

Scanning Tunnelling Microscopy

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December 21, 2012

Abstract

The aim of this experiment was to familiarize the operators with the use of the STM, and scan a variety of common samples. Atomic resolution was achieved on Highly Oriented Pyrolytic Graphite (HOPG), and the gold sample showed characteristic features. The measurements for the HOPG were found to be within the expected. However the nano-calibration grid and the TaS_2 samples appeared to be contaminated. This prevented a more in depth analysis, such as accounting for thermal effects and the possibility of Hysteresis in the microscope tip.

1 Introduction

A Scanning Tunnelling Microscope (STM) is an instrument that is used to image surfaces at atomic scales. STMs work by measuring the quantum tunnelling current between an atomically fine tip and a conductive sample. The first STM was developed in 1981 by Binnig, Quate and Gerber, who were awarded the Nobel Prize for its development.[2] A good STM can be expected to have a 0.1\AA (0.01 nm) depth resolution and a 1\AA (0.1 nm) lateral resolution. This makes it well within the bounds of possibility to scan individual atoms with the correct sample. This accuracy allows for the study of localized electronic properties down to the atomic scales.

2 Theory

The quantum tunnelling current comes from the electron's wave nature. When two electrically conductive surfaces are sufficiently close together their electron's wavefunctions overlap, and electrons are able to tunnel between the two materials, resulting in a small current flowing between them. The transmission probability, which is equivalent to the tunnelling current (I_t) decays exponentially with the barrier width (d) as shown in Equation 1, where α is a constant.

$$I_t \propto e^{-\alpha d} \quad (1)$$

This relationship shows that the tunnelling current is extremely sensitive to the separation distance. A 1Å difference in separation is capable of causing a current difference of almost an order of magnitude. This explains why the vertical resolution is so much more accurate than the lateral resolution. The current measured by the STM is best described by the Bardeen Current, as shown in 2. In this equation, $f(E_T)[1 - f(E_s + eV)]$ is the Fermi functions of the tip and the sample, $|M_{T,S}|^2$ is the Tunnel Matrix, which is calculated from the wavefunction of the electrons in the tip and sample. $\delta(E_T - E_S)$ restricts the tunnelling to states where the tip and sample have the same energies)

$$I = \frac{2\pi e}{\hbar} \sum_{T,S} f(E_T)[1 - f(E_s + eV)]|M_{T,S}|^2\delta(E_T - E_S) \quad (2)$$

Equation 2 can be solved to give Equation 3. In this equation, $\rho(r_t, E_F)$ is the local density of states at E_F , which corresponds to the charge density from states at the fermi level E_F

$$I \propto \sum_S |\psi_S(r_t)|^2\delta(E_S - E_F) \equiv \rho(r_t, E_F) \quad (3)$$

This means that the STM does not directly probe the position of the sample's nuclei. Rather, it probes the electron density of the sample. This means that the image is effected by the nature of the surface, as well as the polarity and magnitude of the tunnelling current.[4]

3 Experimental Set-up

A typical STM consists of a number of essential components, including a sharp probe tip, a piezoelectric motor for fine control, a coarse motor for positioning the sample and probe tip, and control electronics that uses the coarse motor and piezoelectric controls to move the tip based on the tunnelling current. A schematic diagram of the STM is shown in Figure 1. A tunnelling current occurs when a potential difference is applied across the tip and the sample, and is used to both prevent the tip from crashing and to measure the electrical properties of the sample.

