AC 2011-2026: VISUALIZATION AND MANIPULATION OF NANOSCALE COMPONENTS INSTRUCTION FOR ENGINEERING TECHNOLOGY STUDENTS

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Visualization and Manipulation of Nanoscale Components Instruction for Engineering Technology Students

1.0 Introduction

Visualization systems for nanoscale components can offer the opportunity to study existing structures without the need for the facilities and equipment to fabricate them. Life-sciences faculty and students are well-acquainted with optical microscopy as a discovery tool for organic structures, and materials science courses have used such systems to visualize grain structures in alloys and for other purposes. Optical microscopes[1] have limitations due to physical phenomenon in lens materials and the need for shorter wavelengths of light than discernable by the human eye to differentiate objects of closer spacing excludes these instruments from use at the nanoscale. The scanning electron microscope (SEM) provides a solution through its use of an electron beam and scattering electrons, but requires a high vacuum environment for the sample area as well as preparation of non-conductive samples by sputtering or other deposition techniques.

The atomic force microscope (AFM), developed by Benning, Gerber, and Quate at IBM-Zurich/Stanford Research in 1985 followed the invention of the scanning tunneling microscope in 1981 by Binnig and Rohrer.[2,3] This instrument, also a member of the scanning probe microscope family, utilizes a finely profiled nanoscale tip (probe < 10nm) at the end of a flexible mechanical cantilever to progressively scan across the surface of the sample to be visualized. Probes are typically made from SiN4 or Si, but other materials are used for specialized studies. A laser beam reflecting from the back side of the tip provides a moving light source to a phototransistor array, providing detailed z-axis probe deflection and other data in the process that is stored in the computer relative to spatial variation in the x-y plane. This data is used to generate the topographic image of the sample surface. The amount of force between the probe and sample is dependent on the stiffness of the cantilever and the distance between probe and the sample surface which can be calculated using Hooke's Law. If the spring constant of cantilever (typically ~0.1-1 N/m) is less than surface, the cantilever bends and deflection is monitored.[4,5]

$$\text{Force (F)} = -kx$$

k= spring constant, x= cantilever deflection

The dominant interaction forces between the probe and sample at short range of a few nm are Van der Waals, which are fundamentally quantum mechanical in nature. Long range interactions at the range of 100 nm include capillary, electrostatic and magnetic forces. There are three primary imaging modes. [5,6]

1. Contact mode at a probe-surface separation of < 0.5 nm where the probe predominately experiences repulsive Van der Waals forces
2. Non-contact mode at a probe-surface separation of 0.1-10 nm where the probe experiences attractive Van der Waals forces

3. Intermittent or contact or tapping mode at probe-surface separation of 0.5 -2 nm.

Furthermore, the interaction between the AFM tip and the sample surface can be exploited in a number of ways to learn more about the sample physical properties or, in the limit case, be used to alter the surface in a deliberate manner, as is the case with AFM manipulation and nanolithography, where selective removal or deposition of surface materials can be realized. Visualization using AFM can also be achieved for special applications using phase imaging, magnetic force microscopy, scanning tunneling microscopy, visualization in liquids and single point microscopy. [6]

Development of an AFM facility and curriculum offered the opportunity to study a number of physical and material properties across many disciplines. In the years since the invention of the AFM, several manufacturers have developed both general purpose and specialized AFM tools for industry and educational use. Recent developments have brought some of these instruments down in price and increased portability, making outreach activities and shared use more of a possibility. Further advances in computer interfacing and networks also facilitate remote operation of an AFM, providing the possibility of expanded resources without the expense for additional instruments. These factors all led the team to the choice of AFM as a visualization tool.

The purpose of our paper is to discuss the development of instructional material for visualization of nanoscale components by identifying low cost portable and table top AFMs. This paper is divided into four major areas: Section 1 introduces the visualization of the nanoscale components and project motivation. Section 2 discusses the selection of portable and desktop atomic force microscopes. Section 3 discusses the development of instructional material and a sample of instruction material. Section 4 discusses the dissemination of the project by way of organizing workshops and conclusion.

