College of Engineering

XCoursework Submission Sheet

Coursework Title: AFM & STM lab report	Student number: 551192
Coursework number 1st assignment	Name: Edmund Merrow-Smith.
Module code: EGNM01	Email: edmund@sucs.org
Module title: Probing Nanoscale	Degree course: MSc Electronics and Electrical Engineering
Submission deadline: 10th Nov 2014	
Lecturer: Dr R. Cobley.	

By submitting this coursework, I certify that this is all my own work.

Submission date ……………………………………..

Student signature if this is a non-electronic submission…………………………………………………

SPLD Students

Please tick this box if you are officially recognised by the University as an SPLD student. ☐

LEAVE THIS SPACE BLANK FOR LECTURER FEEDBACK

EGNM01 AFM & STM Lab Report

Edmund Merrow-Smith, 551192

Abstract

This is a lab report containing the results and analysis of an SPM lab exercise for an SPM teaching course at Swansea University. An Ivy leaf, a fragment of Highly-Oriented Pyrolytic Graphite, a fragment of Tungsten Oxide, a film DVD and a music CD are observed under a desktop AFM and desktop STM.

1. Introduction

Students taking EGNM01 Probing at the Nanoscale at Swansea University are taught about various scanning probing microscopy technologies. Although lectures can teach a lot about these technologies, students can still be left under or over estimating the potential of these without first hand experience of trying to get data from them.

To remedy this, a desktop Scanning Tunnelling Microscope (STM) and a desktop Atomic Force Microscope (AFM) were set up, some choice samples were prepared, and students were asked attempt to use the equipment provided to to find out as much as they could about the samples and report their findings and difficulties in this report.

This report presents one student's (the author) findings and their analysis.

2. Experimental methods

The equipment provided was 2 Scanning Tunnelling Microscopes (SPM).

- An Easyscan E-STM desktop STM (Nanosurf, Switzerland) with a tip made from 90% platinum 10% iridium wire, prepared by pulling and cutting with pliers that were cleaned with ethanol beforehand.
- A NaioAFM desktop AFM (Nanosurf, Switzerland)

The software used to handle the data from and control the AFM was Nanosurf Naio v3.3.0.3, and Eastscan E-line v2.1.2 for the STM.

The following samples were provided and prepared as noted:

- An Ivy leaf (likely *Hedera Helix*), prepared by cutting out a small section of leaf flesh and attaching it to the sample holder with 2 strips of cellophane tape to the edges of the sample, leaving the centre of the sample exposed.

The Ivy leaf is a non-conducting biological sample, so it was decided to only use the AFM to inspect the leaf. It was also decided that attempting to observe the stoma on the underside of the leaf would be the best use of the laboratory time to get the most informative measurement. Care was taken to ensure that this section contained as few features such as trichomes (small hairs) and vascular bundles (veins) as possible.

- A fragment of Highly-Oriented Pyrolytic Graphite (HOPG), prepared by applying a strip of cellophane tape and peeling it away to expose a clean surface before attaching to the sample holder.
- A fragment of Tungsten Oxide (WO_3), with no preparation other than cutting to fit and placing in the sample holder.

- A film DVD and a music CD, both prepared by breaking the disk in 2, cutting out a sample from the centre of one of the edges of one of the halves, peeling away the protective transparent coating on the underside of the disk, and attaching the sample to the sample holder underside facing up, same as the ivy leaf was attached.

The make up of a CD and a DVD is a plastic base coated on the underside with a metal (the hole in which form the data), which is then protected by a layer of clear lacquer to protect that metal coating. This lacquer coating was removed in preparation. The now exposed metal conducts electricity, would be suitable for STM. However the pits in surface that the data is encoded with have had the metal stripped away, and the plastic base beneath it does not conduct electricity, so attempting to use STM in constant current mode on these samples would likely result in the tip crashing into the surface upon travelling over a pit, and using constant height mode without having observed the surface beforehand is unwise, so the AFM was again used. These samples are much harder, tougher, and smoother than the ivy leaf, so damage to the tip or sample was less likely, so the PID was changed to apply a much higher target force, in the hopes of getting more detail.