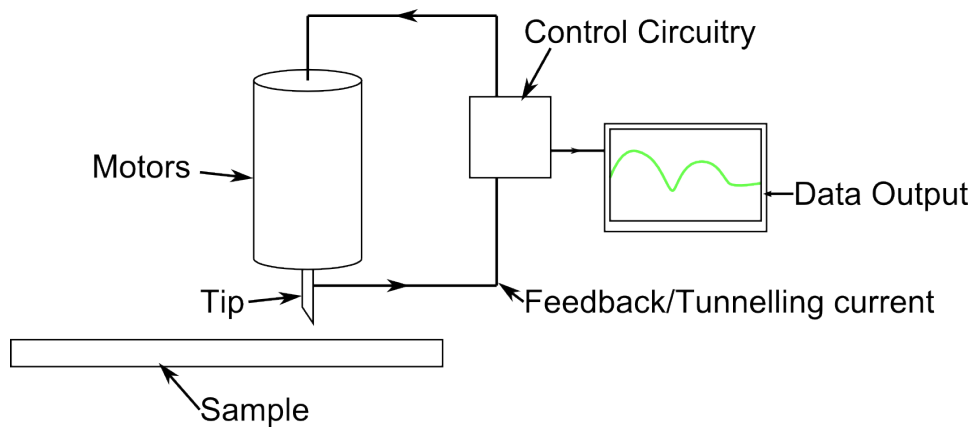


Figure 1: Diagram of an STM

3.1 Tip cutting

New tips were cut from Pr/Ir wire. First a short section of wire was cut and held in a pair of flat nose pliers. A wire cutter was then used to hold on to the end of the exposed wires, and pulled such that the wire "tears" off, leaving an atomically fine point, as shown in Figure 2 [6]. The new tip was then transferred into the STM using a pair of tweezers, where it is held in place by a gold electrode.

