Hands-on Laboratory Experience with Scanning Probe Microscopy: Challenges and Opportunities

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Abstract

Possibly the premiere tool developed thus far through the nanotechnology revolution, the Scanning Probe Microscope (SPM) provides not only imaging at the atomic scale but measurements of material characteristics such as mechanical and electromagnetic properties. The knowledge of and experience using the SPM allows students the opportunity to explore electromechanical attributes of materials at the nanoscale and to modify the material surfaces with additional capabilities of this instrument. Extending the engineering technology curricula into the nanoscale not only contributes to the understanding of the discipline but provides vision into the current and future trends of the industry. This paper describes the implementation of a SPM laboratory experiments for undergraduate and graduate Engineering Technology students. The laboratory is associated with introductory nanotechnology courses which will be described briefly. The primary focus will discuss the challenges and opportunities of offering a hands-on laboratory using such a sophisticated piece of equipment. Challenges include the limitation of equipment, training of student, availability of trainers, scheduling and the cost of maintenance. Opportunities include the extension of knowledge to the nanoregime, improved learning, increased curiosity of students and interest in graduate education or research positions. Other students have already taken positions within the nanotechnology industry directly following an undergraduate degree and have been highly successful. This paper will describe multiple years of laboratory experiences at both levels of learning with a variety of approaches and results.

Introduction

Nanotechnology is a new and exciting field providing great opportunities to expand the field of electronics as well as to address its multidisciplinary nature. In nanotechnology classes students are provided with the theory and concepts of the nanoregime. It is equally important to introduce students to the tools of nanotechnology providing hands-on experience to emphasize the concepts and allow students to expand their knowledge. It would be very difficult, impractical and almost impossible to provide the students with the necessary knowledge and the skills to use a versatile tool like SPM without any hands on experience with an actual system accompanied with the basic theoretical background of principle of operation. This practical exposure is provided by a SPM laboratory experience associated with the course(s).

Two nanotechnology courses (one graduate and one undergraduate) were developed in the School of Engineering Technology. Each course provides and introduction of the topic and background material is reviewed to ensure all students remember their basic physics, chemistry and biology which is required for the course. A primary portion of the course is dedicated to the downscaling of electronics into the nanoregime. Other sections of the course include the tools of nanotechnology, nanomaterials, applications, commercialization and ethics. The course includes a laboratory component which is primarily built around hands-on experience with the SPM.

SPM is a family of surface characterization tools that can probe the nanoregime thus it is well suited to be introduced in a nanotechnology course. During the lab sessions of the course, students are introduced to AFM [1] which is only one of the techniques available with the SPM family. Once the students understand the concepts and practical aspects behind the AFM and achieve the skills required it is not difficult for them to expand on it and learn other techniques available within the SPM family with minimal help. Thus the knowledge students gather from these lab sessions are applicable not only for AFM but for the various other techniques available within the larger family of SPM.

Using AFM alone a large range of samples can be characterized. With the correct tip selection and optimization of scan parameters, the same AFM technique can be applied to characterize the surface of hard samples like calibration grids, silicon wafers, mica and metal films, and semi soft samples (i.e. leaves) all the way to very soft samples (i.e. biological cells.) Another big advantage is minimal or no alteration of sample is required when it is characterized by AFM compared with other techniques available. For example scanning electron microscopy requires fixing the sample on a special substrate and covering it with a thin layer of metal. The AFM imaging is non destructive in most of the situations. However, samples may be damaged if they are extremely soft and may be altered if they are very sensitive to the pressure applied by the AFM tip. AFM can also be used to monitor ongoing/dynamic processes. AFM can be performed in ultra high vacuums, in ambient conditions, in air and in fluids. This allows for characterization of samples in their native environments. The high resolution combined with the flexibility makes AFM a very powerful tool in surface characterization and allows for probing a variety of samples within various disciplines. In addition to measuring the topography, AFM can be used to probe various other properties of the sample under investigation. Using AFM, properties like elasticity and friction of a sample can be measured. In addition AFM can be used manipulate the sample with nanometer precision allowing fabrication at the nanoscale. Equipped with the knowledge of how to operate an AFM alone, a student is capable of characterizing vast range of samples under very different circumstances.