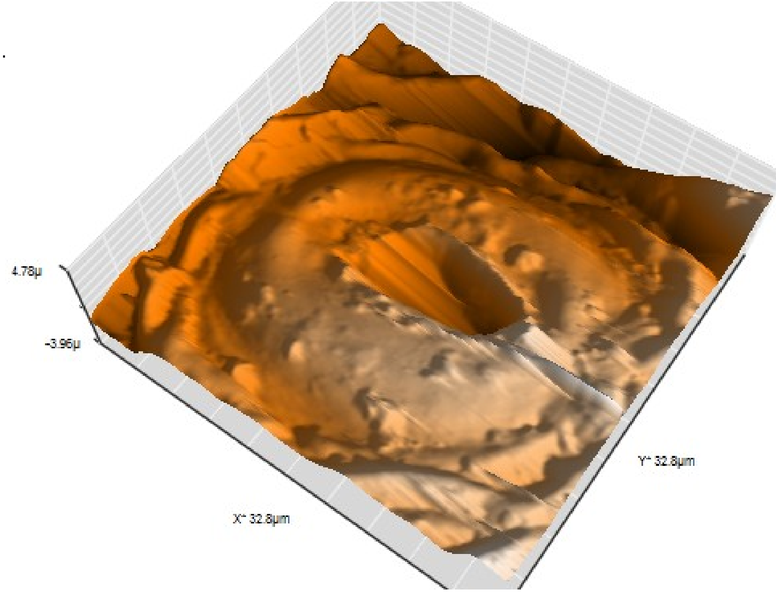


Figure 3.1.1 – raw Z data of forward scan

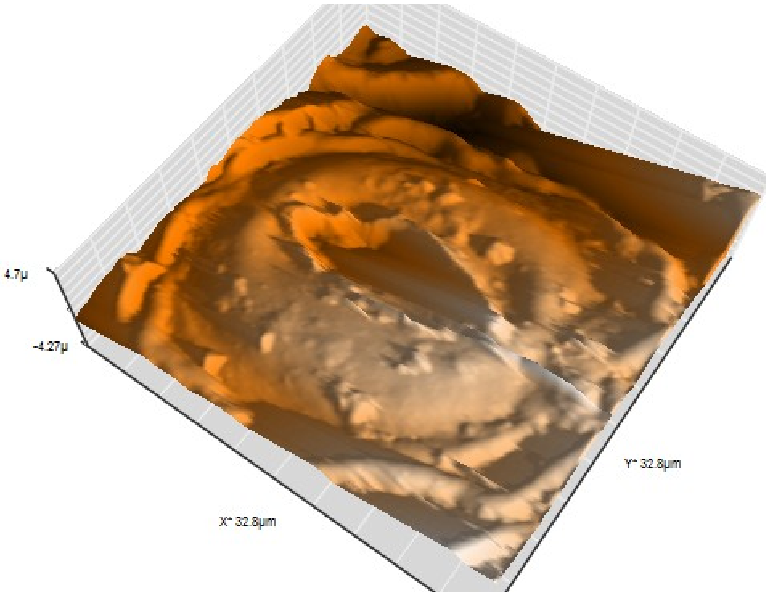


Figure 3.1.2 – backward scan of 3.1.1

3. Results

3.1 Ivy Leaf with AFM

For all measurements, the tip height was controlled by a PID controller and given a target force to apply to the sample as it travelled across the surface. Care was taken with all measurements to use a small target force and a slow speed to prevent the tip from damaging the cell walls of of the sample.

The most successful scan taken was Figures 1.1 (forward scan) and 1.2 (backward scan), which are 3d plots of the raw travel data of the tip. This clearly shows an aperture in the centre of 2 curved guard cells, matching the description of a stoma.

It would appear that the stoma found was open. It was expected that the aperture would be closed, because it was reasoned that the leaf would attempt to conserve moisture in the event of the base of the leaf being cut and the transportation system that carries nutrients from the roots would need to be prevented from running dry. However, in light of the image, it is reasoned that instead, the leaf would be attempting to continue gas exchange to increase photosynthesis in the leaf, in an attempt to grow roots and re-plant itself.