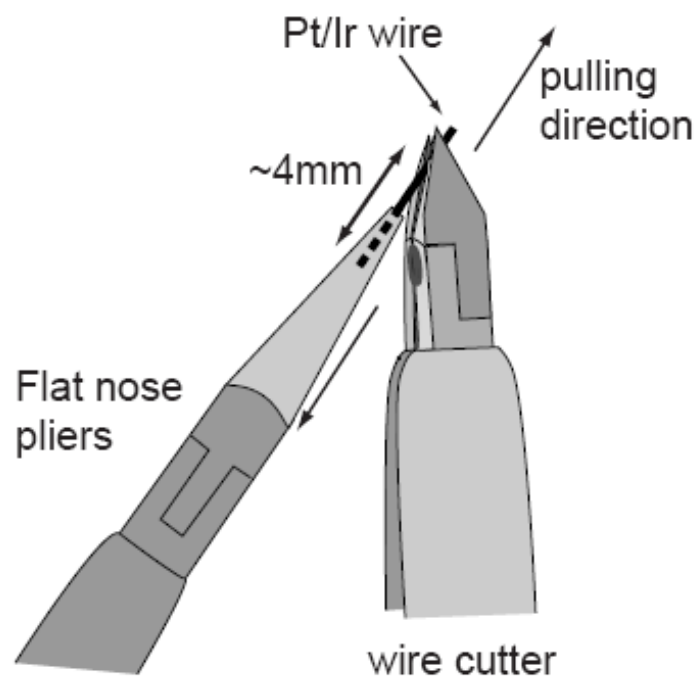


Figure 2: Cutting STM new tips

4 Experimental Results

4.1 Calibration Grid

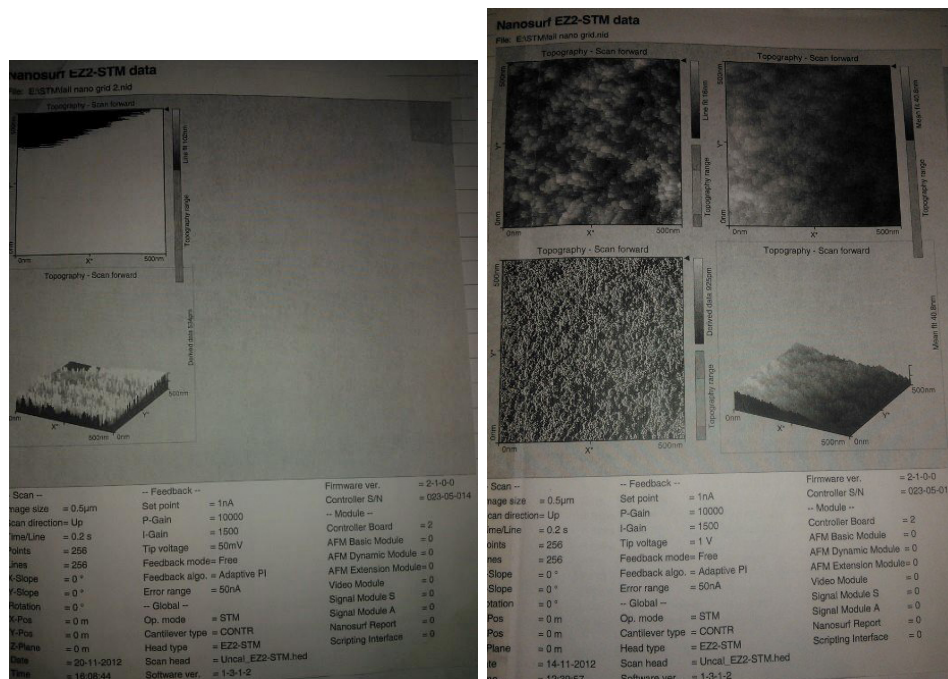


Figure 3: Scans of the Calibration Grid

First, we attempted to scan the calibration grid, with the intention of calibrating our measurements. However, as Figure 3 shows, we were unable to scan the grid. The images received from the grid are indicative of the grid being contaminated.

4.2 HOPG

HOPG, or highly oriented pyrolytic graphite, is a form of graphite with typically atomically fine structure. The lack of macroscopic structure makes it ideal for imaging the constituent carbon atoms. As the separation between the atoms is an established value, these scans can be used to confirm that the tip is atomically fine, as well as the characteristics of the tip, such as the difference between measured distances and theoretical values.[5]

HOPG has a very well defined structure. As shown in Figure 4, the structure consists of two overlaid hexagonal lattices.

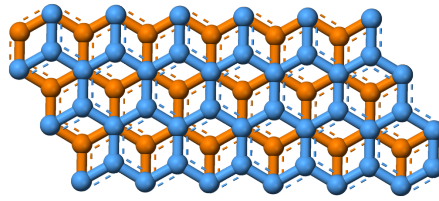


Figure 4: Diagram of Carbon atom orientation in HOPG

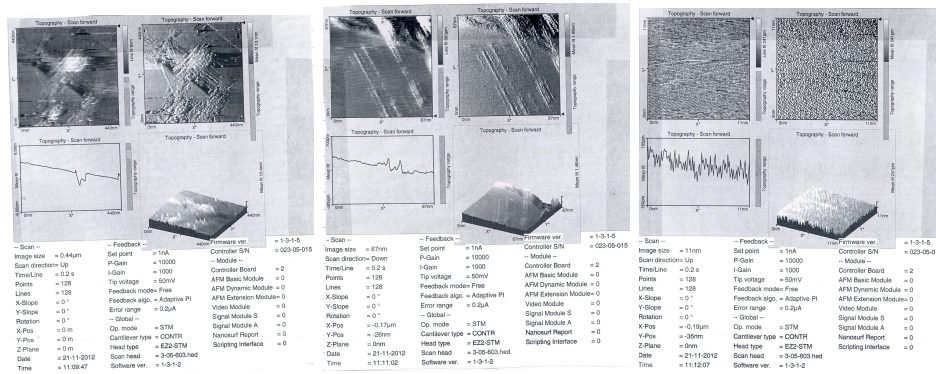
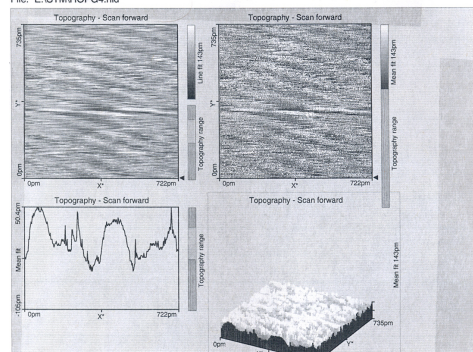


Figure 5: Initial scans of HOPG

Figure 5 shows the initial approach of the tip. In the first two images show some large features, likely due to dirt or imperfections on the surface. We chose to focus on the smoother areas with the aim of capturing an image of the sample's atomic structure. Figure 6 shows repetitive bumps characteristic of a crystalline structure, however the images are still unclear, indicating the presence of scanning artefacts