Equipment and Materials Used

The Innova SPM system from Bruker was used for the training purposes. This system is capable of 100 µm scan size in both X and Y directions and variation of 7 µm in the Z direction using the large area scanner provided [2]. Along with the AFM system itself, other accessories such as chip carriers and cartridges were used for training. The Innova system is equipped with different cartridges for different imaging modes. For this training, contact mode and tapping mode cartridges were used. Innova has many attractive features making it a good system for training students. One such feature is the availability of Innova optics and the winTV32. The visual from the optical camera is accessible to the user through the software winTV32. This live video aids in laser alignment, approaching the sample, and locating an area of interest on the sample when it is not homogeneous. When approaching the sample, the ability to keep track of the distance between the cantilever and the sample by direct visual from a side view and by the camera using the view from top is very helpful. This allows for the cantilever to be brought reasonably close to the sample without crashing on to it without much difficulty. This feature is even more attractive considering the fact that most users of the system are students who don't have any prior AFM experience. Once focused on the sample, the ability to zoom in and zoom out without changing the focal length is also an attractive feature. Another major advantage of the Innova system is the configuration as a sample scanner. The piezo is placed on the stationary part of the system instead of the movable head. This reduces the risk of damaging the piezo which is very sensitive and brittle.

There are some features which were difficult for the beginners and can be better designed. For example, inserting the substrate to the chip carrier is not straight forward and requires some practice before achieving good results. The need for practice inserting the substrate is true for most AFM systems, but other AFM systems (for example the Catalyst from Bruker) have much easier methods for inserting the substrate to the substrate holder. As a remedy for this problem, pre mounted substrates are available for the Innova at a higher price compared to un-mounted substrates. The chip carrier is held in place on the cartridge by a metal clip which can break very easily with extra force. This metal clip is stressed very frequently as the chip carrier is taken out of the cartridge whenever a substrate needs to be replaced and at the end of each lab session. This metal clip is a rather weak point in an otherwise robust design and can lead to early failure of this component.

DNP tips (Bruker) were used throughout the lab experience as these are standard tips used during contact mode imaging and provide a nice range of cantilever spring constants. The substrate has four triangular shaped cantilevers with different sizes. The cantilevers and tips are fabricated from silicon nitride. The tips are handled using tweezers which were specifically designed for the purpose of handling the substrates by providing good grip without chipping the substrate.

Initial training is performed using a calibration grid (Bruker) as the sample. This is a well defined and hard sample with known features which makes it very suitable starting sample for

AFM. The sample is divided into four quadrants. Two of the quadrants have a waffle pattern with 5µm square size and 10 µm pitch [3]. The depth of the pits is 200nm. Other two quadrants have vertical and horizontal lines with 2 µm pitch and depth of 20 nm. The sample is silicon with silicon oxide features on it and it is pre mounted on a magnetic puck.

Additional samples were attached to magnetic pucks which were mounted on the magnetic sample holder when required to image. Samples were attached to the pucks using super glue, floral putty or some other mechanism depending on the sample. Various samples of interest have been investigated over the years including an aluminum foil damaged with static discharge, the silicon wafer of a microprocessor, an optical storage disk, leaves, and red blood cells. Due to the delicate nature of the aluminum foil and the ease for forming wrinkles, the foil was first stretched out on the magnetic puck and taped at the edges away from the scanning area, taking care not to allow the formation of air pockets beneath the foil. Once the foil is secured properly on the puck, the foil is damaged with static discharges. Both the silicon wafer and optical disk were opaque and had a thickness of 1mm or greater and were simply glued to the magnetic pucks. Samples such as leaves and red blood cells require much more extensive sample preparation.

Images were acquired using the software SPMLAB version 7.0 and they were analyzed using the offline version of the SPMLAB. Additional freeware like WSxM accepts the Innova image format and may also be used for analysis.