1.1 Project Motivation

Mohawk Valley Community College [7] (MVCC) and State University of New York Institute of Technology [8] (SUNYIT) are both located in Utica, NY and are part of the State University of New York (SUNY) system. The colleges offer engineering technologies programs in electrical and mechanical disciplines at the two and four year levels respectively. Furthermore, a significant number of MVCC students in these programs annually elect to transfer to SUNYIT to complete their studies at the baccalaureate degree. The colleges, as a result, have established articulation and joint-admissions agreements to facilitate this and have collaborated in curriculum areas in the past.

Recently both the colleges have developed and introduced courses in nanotechnology and semiconductor manufacturing technology. This includes a three credit course on “Introduction to Nanotechnology,” a four credit course on “Fundamentals of Microelectromechanical and Nanoelectromechanical Systems” and an Interdisciplinary survey minor in nanotechnology [9] in the electrical engineering technology program at SUNYIT. Additionally, MVCC has developed a Semiconductor Manufacturing Technology course and introduced nanotechnology in different courses across various disciplines. As a result, the need to provide curriculum in nanotechnology topics exists at both the colleges, and a collaborative development could facilitate students beginning at one institution continuing their work at an advanced level at the other institution. In reviewing the options for the creation of these materials, consideration of educational value, facility requirements, faculty and student educational and experiential knowledge, and timeliness were all factors to be considered. A conclusion from this was that creation of curriculum and laboratory facilities to address visualization of nanoscale phenomenon and structures offered the best overall solution, since visualization can be used as a learning tool not only in the earlier-stated technology areas, but also in the physical and life sciences as well. This offered further opportunities for collegial collaboration as both colleges have active programs in these areas. These considerations led the team to submit a project proposal in 2007 under the National Science Foundation' CCLI program currently named as Transforming Undergraduate Education in Science, Technology, Engineering & Mathematics[10] (TUES), and the subsequent award from this source has funded the investigation described herein.

2.0 Selection of Atomic Force Microscopes (AFM)

At the outset, it was identified that having an AFM at each college campus would be advantageous from both faculty/student accessibility and for curriculum development. Shared access to both instruments for both campuses was also deemed important, since this would increase the number of students that could be accommodated in a lab setting. Available AFM systems included a great variety of features and options dependent upon intended usage. Some systems lent themselves to a narrower range of uses, such as materials properties analysis, and in so doing were configured to require minimal setup for the intended operation. Others were found to be configurable for more general use, but required optional modules or software to complete a variety of tasks. In general, a survey of the low to medium priced units ($20K to $60K with educational discounts applied) indicated that the following capabilities would be available:

- Contact-Mode Imaging
- Intermittent-Contact or Tapping Mode Imaging
- Scanning Tunneling Microscope Capabilities
- Optional Mode Capabilities (Magnetic Force Imaging, etc.)
- Optical Microscope for sample placement
- Cantilever/Tip Options
- PC-based software for analysis and report compilation
- Vibration Isolation Platforms
- Reference Samples for Visualization

At the same time, the project team was involved in the study of the AFM and the techniques involved in AFM analysis through participation in training events, webinars, review of technical application notes and other workshops sponsored by AFM manufacturers and others and demonstrations of various systems at local and regional colleges. These activities included visits to the Penn State Nanotechnology Applications and Career Knowledge [11] (NACK) Center, College of Nanoscale Science and Engineering at SUNY Albany State University [12], Syracuse University [13] and other facilities, visits from AFM vendors, and discussions with faculty from other universities and colleges with the aim of identifying key features and advantages of various AFM systems. The use of the vendor-prepared samples and samples initially prepared by or provided to the project members offered insight and the opportunity to gain experience with the AFM technique. A visit to the AFM manufacturer's U.S. office and e-mail dialogue provided opportunities to "compare notes" in operation of the AFM and to better understand the capabilities of the newly acquired instrument.