Table 3.1 – settings used to capture figures 3.1.1 and 3.1.2

Image size	50µm
Scan direction	Up
Time/Line	1.1 s
Points	256
Lines	256
Const.Height-Mode	Disabled
Setpoint	12nN
P-Gain	10900
I-Gain	1000
D-Gain	0
P-Gain2	0
I-Gain2	1000
D-Gain2	0

The concentric wrinkles surrounding the 2 guard cells would appear to be wrinkles in the cell walls of surrounding cells, whose surfaces had to compress to allow room for the guard cells to open.

There is a very prominent, smooth slope in aperture that reverses gradient in backward scan. This is most likely not a feature on sample but rather an artefact of the PID controller not dropping the tip height fast enough in response to tip travelling over the sheer drop at the inner edge of the guard cells. The corrugations in these slopes are in line with the direction of the tip travel, and the corrugation height matches the terrain of the cell as the tip lost contact with the guard cell at the inner edges, supporting this hypothesis.

3.2 CD and DVD with AFM

The first scans were taken to ensure the AFM and sample were set up correctly. Figure 3.2.1 shows that the PID settings in table 3.2 were mostly satisfactory, but sample surface was not level with the sample holder, causing a tilt in the entire image. The direction of this tilt was such that the scan starting location was downhill of the target scan area on the sample, and the start point had been set at the top of the scan Z range, causing the tip to climb uphill as it scanned, to the point of maxing out the requested height range and producing a blank image above a certain contour.

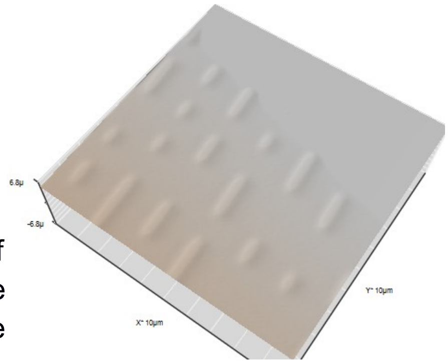


Figure 3.2.1- Preliminary scan of CD

For figures 3.2.2 and 3.2.3, it was decided to take the biggest scan image the AFM could manage in the hopes of observing the curvature of the pits with the disk. However, the sample was taken from a location on the disk too far from the disk centre, and all the rows of pits appear to be straight, parallel lines. What can be observed is the PID retracting the tip too far when exiting pits, causing bright artefacts to appear consistently on the same side edge throughout each image, but on opposite sides in the 2 images. This is a symptom of the PID controller being set a gain that made it too aggressive. Here the 2D colour map of the raw tip deflection data, rather than the raw height data 3D plot, as it is easier to compare the straightness of the lines and it does not include the gradient that spans the whole image from the tilt of the sample.

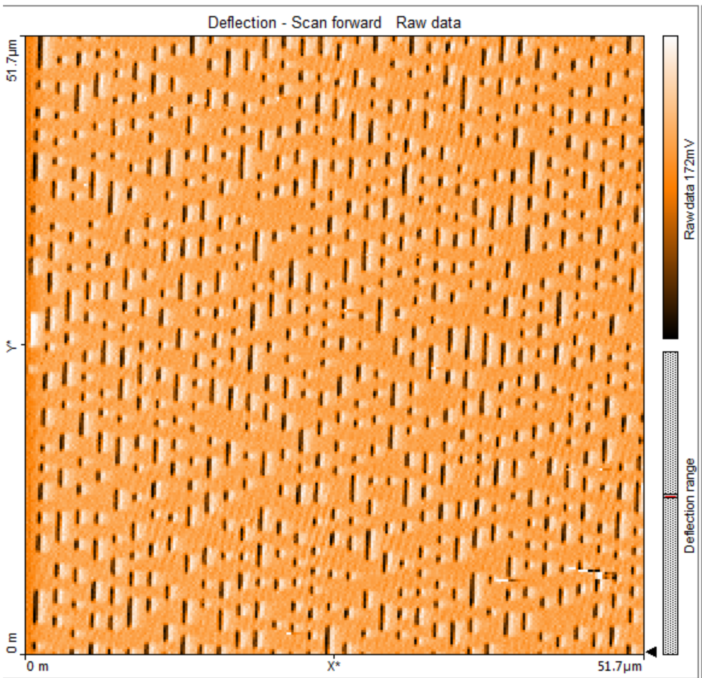


Figure 3.2.2 – CD, forward scan, maximum zoom out (51.7µm)

