Design of an Undergraduate Atomic Force Microscopy Laboratory for a Materials Science Lecture Course

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Abstract

The availability of relatively low cost, robust, and easy to use atomic force microscopes (AFMs) makes it possible to introduce undergraduates to this exciting materials characterization technique. The University of San Diego's (USD) Engineering Department includes an introductory junior-level Materials Science course required for all engineering majors. In an effort to provide students with a hands-on learning experience in materials characterization, a one-time one-hour lab was introduced in Fall 1998. This lab was designed by an undergraduate electrical engineering student at USD during a summer research experience. The student chose to combine the topic of IC development with microscopy with an emphasis on the AFM. The laboratory was divided into three stations where students examined the same three samples by eye, using an optical microscope, and using the AFM. This allows students to develop an understanding of Atomic Force Microscopy and an appreciation for the viewing power that AFMs provide. Despite some logistical problems, the lab was successful in introducing students to hands-on materials characterization using a modern tool.

Introduction

Since its creation in the 1980s [1,2] the Atomic Force Microscope (AFM) has become an important modern materials characterization tool. It is used in fields as diverse as the examination of DNA to the failure analysis and defect inspection of Integrated Circuit (IC) components. The increasing importance of this characterization technique has made it important to familiarize undergraduate engineering students with the AFM. However, obstacles such as cost and specialized training required before operating the instrument have hindered the access of undergraduates to AFMs. The availability of relatively low cost, robust, and easy to use AFMs [3] overcomes these equipment obstacles. The next challenge is to develop suitable curriculum using the AFM. Curriculum can vary greatly for this tool which is used in a variety of disciplines from biology to chemistry to physics to materials science to electrical engineering. Currently, hands-on undergraduate experience with Atomic Force Microscopy is most commonly found in upper level classes with laboratories or in independent research.[4]

Established in 1986, the young engineering program at the University of San Diego (USD) includes electrical engineering and industrial and systems engineering. The curriculum includes one introductory junior-level Materials Science course required for all engineering majors. With a typical enrollment of 15 students, this course includes three one-hour lectures per week and no laboratory. In the summer of 1998 with funding from the University and the National Science Foundation (NSF) [5], an Optoelectronics Laboratory including one Burleigh Instruments, Inc.

Metris-2000 AFM was established at USD. Thus, for students at USD to work with the AFM required overcoming the obstacles of no lab period, only one AFM, and no student expertise in materials characterization. Because of the enhanced learning for the students in a hands-on experience, we did not consider doing a class room demonstration. Instead, we chose to implement a one-time one-hour lab in Fall 1998. Since this lab would take the place of a class period, students would not be required to come outside of class time. Due to limitations of space and equipment, we chose to split the laboratory into three lab stations. The lab could also be used for classes for non-science majors because of the basic level at which it has been written.

Laboratory Experiment

In designing this experiment, we began with the sample experiments provided by Burleigh in their Metris-2000 Atomic Force Microscope Workbook [6]. These experiments appeared most suited to an entire laboratory period dedicated to one sample. Also, several experiments involved biological materials that would not necessarily be considered relevant by engineering students and some posed liability issues. After consideration of a number of different samples, we decided to have the students examine three different samples throughout the experiment: a ceramic, a calibration grid, and an IC wafer. The ceramic sample was included in Burleigh's sample experiments. It provided a good illustration of a material where different characterization tools yield very different results since it shows nothing to the naked eye, little under the optical microscope, and interesting features under the AFM. Burleigh also provided the calibration grid for use in calibrating the distance measurements of the AFM. This regular array of metal lines on a glass substrate looks like a dark strip by eye. Lines are visible with an optical microscope but are easily measured with the AFM. The sample grid thus gives students quantitative information to help them in understanding the size of what they are viewing. The IC sample provided by one of the authors [SML] increases enthusiasm by including an area of study that is popular amongst most undergraduate students particularly electrical engineers. Inclusion of the IC also led to discussing IC fabrication as part of the first station.

During the laboratory, one of each of the three samples was provided at each of the three stations. Two instructors are required to effectively administer this experiment. The students are split into three groups with each group starting at a different station. The stations are designed to increase the resolving power of the "microscope" and thus the level of complexity apparent on the sample as students move from Station One to Station Three. Station One includes a discussion of IC fabrication provided by one of the instructors. Examples of blank and fabricated wafers as well as mounted devices were used to enhance student learning. In addition, students record their observations of the three samples with the naked eye and attempt to determine the surface features of each sample. At Station Two, students work with an optical microscope. Students describe the differences in what they are capable of viewing compared to Station One, and note any differences in what they think the surface features look like. Most undergraduate students are familiar with the use of an optical microscope, so an instructor is not required at this station. Station Three begins with a brief discussion of the theory and design of the Atomic Force Microscope provided by the other instructor, who in this case was the student who designed the laboratory. Students learn how the microscope is interfaced to a personal

computer (PC) and they become familiar with the software used to control the instrument. One scan of the IC wafer is performed per lab group and pictures of the other two samples are provided from scans taken prior to the laboratory to save time.

In lab, students are asked to draw scale pictures of what they view in order to illustrate the relative size of the objects they observe at each station. Measurements of the observed features are taken to show students how the PC used in conjunction with the AFM aids in measurement of surface features. In their individual laboratory reports, students are asked to explain what role the PC plays in image and data acquisition with the AFM and to write a paragraph on each sample describing how the images differed at each station. Further information including a copy of the laboratory handout and some AFM images is available at www.acusd.edu/usdengr/Opto/Optohome.html.

