Advances in Atomic Force Microscopy Towards Nanoscale Scanning

Juan Saucedo, University of Illinois at Chicago Faculty Mentor: G. Ali Mansoori, Ph.D., Chemical Engineering

The atomic force microscope was used to scan standard semiconductor surfaces, written compact disk surfaces, and regular notebook paper surfaces. The purpose of obtaining the standard sample images was for testing the Explorer scanning probe microscope, which scans in the micron range, and also testing the Discoverer scanning probe microscope, which allows us to scan in nanometer range. All images were obtained in the micrometer scale due to not being able to upgrade the computer scanner files that help operate the nanometer scale scanner. Our final goal was to be able to observe and determine the surface structures of asphaltene micelles with the Explorer and Discoverer scanning probe microscopes.

Introduction

The nano scale is increasingly becoming very important in today's advancing technology. It is strongly focused towards new nanoscale devices, which will benefit in all sorts of applications in the near future. Some of the applications would be in aerospace, chemical, agriculture, electronics, bioengineering, energy industries, medicine, and biological [1]. These nano devices will not only take less space, but will be more beneficial as in the medical field where in the near future there is the possibility of nano devices being implanted inside living cells for reconstruction etc. [1].

Binnig, Quate, and Gerber invented the atomic force microscope in 1986. The purpose of their invention was to be able to acquire sample images in the atomic level of practically conductive and non-conductive samples with a better lateral and vertical resolution [2]. The atomic force microscope is widely used for observing and analyzing sample surfaces in the atomic scale. The atomic force microscope helps determine possible problems that are emerging in the new technology areas and is also used to modify and pattern various types of materials on surfaces [4]. Before the atomic force microscope (AFM), there was the scanning tunneling microscope (STM) along with the stylus profilometer (SP) and others, but neither would accomplish what can be done with the atomic force microscope [2]. For example, with the scanning tunneling microscope it is not possible to scan non-conducting surfaces due to the tunneling current required between the tip and sample during the scan. The stylus profilometer (SP) is capable of scanning conducting and non-conducting surfaces, but it will not give as high resolution as the atomic force microscope. Another problem with the SP is that the sample can be damaged when scanning the surface because of the force that is applied between tip and

sample. Unlike the AFM, the AFM's tip makes a soft physical contact with the sample's surface, which will not damage the surface [2].

The basic parts of an AFM are shown in Figure 1[3]. The laser beam comes from the laser diode and is focused onto the backside of the cantilever. The laser beam reflects from the back of the cantilever and goes to a mirror where the beam reflects and is directed to a four point photodetector. The sensitive photodetector detect the deflection coming form the laser beam when the cantilever moves as the sample is scanned. Any detection by the photodetector results in a signal that is sent to the feedback loop. The feedback loop will register to move the piezo in the z direction taking the laser beam back to the original position on the photodetector. The sample is scanned with a constant force due to the z piezo motion producing a topographical map of the region scanned. In our research we concentrated in the ability of obtaining standard sample images in the micrometer and the nanometer scale range with two different types of scanning probe microscopes (SPM's). Our final goal was to observe and determine the surface structures of asphaltene micelles with the Explorer and Discoverer scanning probe microscopes.

Experimental Details

The apparatus in the lab consists of an Electronic Control Unit (ECU 1), 1010 Stage Box, PR 1000 Decoder, 1000 Modulation Unit, Explorer and Discoverer Scanning Probe microscopes. The Scanning Probe microscopes are operated with computer software version 3.06. The standard samples were all scanned with the Explorer Scanning Prove microscope, which it only allows to scan on the micron scale. The Discoverer microscope was not used due to some missing computer files that allowed the scanners to operate in the nano scale range. The standard samples were situated on the magnetic sample holder and then the Explorer head was mounted onto the base. The samples that were scanned were: a regular notebook paper, a semiconductor grid, and a written compact disk. Samples were scanned in contact mode and did not require special cleaning or a specific location to be scanned because they were used as testers. All samples were scanned in order to determine the capabilities of the Explorer microscope.



Figure 1: Basic components of an Atomic Force Microscope. The laser beam emerges from the laser diode onto the back of the cantilever where it goes to a mirror and reflects into the 4-point photodetector. The photodetector sends a signal to the feedback loop resulting in an image due to the compression and expansion of the piezo scanner.

Experimental Results and Discussion

The following images were the first obtained with the Explorer scanning probe

microscope operating on contact mode. The images were obtained for the purpose of

observing the capabilities of the Explorer microscope. Images obtained were of a regular notebook paper, a semiconductor grid surface, and a written CD surface.