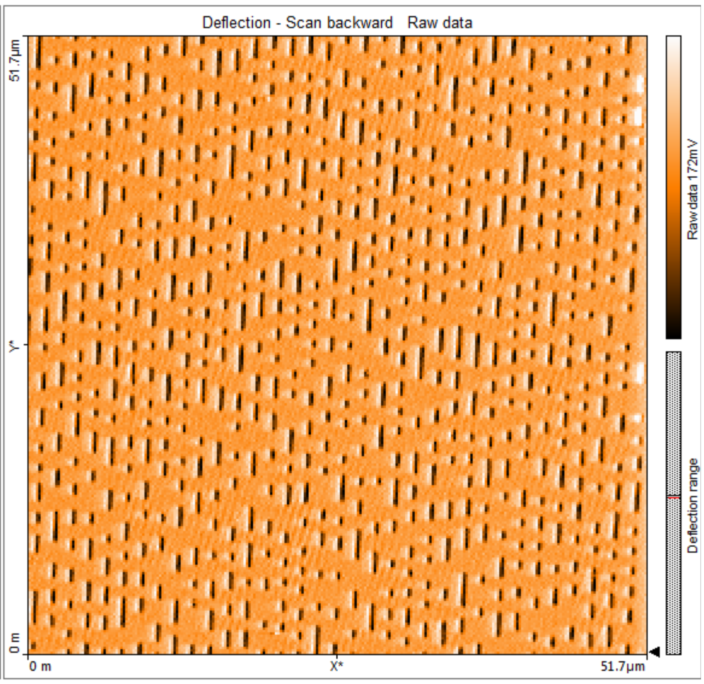


Figure 3.2.3 – backward scan of 3.2.2

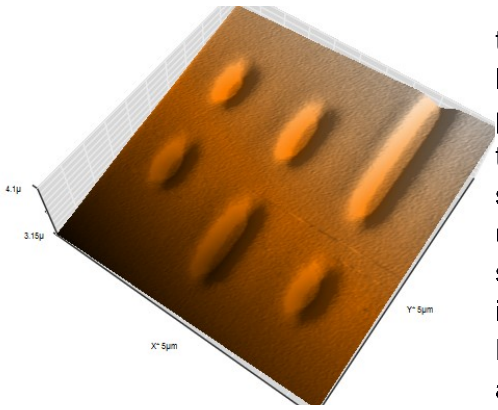


**Figure 3.2.4 –
3D plot of tip height (5µm)**

Then it was decided to attempt to measure the spacing of rows and the thickness and depth of pits. Figure 3.2.4 shows a 3D plot is little help with this. A careful analysis of the deflection data, however, proved much more fruitful. Figure 3.2.5 is a composite image of the tip deflection data of the backwards scan with a strip of the forward scan laid over it in the centre. This reveals that the tip was now undershooting on its way out of the pits and losing contact with the sample on the way into the pits, causing the tip deflection to max out in both directions, blurring the edges of the pits in the image. However, being able to compare the forward and backward scan artefact positions in figure 3.2.5, it becomes possible to make an estimate of the true position of the pits.