Nanosurf EZ2-STM data

File: E:\STM\HOPG4.nid



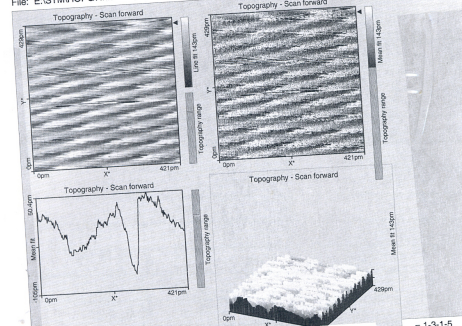
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-- Scan --
Image size = 0.72nm
Scan direction= Down
Time/Line = 0.1 s
Points = 256
Lines = 256
X-Slope = 0 °
Y-Slope = 0 °
Rotation = 0 °
X-Pos = -0.19µm
Y-Pos = -35nm
Z-Plane = 0nm
Date = 21-11-2012
Time = 11:13:57

-- Feedback --
Set point = 1nA
P-Gain = 10000
I-Gain = 1000
Tip voltage = 50mV
Feedback mode= Free
Feedback algo. = Adaptive PI
Error range = 0.2µA
-- Global --
Op. mode = STM
Cantilever type = CONTR
Head type = EZ2-STM
Scan head = 3-05-603.hed
Software ver. = 1-3-1-2

Controller S/N = 023-05-015
-- Module --
Controller Board = 2
AFM Basic Module = 0
AFM Dynamic Module = 0
AFM Extension Module = 0
Video Module = 0
Signal Module S = 0
Signal Module A = 0
Nanosurf Report = 0
Scripting Interface = 0
Firmware ver. = 1-3-1-5
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Nanosurf EZ2-STM data

File: E:\STM\HOPG7.nid



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-- Scan --
Image size = 0.42nm
Scan direction= Down
Time/Line = 0.2 s
Points = 256
Lines = 256
X-Slope = 0 °
Y-Slope = 0 °
Rotation = 0 °
X-Pos = -0.19µm
Y-Pos = -35nm
Z-Plane = 0nm
Date = 21-11-2012
Time = 11:21:41

-- Feedback --
Set point = 1nA
P-Gain = 10000
I-Gain = 1000
Tip voltage = 50mV
Feedback mode= Free
Feedback algo. = Adaptive PI
Error range = 0.2µA
-- Global --
Op. mode = STM
Cantilever type = CONTR
Head type = EZ2-STM
Scan head = 3-05-603.hed
Software ver. = 1-3-1-2

