction. We would also like to thank Stephan Stuecklin and Dr Peter van Schendel from Nanosurf who provided us with useful data for our samples and helped us get to the bottom of our unsuccessful attempts at measuring the nanogrid.

IX. REFERENCES