Width: **5µm * 51 pixels / 512 pixels = 0.498µm**

Spacing: **5µm * 159 pixels / 512 pixels = 1.553µm**

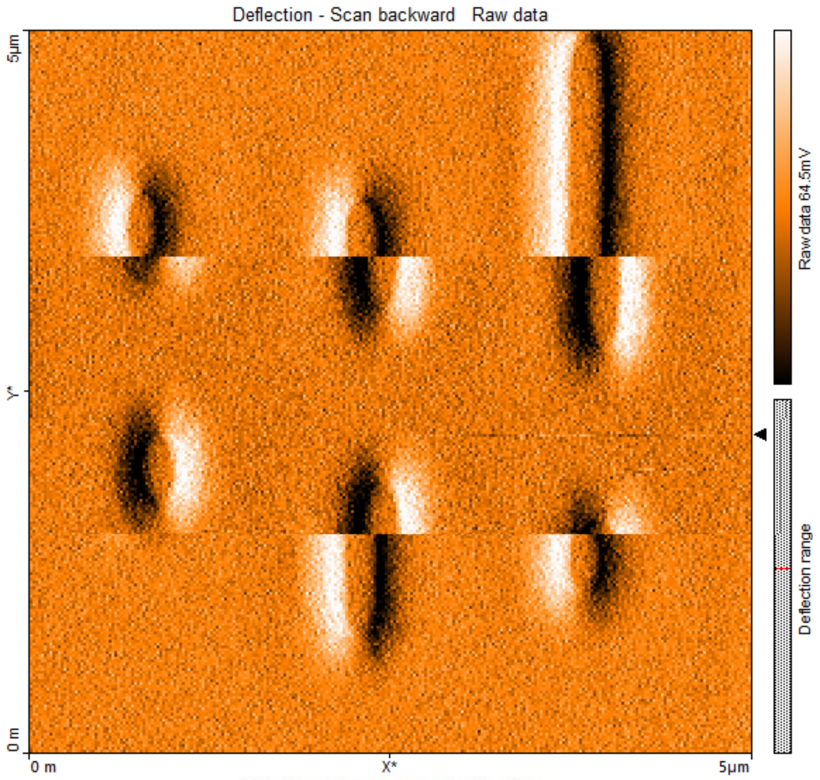
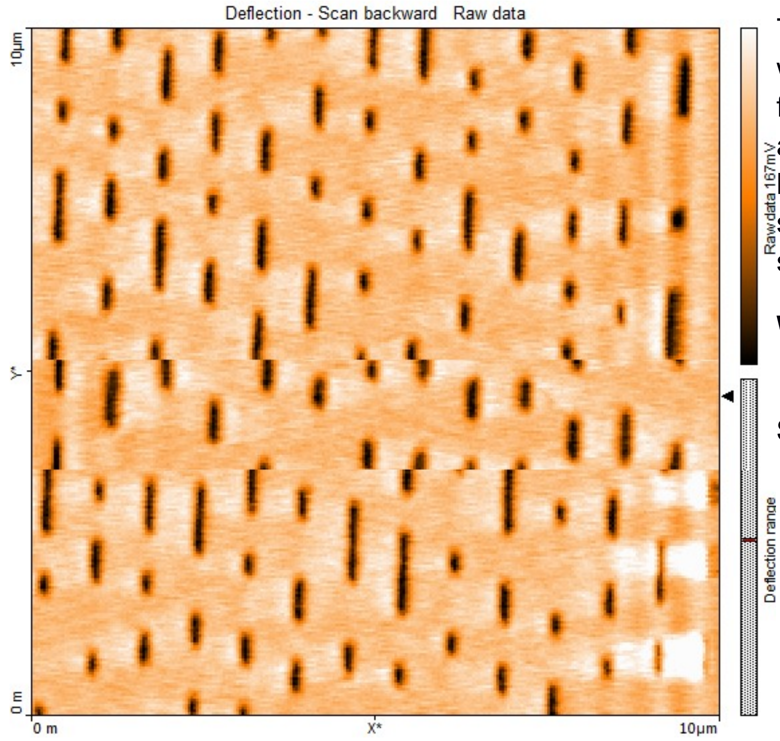


Image size	5µm
Scan direction	Up
Time/Line	699ms
Points	256
Lines	256
Const. Height-Mode	Disabled
Setpoint	50nN
P-Gain	10000
I-Gain	1000
D-Gain	0
P-Gain2	0
I-Gain2	1000
D-Gain2	0

Table 3.2 – settings of figure 3.2.5

Figure 3.2.5 – composite image of forward and backward scan deflection data of CD



The case was not the same for the DVD, where the previous analysis was repeated for figure 3.2.6, showing that the sample appeared to drift between the forward and backward scans. However, either scan is now satisfactory for measuring the width and spacing.

Width: **10µm * 10 pixels / 512 pixels = 0.195µm**

Spacing: **10µm * 38 pixels / 512 pixels = 0.743µm**

Figure 3.2.6 – composite image of forward and backward scan deflection data of DVD