Atomic Force Microscopy

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Section 1

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I. ABSTRACT

In this experiment we measured the topology of some samples using an Atomic Force Microscope (AFM) and its atomically-thin canteliver probe, namely a 10μm calibration grid, a CD master, and a gold-coated diffraction grating. Using the AFM in static force mode, and dynamic force mode we were able to identify and measure the width and spacing of the structures on the calibration grid to be roughly 5x5μm and spaced in 10μm intervals, and the size of some of the CD master ridges, two were measured to be 13.430 and 8.438 μm. No structures in the gold-coated diffraction grating could be identified. In all, the AFM provides very detailed measurements of topology for samples used in this experiment.

II. INTRODUCTION

The purpose of this experiment is to test the features of the Atomic Force Microscope (AFM) in its different modes of operation, static force mode and dynamic force mode by using the AFM to measure the topology of different samples. The samples we will be investigating will be a 10μm calibration grid, a CD master, and a gold-coated diffraction grating. These techniques can be extended to analyse the topology of many different types in surfaces that can be extended to many different fields of manufacturing, and scientific research.

The need for a higher resolution microscope was needed, as the resolution of optical techniques was rather limited. The ATM has a scale resolution of nearly 1000x better than standard optical techniques. The first scanning probe microscope was invented in the early 1980's at the IBM research facility in Switzerland. This first type of microscope was a scanning tunnelling microscope (STM) which relied on the electrical conductivity of the material and was limited to that subset of materials. The AFM is a variation of the STM and can be used on non electrically conductive materials.The AFM can operate at scales from micrometres (μm) to the mere nanometres (nm) and has been used to measure a wide array of samples like: silicon wafers, data storage media, integrated circuits, DNA, bacterial flagellae, muscle proteins, and many other diverse synthetic or biological materials. The AFM has been a very valuable tool in the last decades for imagine and measuring samples at these small scales.

We will be using the AFM to measure a 10μm calibration grid and a CD master in static force mode, or also known as contact mode using the SICONA tip, which has a longer cantilever and is good for use in static force mode. Using the software we scanned portions of the calibration grid and confirmed the measurements of the grids on the sample. We then, also using a SICONA tip in static force mode, scanned for ridges on the CD master template. We measured some ridges that were visible in the software scan windows. Then switching to an ALCA tip and using dynamic force mode we measured a sample of gold-coated diffraction grating to observe the gold particles on the sample.

III. THEORETICAL BACKGROUND

A. The Atomic Force Microscope

The atomic force microscope (AFM) is an assembly that consists of: a nearly atomically sharp probe tip, a laser directed on the tip of the probe from an angle, conversely a detector to measure the reflected laser from the tip of the probe, and a translating assembly which can move the probe up, down, left, and right. There is also a piezoelectric crystal attached in the bracket holding the probe.

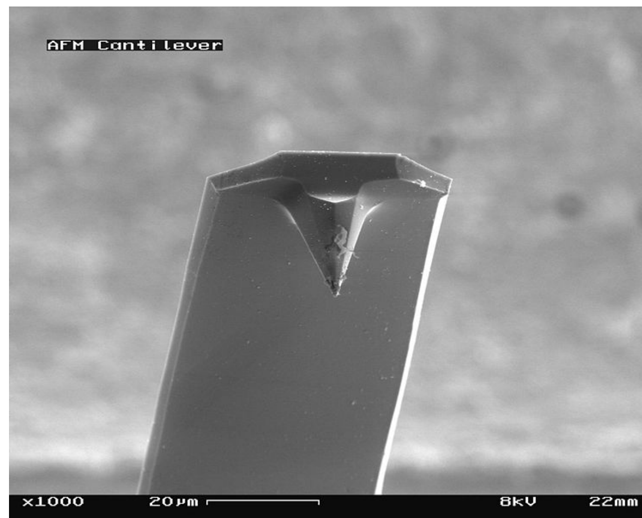

FIG. 1: The tip of the probe, showing the cantilever with the tip on the end, the tip can be seen to be on the order of a fewμm.[1]

The probe consists of a base that is placed into the AFM assembly and held down by a cantilever holder spring. Attached to the base is a very thin cantilever arm, at the end of which, when operating has a nearly atomically sharp probe tip pointing down towards the specimen, as seen in Fig.1. The probe is precisely shaped using a process called silicon micro-machining, combining photographic patterning with chemical etching to shape silicon along precise crystallographic planes.