Controller S/N = 023-05-015
-- Module --
Controller Board = 2
AFM Basic Module = 0
AFM Dynamic Module = 0
AFM Extension Module = 0
Video Module = 0
Signal Module S = 0
Signal Module A = 0
Nanosurf Report = 0
Scripting Interface = 0
Firmware ver. = 1-3-1-5
```

Figure 6: Zoomed in scans of HOPG

Next, we rotated the angle at which the sample was scanned. The purpose of this was to see if the distortion in the previous scans was due to the error in the feedback circuits, i.e. the feedback system was not sensitive enough to track all the features on the sample. Figure 7 shows the next set of scans. In these images, the individual atoms are very clearly. Next, we doubled the scan points per line to improve the clarity of the image.

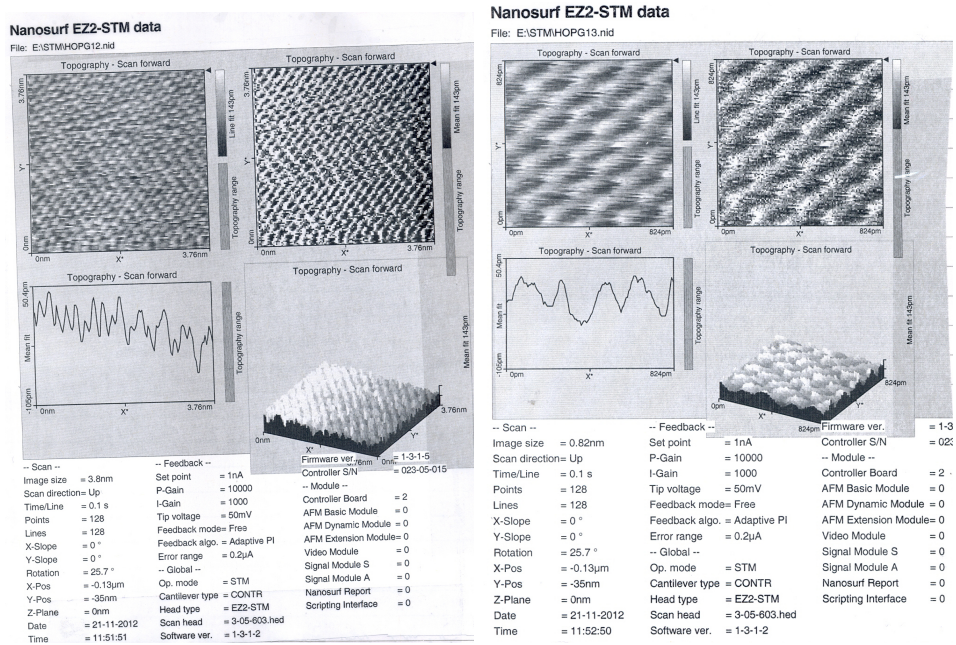
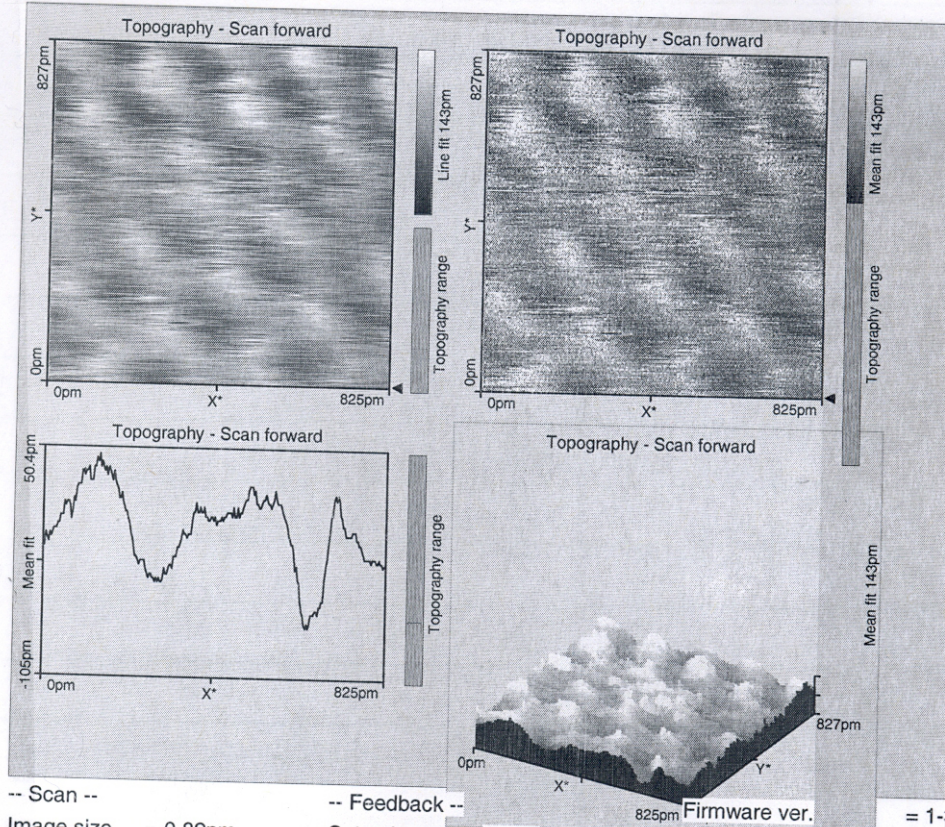


Figure 7: HOPG sample with visible atoms

Figure 8 shows a configuration of atoms as expected (As shown in Figure 4). The centre to centre distance for the atoms that make up the HOPG has a literature value of 0.1415 nm. This is of the same order of magnitude as what can be observed from Figure 8.

Nanosurf EZ2-STM data

File: E:\STMHOPG14.nid



-- Scan --		-- Feedback --		Firmware ver. = 1-	