Notebook Paper

Image 1: Notebook paper image with the following parameters used to scan the surface. The set point: 5nA, scan range: $130\mu m$, scan rate: $200\mu m/s$, resolution 200, proportional 1.50, integral 0.50, and derivative 0.01.

There seems to be no pattern from the image obtained. Smaller scan ranges were taken to see if there is a noticeable difference between images.



Image 2: Notebook paper image with the following parameters used to scan the surface. The set point: 5nA, scan range: $5\mu m$, scan rate: $200\mu m/s$, resolution 200, proportional 1.50, integral 0.50, and derivative 0.01.

Image 2 was taken with a different scan range. All other parameters were kept the same as in image 1. There seems to be a pattern, but the scan range that was used is not small enough to seem any significant structure.



Image 3: Notebook paper image with the following parameters used to scan the surface. The set point: 5nA, scan range: $1\mu m$, scan rate: $200\mu m/s$, resolution 200, proportional 1.50, integral 0.50, and derivative 0.01.

Image 3 has a smaller scan range and all other parameters are kept the same for comparison purposes from the other two images. Image appears to have the same pattern as image 2. The difference between image 2 and 3 is that in image 3 the resolution is better and the image is a bit more detailed.

Semiconductor Surfaces



Image 4: Semiconductor image with the following parameters used to scan the sample's surface. The set point: 15nA, scan range: $50\mu m$, scan rate: $200\mu m/s$, resolution 200, proportional 1.00, integral 0.30, and derivative 0.01.

Image 4 is a semiconductor grid. All parameters are changed except for the scan rate, resolution, and the derivative. The set point is increase in order to scan the surface. The set point basically is the force between the tip and the sample as it scans. The unseen image part could be that the cantilever had a tilt when performed manual tip approach.



Image 5: Semiconductor image with the following parameters. The set point: 10nA, scan range: $20\mu m$, scan rate: $200\mu m/s$, resolution: 200, proportional: 1.00, integral: 0.30, and derivative 0.02.

In image 5, the set point, scan range, and the derivative parameters were changed in order to obtain positive feedback when scanning. If the scanning is done with false feedback, the image might not be accurate or simple there would be no image obtained. The scan was conducted on the same sample location as image 4, but at a smaller range.



Image 6: Semiconductor image taken on the same sample location as in image 4 and 5 but at a smaller scan range and higher resolution. Parameters were as follows: set point: 10nA, scan range: $10\mu m$, scan rate: $200\mu m/s$, resolution: 300, proportional: 1.00, integral: 0.30, and derivative 0.02.

Image 6 was scanned at the same location as in image 4 and 5. The only parameters that were changed were the scan range, which it was brought down by $10\mu m$ and the resolution was increased to 300. The image is the same as image 5, but at a closer view.

Written CD Surfaces



Image 7: Written CD image taken with the following set parameters. The set point: 15nA, scan range: $100\mu m$, scan rate: $200\mu m/s$, resolution: 200, proportional: 1.50, integral: 0.50, and derivative 0.01.

Image 7 is a written compact disc scan. The upper section of the image is the CD and the lower section is part of a semiconductor grid. As it was mentioned before, these are standard samples and this sample happens to consist of two parts.



Image 8: Written CD image taken with the same parameters as image 7 except a different scan range was used. Parameters were as follows: set point: 15nA, scan range: 50µm, scan rate: 200µm/s, resolution: 200, proportional: 1.50, integral: 0.50, and derivative 0.01. The image has a better resolution.

Image 8, the scan range has been decrease by 50µm and all other parameters are kept

constant. The CD groves take a better shape towards the lower section of the image.



Image 9: Written CD image with taken with a lower set point and scan range. Parameters were as follows: set point: 10nA, scan range: $20\mu m$, scan rate: $200\mu m/s$, resolution: 200, proportional: 1.50, integral: 0.50, and derivative 0.01. Here you can see what seems to be the actual grove on the CD surface.

In image 9, the set point and the scan range were decrease and the other parameters were kept the same. Notice the difference in the image, only one grove is visible but at a higher magnification.

The following images were the second batch that was taken. These images were taken with a better laser alignment and were all of a semiconductor grid. There is a better image resolution due to the laser alignment.

C 070902f.tfr (200 × 200)

Semiconductor Surfaces

Image10: Semiconductor image taken with the given set parameters. Parameters were as follows: set point: 10nA, scan range: 20µm, scan rate: 200µm/s, resolution: 200, proportional: 1.00, integral: 0.30, and derivative 0.01

The semiconductor grid in image 10 was taken with the same parameters as the previous semiconductor images. The image appears to be a bit clearer, possibly because of the laser alignment.