2.1 Selection of the First AFM

As a result of the study and investigation of various capabilities of AFMs, specifications for quotations were developed and bids sought from a number of manufacturers researched. Each manufacturer was requested to quote on training required either on or off-site as part of the bid, and to include sufficient materials and consumables for initial use. From the bids received, the team selected the NanoSurf EasyScan 2 AFM manufactured by nanoScience Instruments [14] as the initial instrument for purchase. Options for contact and intermittent contact mode operation were selected, but none of the optional modes were included, and the vibration-isolation platform was not initially purchased with the system. An extended sample kit was purchased to provide references for lab activity development. This instrument is quite portable, using a laptop computer as the user interface, and was used at both campuses for presentation as well as for outreach activities with college faculty and school groups in the initial phases of the project.

2.2 Selection of the Second AFM

For the purchase of the second AFM a visit by one AFM vendor to a SUNYIT' IEEE Student Chapter meeting provided a demonstration of a newer model AFM. The vendor presentation provided the students and faculty from different disciplines an opportunity to acquaint themselves with AFM technology and possible applications. This presentation was well received and generated student interest in the use of AFM and later involvement in the project. Based on the feedback and experience of purchasing the first portable AFM, specifications for the second AFM system were developed.

It was determined that the second system should be capable of performing extended analysis techniques including MFM (magnetic force microscopy), single point spectroscopy, and lateral force mode. Many material properties can be disclosed through observation of interaction between the AFM tip and the surface while in either contact mode or intermittent contact mode. This required optional mode capabilities, and it was felt that any additional instrumentation or breakout boxes to couple signals to the AFM or monitor those from the tip might best be served by having the second AFM located in a more permanent environment. As such, a less portable system was felt to be acceptable if it met the team's requirements. A bid specification was created and released for a system meeting a number of these criteria and including a vibration isolating platform as well as a scanning force microscopy capability. Subsequently, a second system, a Veeco [15] (Bruker Corporation) Caliber, was purchased and located at the SUNYIT campus in dedicated facilities. This system includes a dual video monitor display, video camera capabilities, a static vibration-isolation solution, and can perform all of the analysis modes described earlier. As was the case for the previous system, on-site vendor training was also contracted for and provided to the team. This was also found to be of great value in developing familiarity with the new system even though the team had become familiar with AFM from the earlier purchased system.

3.0 Development of Instructional Material

From the outset, it was planned that any curricular materials developed by the project be incorporated into existing or planned courses in programs at both institutions. [9] These courses included the Introduction to MEMS course at SUNYIT and the Introduction to Semiconductor Manufacturing course at MVCC, but the team also anticipated interest in AFM on the part of the instructors in related disciplines. As such, a strategy was pursued to provide the instructors of said courses with deployable modules. The model used included a multipage narrative for faculty members who may not be familiar with the specifics of AFM but acquainted with the general topics of visualization. This narrative detailed the history, theory, and operation of AFM, modes of operation, and other topics. Two interactive presentations on the main modes of operation (contact mode and intermittent contact mode) of AFM with references to the extended modes were created. Portions of each of these presentations were pilot tested in lectures at SUNYIT as part of the Introduction to MEMS course. Portions of each presentation were also combined to create materials for faculty presentations at MVCC and SUNYIT and at a recent faculty development workshop conducted in November, 2010 at SUNYIT.

Classroom PowerPoint presentations and demonstrations of the AFM were provided for (SUNY Class ETC 290) using prepared samples and indicating some uses for the instrument. In the MVCC course MT 209 Materials Science, a unit on AFM imaging and a demonstration of AFM imaging was provided using materials from the Nanoscience Advanced Sample kit that included metallic foil and PS/PMMA thin film samples. Further development of sample creation and preparation capabilities is required to broaden the use of the AFM in MT 209 and other courses and work is underway to assess the current lab capabilities and identify necessary equipment.