FIG. 2: A graphical representation of the tripod assembly part of the AFM, which holds the laser, the photodiode, the piezoelectric crystal, and the probe. Two of the circular springs can be seen with the 3 magnetic coils on each arm of the tripod, which are used to translate the probe assembly up, down, left, and right in fine detail using the feedback control loop.

There is a coarse translating assembly that moves the assembly up and down using a stepper-motor and fine gears. This is used initially to move the head down to the sample being scanned. Similarly the AFM module has 3 coarse adjustment screws that can be used to move the assembly up and down initially and level the apparatus. The bracket that holds the laser, diode, and piezoelectric crystal is a type of horizontal tripod suspended on the head with flat springs, and has 3 electromagnets mounted behind the arms, as seen in Fig.2. If the top electromagnet is activated

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[1] By SecretDisc (Own work) [GFDL (http://www.gnu.org/copyleft/fdl.html) or CC-BY-SA-3.0 (http://creativecommons.org/licenses/by-sa/3.0)], via Wikimedia Commons

the tripod swings up and down, similarly the two bottom electromagnets can swing the tripod arcing left and right. When all 3 electromagnets are used in tandem, the combinations can move the head backwards, forwards, left, and right in fine detail.

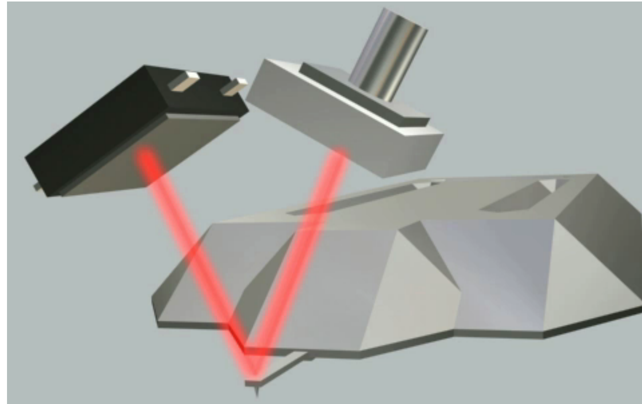

FIG. 3: A graphical representation of the probe with the cantilever and tip, showing how the laser reflects off the cantilever and into the adjacent photodetector screen. Displacements of the cantilever will cause subsequent displacements of the laser on the photodetector screen.

The laser and photodetector are mounted to the tripod as well. The laser reflects off the cantilever and into the photodetector. As the photodetector can detect where the reflected beam is hitting its face, front to back which is important for the operation of the AFM. A representation of this can be seen in Fig.3.

The piezoelectric crystal which is in the bracket is important during operation in dynamic mode, which will be explained shortly. However it should be noted that piezoelectric crystals have a property that when voltage is applied across them, they can shrink and expand in certain directions. Using this property the cantilever can vibrate quickly, close to its resonant frequency.

B. Static Force Mode

In static mode (or contact mode), the tip is slowly moved downwards by the assembly until it contacts the sample and bends slightly. At this point, the laser is reflecting off the tip and only the photodetector at a certain point, as the tip moves across the sample the software will try to keep the point at the same spot, meaning there is a constant force applied to the tip from the sample by the apparatus. The force is adjusted by using the electromagnets in the tripod, the probe is translated up or down by applying a current to the upper electromagnet, this is part of the feedback loop of the laser detection system. This is interpreted by the software as the topology of the sample at that point. The tip is dragged across the sample and the topology of the sample is recorded by the software. A tip that is longer (lower stiffness), allows for the signal read to be boosted since it can bend easier.

In this mode there are several consequences that cam be explored. Since the tip is contacting the sample, it can become dirty, or degraded after a short time of use, consequently the sample could also become damaged especially if the sample is more fragile, consequently this mode is used more for harder surfaces. However, if the surface has a topology with large steep slops, as the tip is dragging along it can hit one of these features and actually break off the tip! This is when dynamic force mode can be used.