Results and Student Feedback

After the design of this experiment, two volunteer students tested it in the summer of 1998. As a result of their comments, some modifications were made to improve the clarity and timing of the experiment. In the Fall of 1998, this lab was performed by thirteen students. Student response to the laboratory was quite positive. On the final course evaluations, one student mentioned wanting "more time with the lab". In their lab reports, students were given an extra credit option of providing feedback on the laboratory.

According to the students, the most important thing that they learned was the concept of an AFM, how it can be used, and why it is a useful tool. Several students also remarked on the power of the AFM compared with the optical microscope and unaided eye.

- [The AFM] was a useful tool that gave me insight on how someone could verify the hypothesis of other people.
- There is a need for material science when dealing with integrated circuits. It also helped to see how material scientists work and the tools they use i.e. the AFM. Material science is very closely connected with electrical engineering.

For some students the best part of the lab was learning about the operation of the AFM while for others it was learning more about and observing an IC under the microscopes. Given that these students are primarily electrical engineers and that they have not had a prior semiconductor device course, this interest in the IC is to be expected. Students recognized the usefulness of the AFM's resolving power to materials that electrical engineers use.

- The best part was just getting to observe an integrated circuit- because one day I might be working with them.
- The best part of the lab was looking at the IC sample under the optical microscope. It was really cool to see the patterns etched into such a small area. I am still amazed.
- I think that the observations on the grid and ceramics also put things into perspective on how small of transistors we are really dealing with, which is great because I really had no clue before this lab.

The students' lab reports show evidence of a discovery of a new technology available to them. Most of the students expressed how the AFM provided them with the ability to see features indistinguishable with the naked eye and the optical microscope. The three-stage setup of the lab aided students in understanding the benefits of increasing resolving magnification. Most students' reports were well done. Several students did an excellent job describing their observations at the three stations verbally and with sketches. The student reports also revealed some confusion in the questions posed at the end of the laboratory handout. Some reorganization and rewording of the questions should eliminate this problem and yield even better reports.

Suggestions for Improvement

There were several areas of the laboratory experiment that needed improvement. Most of the students expressed a concern with the amount of time allotted for the lab. One suggestion was to cut the time given to the first station and include the discussion of IC fabrication in a class period prior to the lab. This would allow the instructor at Station One to talk about the operation of the AFM during the first part of the lab enabling the instructor at Station Three to have more time for instruction and questions specific to the operation of the AFM. The hands-on experience with the microscope and the IC sample should not be omitted, though.

There are tradeoffs involved in keeping the time short so that students do not have to spend time outside of scheduled class while providing sufficient opportunity for hands-on investigation with the microscopes. Students expressed a desire to work more with the AFM as well as to have more time at each station. Since each group of students started at a different lab station, two of the groups did not benefit as much from the increase in the level of complexity during the laboratory time. One student group had trouble at the optical microscope station and advocated for having a lab helper available there. The difficulty arose when students could not focus on the necessary features soon enough to gather their data. More detailed instructions of what they are looking for might help alleviate this problem. All three of the stations need to be prepared before the students arrive to prevent delays. It is perhaps most crucial that the instructor at the AFM station insure that the controls for the microscope are optimized and that any necessary pre-scanned images be easily accessible on the computer.

This laboratory was performed in the third week of the semester. By this time, the class had learned about atomic structure and bonding, crystal structure, and defects. The only materials characterization technique that had been introduced was X-Ray Diffraction. Microscopy was covered the week after the laboratory. The timing of the experiment was chosen so that it would occur sufficiently prior to the first exam so that students would not yet be overwhelmed with work. However, in the future, microscopy should be covered in lecture before the laboratory is performed. This would enable more discussion of the material properties that the AFM could aid in determining such as surface morphology, structure, chemical composition, and defects which would be beneficial to student learning.

Finally, class size is an important factor in determining the success of this lab. With classes significantly larger than thirteen students, it might be best to run the laboratory multiple times. Increasing the group size much beyond four students detracts from the hands-on learning of the students particularly at the AFM station.

Summary

The AFM laboratory successfully provided junior level students with a basic understanding of Atomic Force Microscopy. By combining a popular engineering application, IC fabrication, with a complex characterization tool such as Atomic Force Microscopy, an enthusiastic learning environment was created. To allow students a hands-on experience while keeping the entire experiment within a one-hour class period, the laboratory was divided into three stations. During the laboratory, students examined three samples: a ceramic, a calibration grid, and an IC. In Station One, students observe each sample with the naked eye and discuss IC fabrication. Students view each sample using an optical microscope at Station Two and the AFM at Station Three. After performing all three stations, students develop an understanding of the increase in magnifying power and how different tools can provide different data. Students who participate in this experiment benefit in several ways. They gain information about a modern characterization tool available to them that they can consider using in future research and design. They also broaden their understanding of the characterization of materials by experiencing a high-resolution technique used for examining surface topography characterization first hand.

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^{1.} H. Kumar Wickramasinghe, "Scanned-Probe Microscopes", Scientific American, 261, 99 (1989).

^{2.} Robert Pool, "The Children of the STM", Science, 247, 634 (1990).

^{3.} For example, the Metris-2000 by Burleigh Instruments, Inc.

^{4.} For example see J. Amato, Colgate University NSF Grant DUE 9650115,

J. Drucker, University of Texas at El Paso NSF Grant DMR 9724305,

D. Schaefer, Towson State, DUE 9851238, and M. Walczak, St. Olaf DUE 9552159

^{5.} NSF Career Grant ECS-9796220 & NSF Instrumentation & Laboratory Improvement (ILI) Grant DUE-9796201

^{6. ©}Burleigh Instruments, Inc. 1996.

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