Image size	= 0.82nm	Set point	= 1nA	Controller S/N	= 02
Scan direction	= Down	P-Gain	= 10000	-- Module --	
Time/Line	= 0.1 s	I-Gain	= 1000	Controller Board	= 2
Points	= 256	Tip voltage	= 50mV	AFM Basic Module	= 0
Lines	= 256	Feedback mode	= Free	AFM Dynamic Module	= 0
X-Slope	= 0 °	Feedback algo.	= Adaptive PI	AFM Extension Module	= 0
Y-Slope	= 0 °	Error range	= 0.2µA	Video Module	= 0
Rotation	= 25.7 °	-- Global --		Signal Module S	= 0
X-Pos	= -0.13µm	Op. mode	= STM	Signal Module A	= 0
Y-Pos	= -35nm	Cantilever type	= CONTR	Nanosurf Report	= 0
Z-Plane	= 0nm	Head type	= EZ2-STM	Scripting Interface	= 0
Date	= 21-11-2012	Scan head	= 3-05-603.hed		
Time	= 11:54:07	Software ver.	= 1-3-1-2		

Figure 8: Higher resolution HOPG scan

4.3 Gold

Next, we scanned a gold sample. Gold is a material that is of significant interest to the study of nanotechnology. It is one of the least susceptible metals to oxidation, and is an extremely good conductor of electricity, as well as a variety of other properties that make it potentially useful for the development of nano-scale devices such as it's ease of doping. The first two properties have the added effect of making it a good candidate for probing with a STM. Due