C. Dynamic Force Mode

In dynamic force mode, the piezoelectric crystal described above is used, the voltage across it is cycled repeatedly so that it stretches and contracts in the vertical direction. This oscillates the probe tip up and down at nearly its oscillating frequency, as a result the reflected laser also oscillates up and down on the face of the photodetector. Variations in the measured oscillation amplitude and phase in relation to the driver frequency are indicators of the surface-probe interaction.

There are two types of interaction that are possibly when the probe is either operating in contact, or *non*-contact mode. In contact mode, sometimes also called tapping mode, the distance of the probe is calibrated so that, while oscillating up and down, the tip of the cantilever just barely makes contact with the sample at its lowest point. This repulsion from making contact with the sample could be detected and used to measure the topology of the sample,

with the added advantage of being able to get up over sharp and tall features of the sample without damaging the tip.

When operating in Dynamic Force non-contact mode, While oscillating the tip is brought close to the sample. Due to Van der Waals attraction, which occurs when the tip is close to the sample (>10-100 Å), but not too close to cause repulsion from interatomic forces (1-2 Å). When this attraction is barely detected, the feedback loop is again used to keep this average deflection as measured by the photodetector. This mode can often measure the topology of the sample just as accurately as the other, contact, modes, but with the advantage that it doesn't damage the tip *or* the sample. It can be used when the sample is fragile, such as biological samples which are easily deformed or damaged on contact.

IV. EXPERIMENTAL DESIGN AND PROCEDURE

The equipment used in this laboratory was:

- Atomic Force Microscope
- SICONA tip
- ACLA tip
- Easyscan Controller
- Easyscan 2 software
- 10μm AFM calibration grid
- CD sample
- Diffraction grating sample

As both the calibration grid and the CD sample have fairly uniform patterns the SICONA tip and a static force operating mode were used. The SICONA tip was first mounted into the cantilever on the Atomic Force Microscope (AFM) using the method outlined in Appendix 1 of the lab manual. The microscope was then mounted on the stage and adjusted to allow enough space sample holder to be placed underneath it. This was done by adjusting the positioning screws, with the aid of a spirit level to ensure that the device remained level.

After the first sample, a 10 μm calibration grid, was placed under the microscope we initialised the microscope and set the operating mode on the Easyscan software to a SICONA tip and static force mode. At first, the software wasn't detecting the tip, we needed to reposition the tip and restart the software twice until the tip was recognized. From the positioning window it is possible to see through two cameras in the microscope, both of which show a different view of the tip and sample. Using the top view we positioned the sample so that the tip rested above an area that was free from contaminants and scratches. Then, using the side view, we used the 'Advance' function of the software to slowly lower the tip so that it was very close to the sample. The 'Approach' function was then used so that the software would adjust the height of the tip to find 'contact' with the sample. After the approach was completed the scanning of the sample area started.

The settings used for the calibration grid were:

- Area: 50μm
- Time/line: 1s
- Points/line: 256
- Rotation: 0°

It was important to invoke the 'Finish' command in the software so that the AFM would stop scanning after the desired region had been scanned. Had we not ensured that this was checked the AFM would continue scanning, losing the data from the first iteration.

The calibration grid showed a number of evenly spaced raised squares. Using the cross section tool we selected a line that passed through the centres of a number of the squares. We then clicked on 'Cut Out Line' button which displayed a line graph showing the height against the displacement for the cross section that we defined.

We used the zoom function so that we could accurately measure the size of a single square on the grid. We set the function to zoom in on an area that encompassed only one of the squares from the grid and changed the 'Time/line' parameter to 0.5s to speed up the scanning process. Once initiated the AFM started rescanning the area. Once we were presented with the image of a single square we used the measure tool to take measurements of its height and width.

For our second sample, the CD Master fragment, the procedure and set up was essentially the same as before. We did however change the scanning parameters to:

- Area: 15μm
- Time/line: 0.7s
- Points/line: 256
- Rotation: 0°

Due to the CD master being warped/bent slightly, we need to repeat the scan multiple times to find a good, flat position. The tip kept crashing into the sample and as a result we didn't acquire any useful data. After repositioning the CD Master multiple times, we were able to get a scan which resembled the ridges of a CD. From the scan of the CD fragment we measured the size of the pits that could be seen on the surface as well as the track spacing.

Our third sample, the gold coated diffraction grating, had a surface that was far more irregular than the previous two. Because of this a dynamic operating mode is preferable. ACLA tips are used when operating in dynamic mode, so we removed the SICONA tip and replaced it with an ACLA using the method outlined in Appendix 1 of the lab manual. We then changed the operating mode to accept an ACLA tip and use a dynamic force.

We started the scanning process in the same manner as before and set the scan parameters to:

- Area: 15μm
- Time/line: 0.7s
- Points/line: 256
- Rotation: 0°

We then found a reasonably uniform area of the grating and zoomed in on it, in the same manner as before, in order to try to identify the gold specs. We tried several different samples to see if we could identify any form of gold-specs on the surface.

V. ANALYSIS

The output from the scan of the 10μm calibration grid is shown in figure 4.

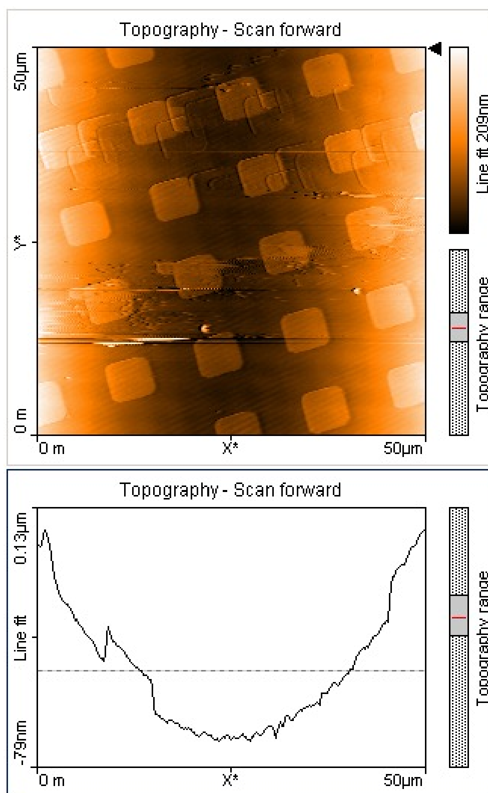

FIG. 4: Colour map and line graph representing 10μ AFM calibration grid

From the scan we can see a number of rows of uniformly spaced raised squares. The graph seems to show that the sample curves up towards the edges. This is likely an anomalous scan as the probability of us taking our image directly positioned over the lowest peak of a valley is low. The cross sectional line graph is given in 5.

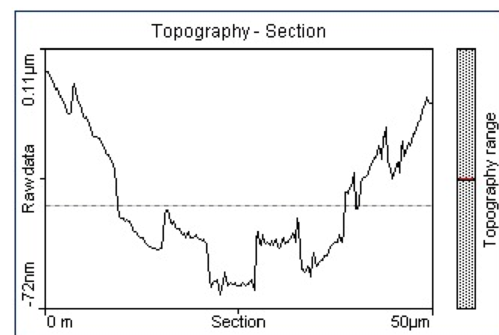

FIG. 5: Cross sectional area line graph showing height against position

This graph shows the curvature that was mentioned and is consistent with inferences taken from the colour map of the same sample.

We zoomed in on one of the squares in order to measure the size of each square. The colour map of the magnified area is given in figure 6. Using the measure tool provided in the software we were able to determine the height width and area of the square, the results are:

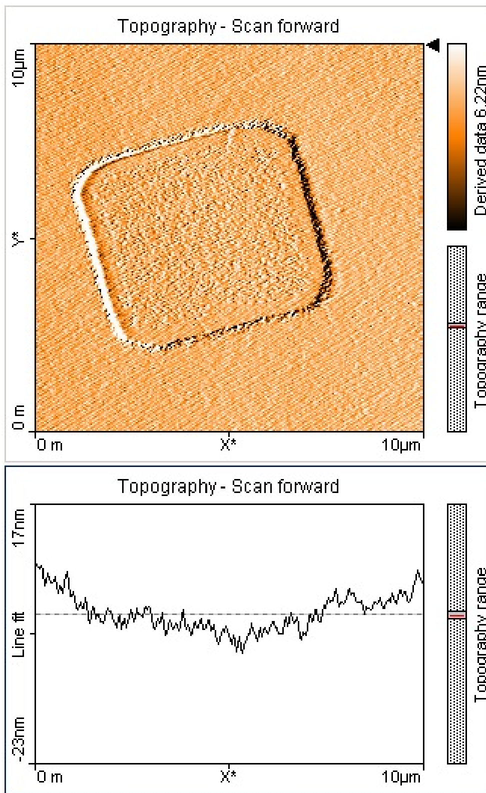

FIG. 6: Colour map and line graph showing a single square from the surface of the calibration grid

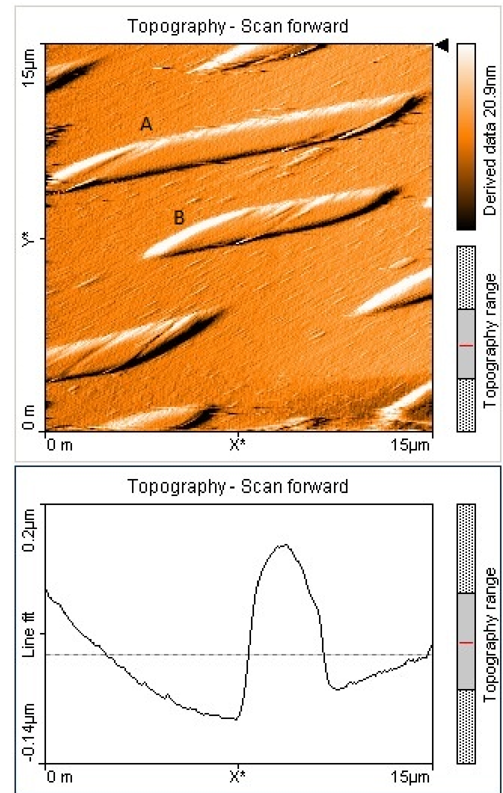

FIG. 7: Colour map and line graph showing the scan of a CD fragment

$$\text{Length} = 5.181 \pm 0.0005\ \mu\text{m}$$
$$\text{Width} = 5.921 \pm 0.0005\ \mu\text{m}$$
$$\text{Area} = 30.68 \pm 0.0007\ \mu m^2$$

The colour map for the CD fragment is shown in figure 7. It can be seen that there are a number of indents that are arranged in tracks on the surface of the CD. Using the measure tool we measured the length of a number of the indents and the spacing of the tracks. The results from these measurements are:

$$\text{Ridge A length: } 13.430 \pm 0.0005\ \mu m^2$$
$$\text{Ridge B length: } 8.438 \pm 0.0005\ \mu m^2$$

Our final scan is shown in figure 8. This is of a gold plated diffraction grating and ideally would show a uniform distribution of parallel lines that make up the grating. Our reading were skewed with every attempt at scanning the sample. Figure 8 is the closest to true form that we were able to extract from the program.

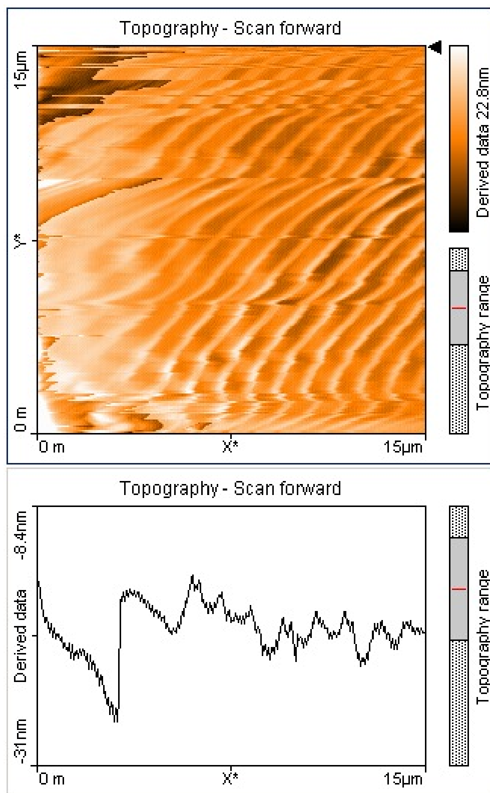

FIG. 8: Output from scan of gold plated diffraction grating.