3.1 Laboratory Activities

The laboratory activities developed included a short description of the main idea, some background, a procedure with data gathering entry, and review questions. Among these exercises was a detailed introductory activity that utilizes the AFM calibration grid as an analysis sample. The main purpose of this exercise is to acquaint the student with the on-screen menus for the AFM, steps necessary to prepare the AFM for use including replacement of a cantilever/tip, and the sample approach and visualization process. Given the expense of AFM tips (beginning at $20+ for basic types) and the need to develop familiarity with the tool, this was felt to be a critical, though time consuming, first step. Later exercises developed for intermittent contact mode and feature measurement assumed that the user was more familiar with the AFM and built on this foundation to begin inquiry into sample properties and improvement of the captured image. As part of the faculty workshop development, excerpted versions of these activities were created for demonstration and participant use.

3.2 Sample of Instruction Material

One of the lab activities developed using the NanoSurf AFM is given below. So called “Screen shots” of the AFM controls are included to facilitate setup and scanning operations, and some default settings for the AFM are provided. The laboratory exercise uses a CD sample for evaluation. This provides a starting point for getting an acceptable image. The methodology in all activities developed was similar, beginning with instruction on loading the sample onto the AFM stage to avoid damaging the probe tip. Use of the video camera to localize the area of study was the next phase. Systems without a camera generally included an optical microscope for this purpose. It is important to keep in mind that the maximum scan area of the AFM is only about 100 x100 μm. The approach starts with an initial scan of the sample followed with a more detailed scan to identify and characterize desired features, perform measurements, or other detailed studies. Other activities developed included intermittent contact imaging and measurement of physical features using the AFM. The following AFM activity was performed to measure nanoscale features.

Atomic Force Microscope Activity 3: Measurement of Nanoscale Features

Equipment Required: Atomic Force Microscope, CD, DVD, and BluRay disc samples

Main Idea: The Atomic Force Microscope uses a sharpened tip with a typical radius of approximately 10 nm at the end. It is possible with such a fine tip to measure surface features on a sample with a degree of accuracy when used in conjunction with accompanying software. In this exercise, samples of digital media including standard music-type compact discs (CDs), digital video discs (DVDs), and BluRay DVDs. In this experiment, the AFM scans samples of each of these disc types to determine the density of information on each by direct measurement of the patterns and tracks on the discs. Intermittent contact (tapping mode) is used in this exercise to avoid damage to the samples being measured.

Background: A compact disc (CD) is a digital media that includes groups of “pits” or depressions in the disc surface that are formed by a stamping process from a master disk. A laser and lens system tracks a single, continuous spiral track. High spots (lands) on the disk reflect more light back and are “read” by the pickup as logic “1” levels and pits reflect less light and are interpreted as the logic “0” levels that make up the digital data stored on the disc. In the CD system, a red laser (wavelength approximately 680 nm) is used to read the disk. Video data requires significantly more storage density, but the same size media is required. This results in tracks that are closer to each other and, in order to accommodate the data, the pit and land sizes must be shrunk to smaller values. BluRay DVD players provide high definition video for HDTV, requiring even more data density. Limits to the resolution with the red laser to denser data exist, and as such, a shorter wavelength laser is required, and the shorter “blue” wavelength laser provides this capability.