to the nature of the metal, we can expect the electrons to be very de-localized. This means that acquiring an atomic scale image of the gold is difficult, since the STM measures the local density of state of the electrons. We can expect the atoms to be defined by a small perturbation in the scan, where the electrons are more strongly attracted to the gold nuclei.[3]

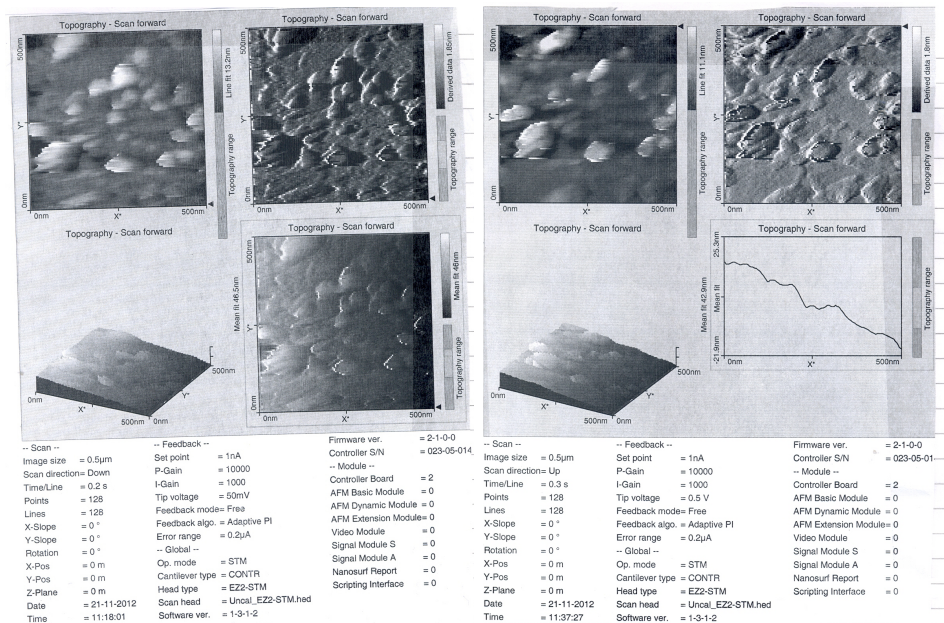


Figure 9: 500nm scans of gold

Figure 9 shows scans of gold at a scale of 500nm. At this level it is possible to observe the non-regular, apparently random structures characteristic of gold. Figure 10 shows the same area, zoomed in more. The features from Figure 9 are consistent with these results.

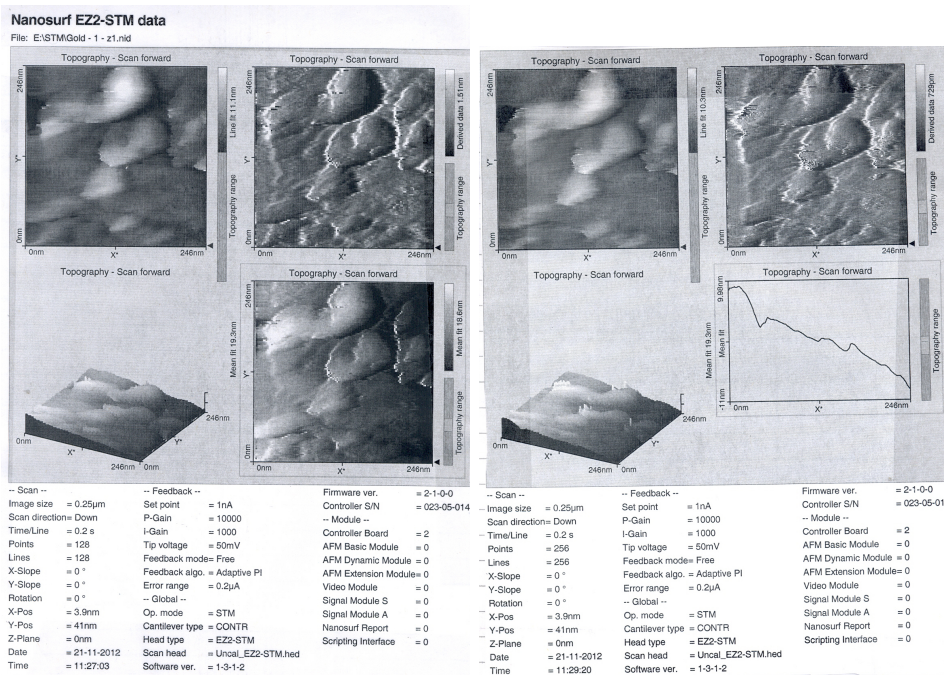


Figure 10: 246nm scans of gold

Next, we increased the scanning voltage, as shown in Figure 11. Increasing the voltage had the effect of flattening the data set. This should make it easier to distinguish the perturbations caused by the gold nuclei. Unfortunately, we were unable to scan the gold at a high enough resolution to attempt to distinguish the individual atoms.

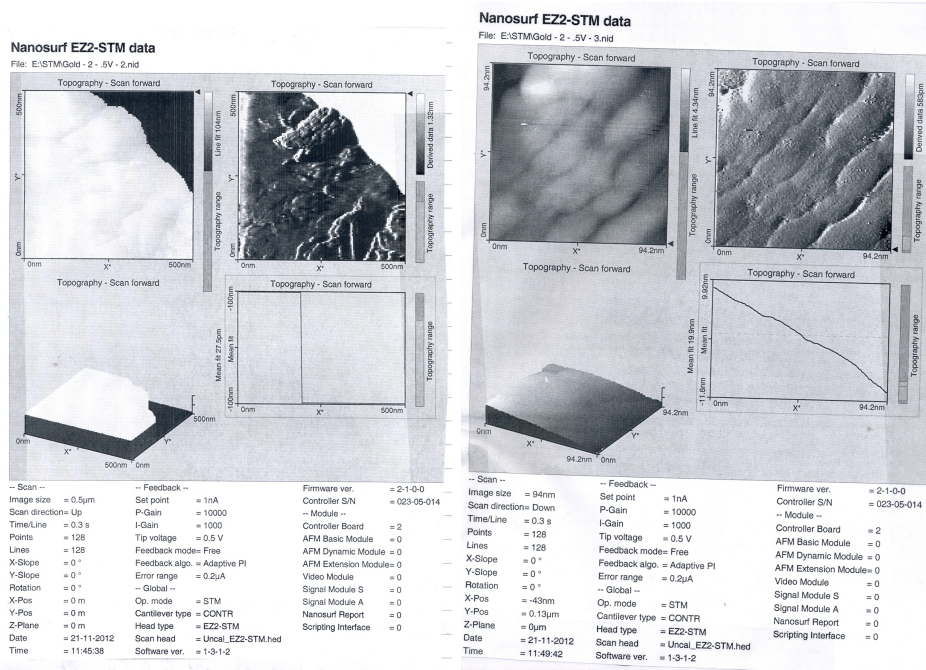


Figure 11: 0.5V scans of gold

4.4 TaS_2