Our final step of the experiment was to scan a small area of the grating that had a reasonably uniform distribution in order to identify 'pillow shaped' gold pieces on the surface. The output from this scan is given in figure 9. We were not able to identify any gold pieces in this scan and suspect that there was some form of experimental error in the reading of this scan that is to blame. The tip may have been damaged or may have had some contaminants on it, or it is possible a more stable surface is needed for a scan to operate in dynamic force mode.

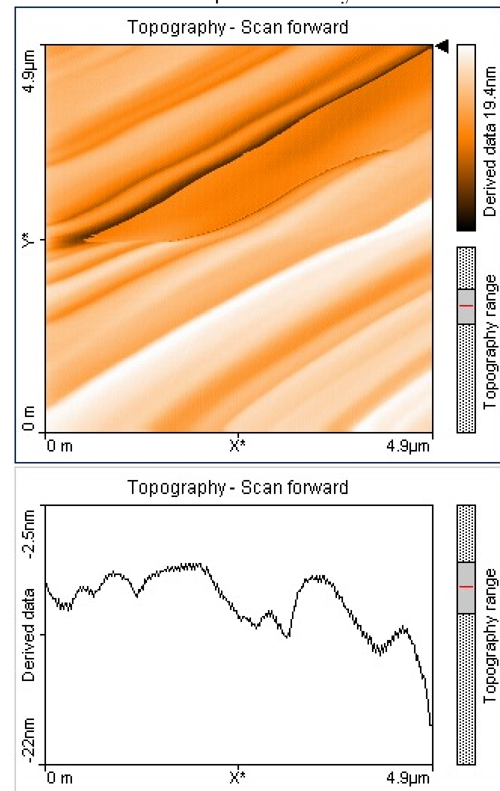

FIG. 9: Output for a small area of the gold plated diffraction grating

VI. CONCLUSION

The Atomic Force Microscope (AFM) was successfully used to scan several samples. We were able to scan and analyse a 10μm calibration grid, a CD master, and a gold-coated diffraction grating.

By using a SICONA tip, made for use in static force mode, we scanned the 10μm calibration grid to verify the working order of the AFM. We found the grid like structures on the grid, they were roughly 5x5μm spaced in roughly 10μm intervals, the scan seems to show that the sample was curved in the middle of the scan window, with the edges of the scan being higher in topology, this was most likely due to some systematic error in the equipment or software that was beyond our control. However, we were able to zoom into a grid and measure it more accurately using tools in the software. We measured a square to have edges of lengths 5.181 $\pm$0.0005μm and 5.921 $\pm$0.0005μm, and thus a cross-sectional area of 30.68 $\pm$0.0007μm.

Still using the SICONA tip operating in static force mode, we scanned the face of a CD master. At first we were not able to obtain a good image, the tip kept crashing into the sample during the scan giving useless and distorted data. This is probably due to the warped/bent shape of the CD master, after repositioning the sample several times and after several scans we were able to find structures that resembled CD master ridges. We had two ridges filling the scan window and were able to measure their lengths to be 13.430 $\pm$0.0005μm and 8.438 $\pm$0.0005μm.

Lastly, using an ALCA tip in the AFM and operating in dynamic force mode, we tried to investigate the surface of a gold-coated diffraction grating. Our scan showed a surface that seems to have uniform ridges on it, we zoomed into a region and found again the ridge-like features, but to a zoomed in degree. We were not able to identify and pillow shaped objects in the scan.

The AFM worked well and produced some very high detailed objects. Scanning time wasn't extraneous, nor was the procedure to scan an object. This method seems like a fast and good way to scan multiple types of objects across different subjects.

VII. REFERENCES

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