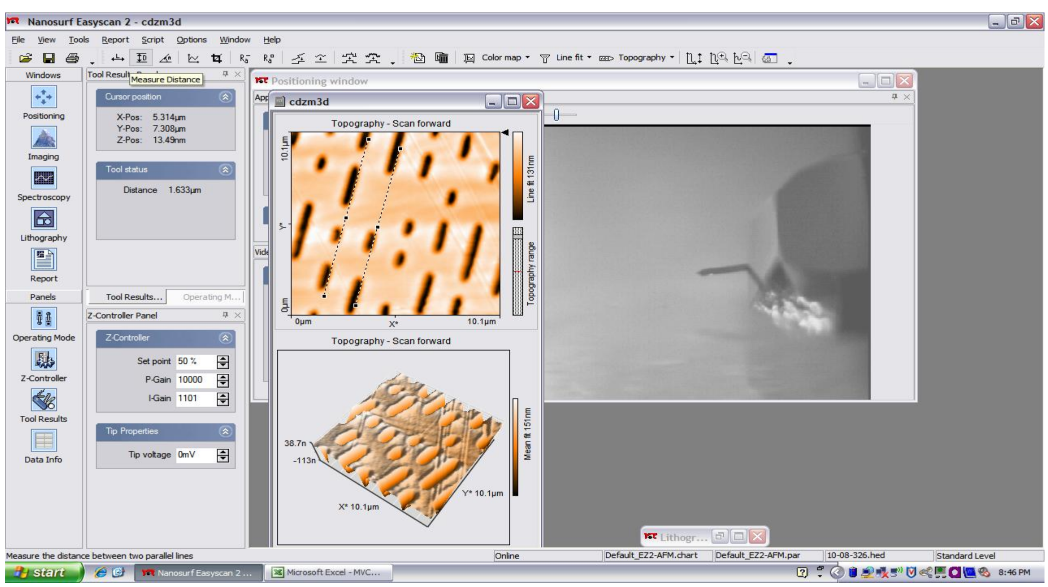
Procedure:

1. Assemble the AFM, inserting an Intermittent Contact cantilever in the AFM head. Adjust the settings on the AFM screen and enter the probe parameters. Note the parameters below.

   Probe Name _________________ Probe Length _________________

   Resonant Frequency ___________ Force (nano Newton) _________________

2. Configure the AFM for intermittent contact mode. Be sure that the AFM cantilever is retracted sufficiently so that it will not be hit by the sample slide. Level the AFM using the bubble level and adjustable legs on the head.
3. Slide the CD sample under the AFM head. Position the sample using the camera view to a clear area on the sample.
4. Set the scan speed to 0.5 seconds/line and the scan size to 44 µm x 44µm. This will provide a fairly large area scan and relatively low resolution, but will provide a "first pass" that you can use to zoom in on a more interesting area of the sample. Use a setpoint of 50%. This means that the maximum force on the cantilever will be limited to a point where the tapping oscillation decreases to at most 50% of its initial value.
5. Use the positioning controls on the AFM computer screen, move the cantilever downward. See the illustration below. Once you are near, but not touching the surface with the cantilever (use the camera view to show this from the top or side), click on the "approach" button on the screen. The AFM will perform a resonance scan on the probe and will begin to approach the sample. Once it is in contact with the sample, scanning will begin.
6. Once the entire surface has been scanned, hit the "stop" button with your mouse and examine the image. A discernable pattern of tracks will appear in the scan, but it won't be possible to measure due to the size. Click on the image and then click the "zoom" button (see the illustration). Now, select an area of interest by holding down the left mouse button to extend the size of the square box. An area of about 10 x 10 µm will show a number of measurable tracks. Once selected, hit the zoom button again. Now start the scan of the zoomed in area using the start button on the screen.
7. When the entire surface of the magnified area has been scanned, hit the stop button on the screen. Click on the camera icon on the screen to capture your image for measurement. Once the image is captured, click on the d key to place a straight line with your mouse parallel to one track. Once the line is placed, the mouse will allow you to place another line parallel to an adjacent track. Click on this point and read the measurement on the screen as shown below.



D = _________ µm (the standard for Audio CDs is a 1.6 µm track spacing).

8. Now click on the L key to measure the pit and land sizes. It is probably easiest to measure a pit (dark area). Note that not all pit lengths are the same. Note the size of pits in your sample.

   L = _________ µm .