Image 11: Semiconductor image taken at a smaller scan range than image 7. Parameters were as follows: set point: 10nA, scan range: 10µm, scan rate: 200µm/s, resolution: 200, proportional: 1.00, integral: 0.30, and derivative 0.01.

Image 11 was scanned with the same set parameters as image 10. The only difference is the scan range. The magnification is better and the image is more detailed.



Image 12: Semiconductor image taken at a smaller scan range and scan rate. Parameters were as follows: set point: 10nA, scan range: 5µm, scan rate: 50µm/s, resolution: 300, proportional: 1.00, integral: 0.30, and derivative 0.01.

A different scan range, scan rate and resolution was used the scan image 12. Image detail is the same as image 11; the only difference is that is magnified.



Image 13: Semiconductor image with a different shape than the previous ones. Parameters were as follows: set point: 10nA, scan range: 20µm, scan rate: 100µm/s, resolution: 200, proportional: 1.00, integral: 0.30, and derivative 0.00.

Image 13 was taken with a different scan range; scan rate, resolution, and derivative. The

image is very detailed probably due to the derivative being set at zero.



Image 14: Semiconductor image with parameters as follows: set point: 10nA, scan range: 10µm, scan rate: 100µm/s, resolution: 200, proportional: 1.00, integral: 0.30, and derivative 0.00.

Image 14 has a smaller scan range and the rest of the parameters are kept the same as the previous image. The image is more detailed because of the scan range.

Conclusion and Suggestions

The images that were obtained with the atomic force microscope demonstrate that

the Explorer scanning probe microscope is capable of scanning in the micron scale.

Although, the scanner files were not update for the Discoverer scanning probe

microscope during the ten-week research period, it will be a task that will be

accomplished in the fall semester of 2002. The next step that has to be done would be to update the software along with all scanner files and also purchase some non-contact cantilever tips if we are to continue with the project of scanning asphaltene micelles. Micelles are soft samples and will get damaged if they are scanned in the contact mode.

Acknowledgements

I would like to thank the REU-NSF program and all the staff for giving me the opportunity to conduct research this summer of 2002. I would like to acknowledge Dr. G. Ali Mansoori for being my mentor and giving me the opportunity to work in his lab. I would like to acknowledge all professors who participated in REU-NSF program. I would like to thank John Sitasz for helping me install the microscopes and Alan Ponder from Topometrix Company for technical support. All are greatly appreciated.

References

- G. A. Mansoori, "Advances in Atomic and Molecular Nanotechnology," *Nanotechnoloty: The Emerging Cutting-Edge Technology*, UN-APCTT Tech Monitor, Sep-Oct 2002 Special Issue
- G. Binnig, C.F. Quate, Ch. Gerber, "Atomic Force Microscopy," *Phys. Rev. Let.* 56 (1986) 930-933
- 3. Dharmendra Patel, James R. Smith, Andrew W. Smith, Nigel Grist, Paul Barnett, John D. Smart, "An Atomic force microscopy investigation of bioadhesive polymer adsorption onto human buccal cells," *Int. J. Pharm.* **200** (2000) 271-277
- M. Wendel, B. Irmer, J. Cortez, R. Kaiser, H. Lorenz, J.P. Kotthaus, A. Lorke, "Nanolithography with an atomic force microscope," *Supperlattices and Microstructures* 20 (1996) 349-356
- 5. Peter Markiewicz, M. Cynthia Goh, "Atomic force microscope tip deconvolution using calibration arrays," *Rev. Sci. Instrum.* **66** (1995) 3186-3190
- 6. J. R. Smith, "A quantitative method for analyzing AFM images of the outer surface of human hair," *J. of Microscopy* **191** (1998) 223-228
- L Theil Hansen, A Kuhle, A H Sorensen, J Bohr, P E Lindelof, "A technicque for positioning nanoparticles using an atomic force microscope," *Nanotechnology* 9 (1998) 337-342
- 8. S. Thalhammer, R.W. Stark, S. Muller, J. Wienberg, W.M. Heckl, "The Atomic Force Microscope as a New Microdissecting Tool for the Generation of Genetic Probes," *J. of Structural Biology* **119** (1997) 232-237
- 9. Slamet Priyanto, G. Ali Mansoori, Aryadi Suwono, "Measurement of property relationships of nano-structure micelles and coacervates of asphaltene in a pure solvent," *Chemical Engineering Science* **56** (2001) 6933-6939