TaS_2 , or Tantalum sulphide, is a compound consisting of two sulphur atoms and a tantalum atom, and typically forms ordered layers. However, we were unable to acquire scans of this material that showed us this layered nature, or its atomic structure. This appears to be due to a defective sample, as multiple tips and scanning modes were used in an effort to find results. Figure 4.4 shows our results. The uniform nature of the samples, despite a variety of different resolutions implies that the sample was damaged in such a way that the structure was degraded or damaged.

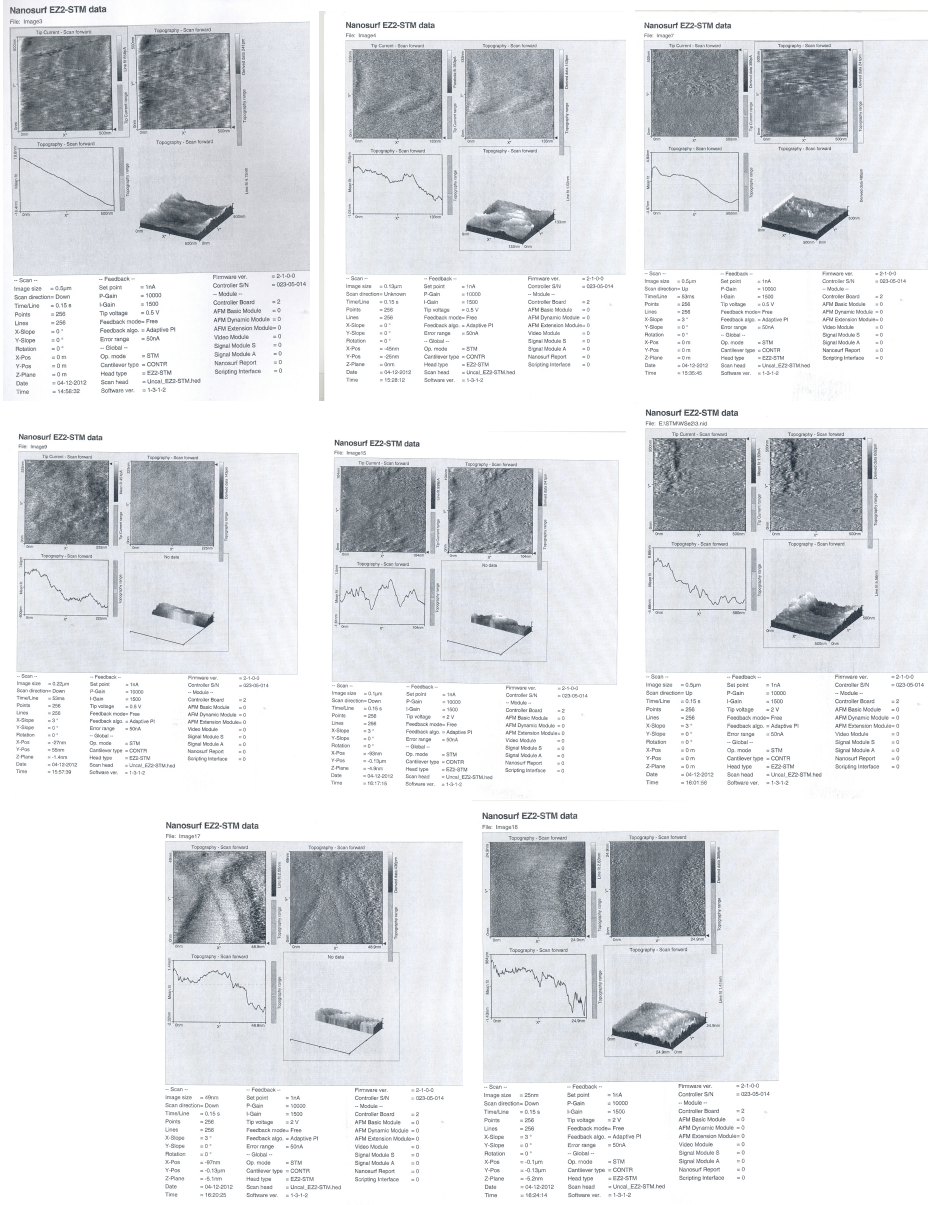


Figure 12: TaS_2 scans

5 Conclusion

In conclusion, the experiment was able to successfully image HOPG and gold samples, however the calibration grid needed to perform more in depth analysis of the results appeared to be contaminated. Similarly, the TaS_2 sample was not able to be scanned.

References

- [1] *Nanosurf AG. Operating Instructions EasyScan E-STM. Version 2.1.*
- [2] Ching-Tzu Chen. *Scanning tunneling spectroscopy studies of high-temperature cuprate superconductors.* PhD thesis, California Institute of Technology., 2006.
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