9. Once you've measured the CD, use the retract button on the screen to move the cantilever from the surface prior to removing the CD sample. Be sure that the cantilever is well removed from the surface to avoid damaging it when you remove the CD sample.
10. Place the DVD sample in the AFM. Approach the sample using steps 4-9 above, but start with a scan size of approximately 15 µm x 15 µm (see step 5). Use the measurement steps in steps 6 and 7 to measure track spacing and pit size. It may be easier to measure between similar pits.

    D = __________ µm L = _________ µm

11. Retract the cantilever as in step 8. Exchange the DVD sample with the BluRay sample. Repeat steps 4-9 but start with a scan of 5 uM x 5 uM since BluRay pit density is much higher than either DVD or CD pit density.

    D = __________ nm L = __________ nm

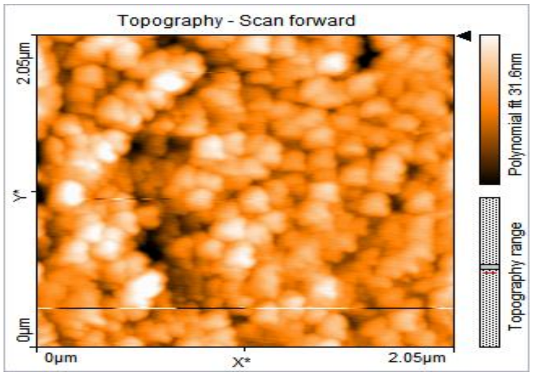
    Note that the BluRay disc pit density is indeed a nanoscale dimension, so in fact, watching a BluRay movie is using nanotechnology!

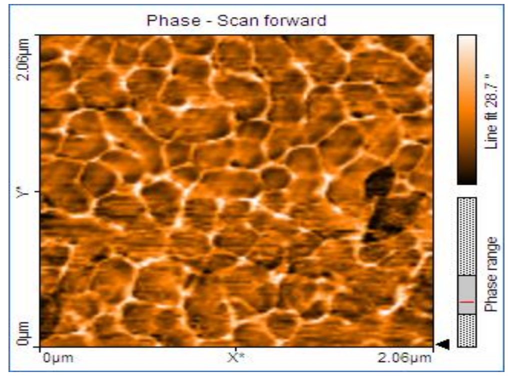
Going Further: The topography screen can show the pit pattern, but through the use of the AFM probe, we can also see the depth of the pits stamped into the surface. Click on the topography image and, from the map type, select 3d map. This view will show the depth of the pits and the shape of the stamping. The AFM typically has a z-axis depth of 8-15 µm, and this is sufficient to probe to the depth of the pits on the CD.

CD-recordable and CD-Rewriteable disks use photosensitive dyes to create dark areas on the disk instead of a stamping operation. The term "burning" a CD doesn't accurately describe what is happening in this case, as no surface depth modification results from writing data to the disk. A laser with sufficient power to actually create these pits would require very careful control.

BluRay DVD disks also contain additional layers on the pit pattern that provide an optically transparent protective coating. Given the nanoscale dimensions of the pit patterns, this is understandable, but since the AFM must actually contact the pit pattern to image the surface, the DVD has to be destructively "dissected" to reveal its geometry.

Additional images were obtained for workshop instruction using contact, non contact and phase imaging modes. Only two are shown below:





Evaporative deposition of Zinc on Glass Phase Image of PS/PMMA film

3.3 Remote Access AFM Facilities

Acquisition of even an entry-level AFM requires a significant investment. In addition, AFM image capture can be an often iterative (and time-consuming) process for new users as there are many variables in settings (scan area, scan speed, number of bits per sample, scan mode, etc.) that may require adjustment to obtain an acceptable image. For those who would prefer to "test drive" on AFM and its capability to image samples of interest, online access to an AFM under the control of a skilled operator provides an opportunity of significant value. The Penn State NACK Center (www.nano4me.org) and others provide facilities for scheduled online remote operation of their AFM systems. Advanced scheduling of the facility and the on-site operator is required and, for remote access, the appropriate sample must be available at the site. Access via networked PC is required, and the use of Skype or other similar tools for voice communications during the session is useful and recommended. Familiarity with the controls for the remote AFM in use is helpful as well if the activity is to be more interactive. In educator and "train the trainer" workshops conducted regularly by the Penn State NACK Center, hands-on access to the AFM is provided for anyone want to use in their class rooms. It was found by the authors that attendance at these seminars was quite beneficial, and information on potential AFM systems was also obtained in the process.