Experimental Section

Initial training on the SPM system was conducted in groups of three students for a total of six hours divided in to 3 two hour sessions. Once complete each student was given 6 hours of individual time with the system to “drive” the system and obtain the topography of a sample of their choice and analyze the data collected.

During the training sessions, all students were new to the principle of operation behind the AFM as well as the practical aspects of it. Instead of giving a demonstration of acquiring a topography image from start to finish, the process was divided into logical sections and training was carried out section by section. These sections consisted of loading substrate, chip carrier and sample, aligning the laser and mirrors, moving the tip down and engaging on to sample, determining initial scanning parameters, and then optimizing the scanning parameters and capturing images. Each section was demonstrated to the group accompanied by detailed descriptions. During these demonstrations students were encouraged to take notes and prepare a checklist of procedures to follow as well as various challenges that may be encountered and how to troubleshoot. At the end of the demonstration period, each student was given the opportunity to perform each step with instructor supervision. At the end of this training, each student was required to acquire a contact mode topography image of the calibration grid. Once the each student was competent with contact mode imaging in air, a demonstration was done to illustrate the method of obtaining a topography image using tapping mode. Due to the limited nature of available time, the technique of tapping mode imaging was only demonstrated.

Once the initial training was complete, students were allocated six hours of lab time to investigate a sample of their own interest. Students were instructed to select relatively hard samples which are best suited for contact mode imaging and less difficult in the prospects of sample preparation and imaging. At the beginning of each student's individual lab session they were instructed and guided through sample preparation. Few major points were stressed during the sample preparation. One such point was that the sample needed to be relatively flat, devoid of any tilts and sudden step heights which are beyond the z-range of the AFM. Another important issue was the sample needed to be properly attached to the magnetic puck such that there is good adhesion to withstand the lateral forces exerted on the sample in contact mode. If the sample was not properly adhered it would be moved with the tip during scanning, providing erroneous results. Different options available for attaching the sample to the magnetic puck was also discussed elaborating the advantages and disadvantaged associated with the each option. Samples were prepared by students themselves using the guidelines given.

At the end of the sample preparation, students were given the opportunity to work alone with the AFM system, and scan their sample to obtain topography images following the same procedures they learned and followed during the scanning of the calibration grid. Along with the training, some students were equipped with the checklist they had compiled during the demonstration sessions.

Results

All student were able to successfully complete the laboratory experience and obtain topographical images of both calibration grid and their sample of interest using contact mode. Offline analysis of the sample was completed outside the time frame allocated for lab sessions. The resulted in various degrees of success in completion of the analysis by the students. This is being addressed in current and future offerings of the course. Some students didn't take the opportunity to spend enough time and get familiar with the offline analysis software. The following images are topography images students have acquired using Innova AFM system during their lab sessions.

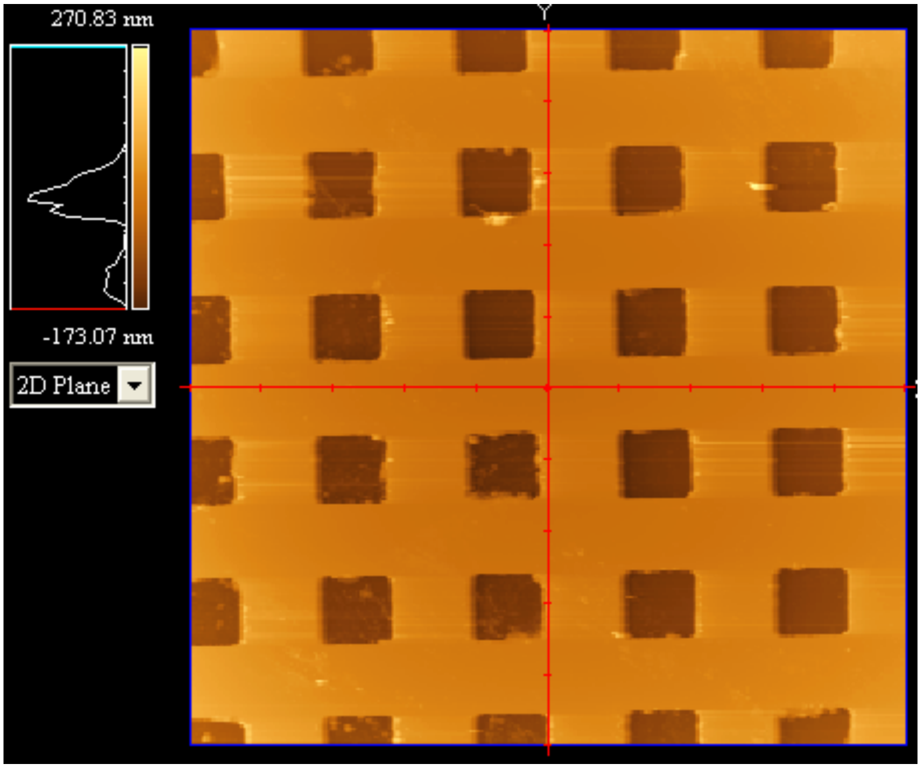
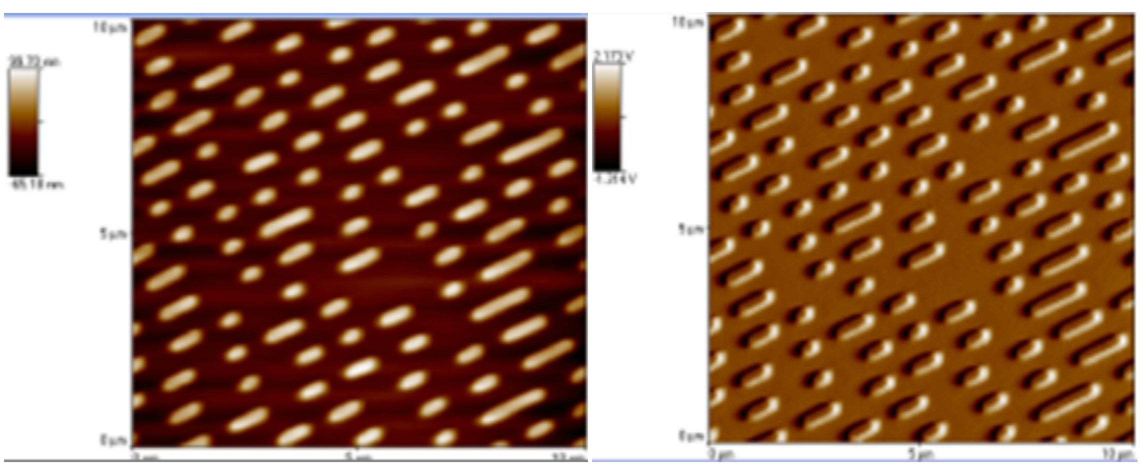


Figure 1: Calibration grid- 50 µm scan

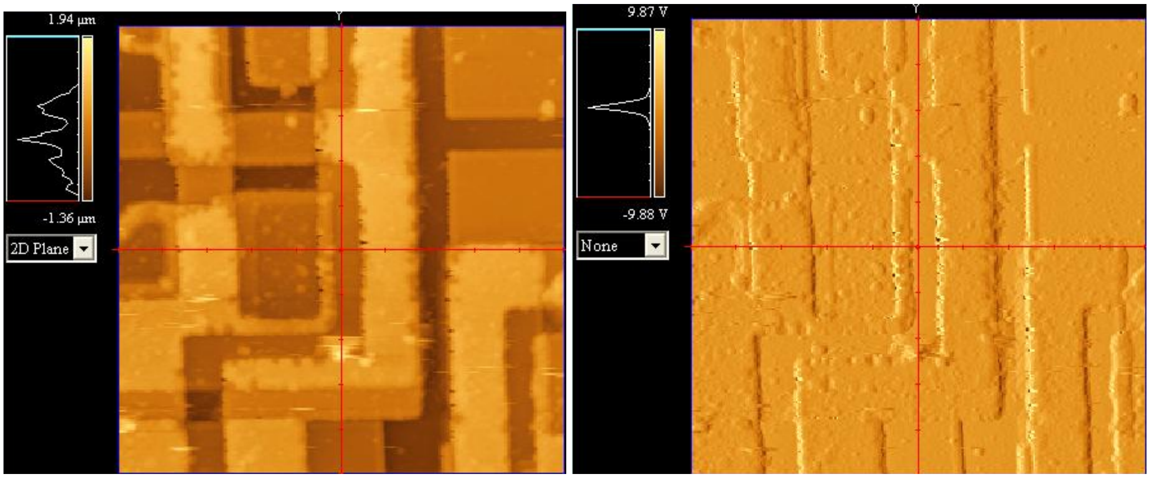
Given in Figure 1 is a topography image of the calibration grid used for training purposes. The scan size is 50 µm and the image is processed using two dimensional plane fit to eliminate the effects of thermal drift.



(a) (b)

Figure 2: AFM images of an optical storage disk.

Figure 2 shows AFM images of an optical storage disk in the format of DVD. The scan size is 10µm and the topography image of the DVD data storage surface is given by Figure 2(a) while the deflection error image of the same scan is given by Figure 2(b). Each small feature given in the image represents one bit of data and larger features corresponds to multiple bits.



(a) (b)

Figure 3: AFM images of a silicon wafer from a microprocessor

AFM images of a silicon wafer from a microprocessor are given in figure 3. Figure 3(a) corresponds to the topography image and Figure 3(b) corresponds to the deflection error image. The scan size of the image given here is 50 µm and the image is acquired with a resolution of 256×256.

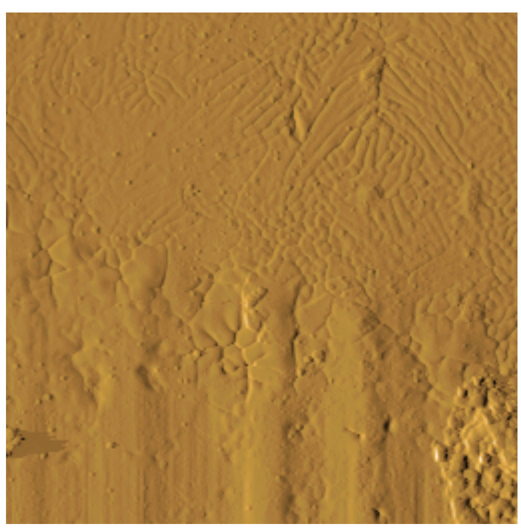


Figure 4: The effect of static discharge on an aluminum foil imaged by AFM (50 µm scan size)

The deflection error image of an aluminum foil damaged with static discharge is given in Figure 4. The grainy surface resulting from the manufacturing method of the intact aluminum foil is clearly visible in the lower portion of the image. The upper portion of displays the deformation produced by the static arc discharge melting the aluminum surface destroying the pattern previously present on the surface.

Conclusion

Hands-on experience with SPM has been conducted successfully for seven years including seven undergraduate and five graduate versions of the course. The laboratory experience has emphasized the material presented in lecture and allowed student to investigate samples of their own interest. At the end of the training session each student was able to acquire the skills and knowledge to be competent on using an AFM in contact mode image. This was verified by their ability to independently image their sample of interest. In addition students received a fair exposure to sample preparation techniques for AFM. Considering these aspects, the training sessions were highly successful and target objectives were achieved. However there is a lot of room for further improvements. The amount of time allowed for each student on the system is minimal and results from the fact only one SPM is available. This also results in logistic and maintenance issues which challenges the instructor and department. Fortunately a good relationship exist with Bruker. Their support has been significant for continuing the nanotechnology courses with an associated lab experience.

References

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