4 .0 Dissemination Activities

A short workshop was organized in 2009 after the purchase of the first portable AFM for faculty and staff during the MVCC Summer Institute. The workshop focused on describing the principle of visualization of nanoscale components and operation of AFM, its capabilities and applications. At the end of the workshop an evaluation was conducted by our designated evaluator. The result of survey data from this workshop was used to prepare materials for the next, longer workshop to be held at SUNYIT.

A second six hour workshop on the visualization of nanoscale components was developed and organized in November 2010. The participants in this workshop included faculty from two year and four year colleges, undergraduate students and personnel from industry. The participating faculty came from Engineering Technology, Physics, Chemistry, Life Sciences and vocational studies disciplines. The workshop consisted of three hours of lectures and three hours of hands-

on laboratory. The lecture part of the workshop consisted of three part of instruction on visualization of nanoscale components using AFMs. The material covered in part one of the lecture included introductory concepts on Atomic Source Microscopy and its applications. Part two of the lecture part included material on visualization by phase imaging, visualization by magnetic force microscopy, visualization by scanning tunnel microscopy, visualization in liquid, single point spectroscopy , description of AFM used and sample of students work. Part 3 of the lecture focused on new trends in visualization of nanoscale components and was given by the personnel from industry. The lecture part of the workshop was followed by a demonstration on the remote access of AFM from the NACK Center of Penn State facility and hands-on laboratory instruction on both the AFMs. The laboratory portion of the workshop included multiple workstations through use of the previously purchased EasyScan 2 AFM and a second EasyScan 2 (borrowed from the vendor for workshop) as well as the Veeco Caliber. The remote access AFM from the NACK Center was also demonstrated for the group during the workshop. Hands-on material was provided to the participants for making the instruction more interactive and useful.
The workshop was wrapped up by the evaluator who conducted a formal evaluation by the participants.

Conclusion

Use of the low cost AFMs for visualization of nanoscale components resulted in enhancing our curriculum in Engineering Technology. The second workshop for faculty and students from an interdisciplinary background showed a positive response and a formal evaluation indicated that the faculty and students are genuinely interested in nanotechnology and their interest in studying physics, chemistry and biology is increased. Interest in the application of AFM to study materials and processes from other disciplines was demonstrated and is being pursued. It was found important to have a “starting point” in using the AFM, and supervision by knowledgeable instructors at the outset to avoid damage to the instrument was critical. Although there is no replacement for individual hands-on experience to develop this competency, the expense of the instrument may make it difficult to have multiple workstations. One option to address these concerns is to provide a number of lab activities to the class at the outset and schedule open hours for the lab with a knowledgeable operator present. More work is needed to develop instructional material and experimental facilities to involve faculty and students across the disciplines. To extend the capabilities beyond basic visualization to analysis of material properties, the extended mode capabilities are required. Although AFM imaging requires minimal sample preparation, the samples must be flat, stable, and planar as the typical z-axis sample heights are limited. Techniques that include Chemical Mechanical Polishing (CMP), thin film deposition, and other processes can be used to prepare samples and also to demonstrate nanoscale fabrication techniques. The remote AFM access at Penn State NACK center is a very useful tool to use in the classroom instruction for learning and becoming familiar with the workings of Atomic Force Microscope.

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Acknowledgement

This paper is based upon work supported, in part, by the National Science Foundation under Grant DUE # 0737204.

Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation