The Modern Engineering Materials Instructional (MEMI) Lab: An Undergraduate Learning Experience for Characterizing Materials from the Nanoscale to the Macroscale

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Abstract
A new laboratory curriculum has been developed that provides undergraduate engineering students at the University of Maryland (UMD) with an integrated lab experience that connects the nanoscale structure of materials to the macroscopic physical properties. This laboratory, called the Modern Engineering Materials Instructional Lab (MEMIL), is a new 2241 ft² state-of-the-art teaching facility. Two experimental systems were acquired for this lab to facilitate the testing of small-scale specimens: 1) a custom-built biaxial micro-tensile tester that permits testing of milliscale test specimens, and 2) an integrated nanoindentation/SPM testing system that permits simultaneous mechanical/microstructural characterization over areas smaller than 60 microns. The laboratory is shared across Departments in the College of Engineering to provide a common undergraduate laboratory for the testing and characterization of materials. The sharing of joint undergraduate laboratory facilities across Departments and fields represents a new interdisciplinary view of the undergraduate curriculum that emphasizes micro- and nano-technology, and connection of nanometer and micron scale characterization to macroscopic properties.

I. Introduction
One of the most exciting areas of research in science and engineering today is the field of nanotechnology. Nanotechnology can cover an immense range of scientific endeavors, ranging from manipulation of single atoms to aspects of bioengineering. It is formally defined as: “Nanotechnology: the art of manipulating materials on an atomic or molecular scale especially to build microscopic devices (as robots)” [1]. It is also an area about which undergraduate engineering students are becoming increasingly excited. Unfortunately, this definition is about as close as many undergraduate engineering students get to directly experiencing it, despite the fact that during their careers nanotechnology will become as common place as the use of computers has become in ours. It is therefore imperative that these students be exposed to the concept not just in the classroom, but in the laboratory as well. The best way for a student to understand nanotechnology is through experiments with modern equipment that measures materials properties at this length scale. For this to occur, undergraduate students need access to nanoscale imaging and metrology equipment.

To address this need, the following goals were established:

1) Develop new laboratory experiments that effectively engage undergraduate engineering students in the scientific processes and exploration of the scientific concepts in nanotechnology.

2) Integrate significant advances in nanotechnology from research into the engineering
undergraduate laboratory curriculum through the development of a new interdisciplinary teaching lab with unique capabilities.

To provide students an enhanced laboratory experience, two significant experimental systems were assembled: 1) a pair of micro-tensile testers, and 2) an integrated nanoindentation/SPM testing system. They serve as the core of a new undergraduate laboratory located in the new 140,000 sq ft Jeong H. Kim Engineering Building at the University of Maryland (http://www.eng.umd.edu/kim). This laboratory facility, called the Modern Engineering Materials Instructional Lab (MEMIL), is a 2241 sq ft shared undergraduate lab for materials testing and characterization which is being designed to accommodate the need of at least 3 different Engineering Departments, Materials Science (MSE), Aerospace (AERO) and Mechanical Engineering (MECH), all of whom participated in this effort.

The new MEMI Laboratory at the University of Maryland is based on the concept developed for the Materials Testing Instructional Laboratory (MTIL) that currently exists at the University of Illinois at Urbana-Champaign (UIUC) [2,3]. This laboratory was established in 1994 to provide a central facility for conducting material property tests throughout the college of engineering. The primary motivation was to provide a hands-on experience for individual undergraduate students. Previously, a TA or technician conducted the experiments, and the students were simply passive observers. By allowing small groups of students (4-6) to actively participate in the tests and data collection, it was felt that they would learn the material better and have a more meaningful laboratory experience. An added benefit of this facility was a reduction in costs. Previously, each engineering department maintained separate laboratories, each of which was too small to allow hands-on experiments, contained aging equipment that received little or no maintenance, and was staffed by a new group of TA’s each semester. The MTIL was a great improvement as it consolidated the individual departmental labs into a single modern facility at the College level. Also, a Ph.D. level lab manager was hired to coordinate use of the facility, maintain and purchase new equipment, train TA’s, and provide a sense of continuity. Students from seven courses that span five departments within UIUC now utilize this lab, which is unique among public research universities in this country. Most importantly, the quality of instruction is much higher than under the old organizational structure.

To accomplish the first stated goal, new modular experiments were developed in materials testing and characterization which give engineering undergraduate students hands-on experience with nanotechnology. The testing techniques employed in these experiments clearly delineate the pathway from nanoscopic and microscopic testing to macroscopic materials properties. Laboratory modules were developed which faculty in the participating Engineering Departments can use to design an appropriate laboratory experience for undergraduate students in their program.

The second goal was accomplished through the use of the new experimental testing systems which are generally not found in standard materials testing laboratories for undergraduate engineering students. These new testing systems are used in conjunction with more standard equipment (e.g., microscopic/macroscopic hardness testers, standard tensile testing systems). The specific equipment consists of: 1) a biaxial micro-tensile tester, and 2) an integrated nanoindententer/SPM testing system. The biaxial micro-tensile tester is a tool for measuring multi-axial mechanical properties of materials where specimens are severely constrained by sample volume, sample cost, and/or geometry. It is a custom-designed piece of equipment built at the University of Maryland that was originally developed for research, but has
been transitioned to the teaching laboratory to extend materials testing concepts from the macroscale to the microscale. The nanoindentation testing system permits the characterization of mechanical properties (e.g., hardness and modulus) at the nanoscale, and shows the connection between nanoscale structure and mechanical properties at multiple length scales. The equipment has established the MEMIL in the Kim Building as a model facility for the instruction of undergraduates in multi-scale characterization techniques for modern engineering materials, and provides students with more detailed insight into the structure/property relationships of these materials. The next sections of this paper will review the two instruments acquired for the MEMIL at UMD: 1) Biaxial Micro-tensile Tester and 2) Nanoindenter/SPM Testing System.

II. Biaxial Micro-tensile Tester

The area of mechanical testing of materials using microscale specimens is approximately 10-15 years old. It has evolved primarily as a result of the need to better characterize materials used in Micro-Electro-Mechanical-Systems (MEMS), an area of technological development that has gone from generating curiosities such as wobble motors, to its current phase in which practical devices of common usage, such as accelerometers and pressure-sensing transducers, are being manufactured and used [4]. MEMS devices generally range from a few microns to a few millimeters in size. More work is needed in the determination of the mechanical properties of MEMS elements and the influence of the manufacturing process in these properties, and how the nanoscale structure of the materials used in these devices correlates with the properties. Thus, there exists a need to achieve widespread use of MEMS materials property characterization, which minimally requires the availability of related MEMS mechanical testing equipment, and is relevant to the education of our current crop of undergraduate students.

The most comprehensive reviews to date of mechanical testing of MEMS specimens are those by Ericson and Schweitz [5] and Sharpe [6]. The types of tests and experimental techniques used by these researchers are varied and are performed by three main approaches, namely, static deflection, static tension or compression, and dynamic testing [5]. The overall trend is toward having the capability to make direct measurements on the specimen itself [6]. A biaxial microtensile tester that satisfies this microscale materials characterization need has been built which parallels the existing uniaxial micro-tensile tester built by Sharpe in the Microsample Testing Laboratory at Johns Hopkins University [7-10] (Figure 1). It has been designed with components that accommodate the testing needs of MEMS specimens and thin films as follows:

a) integrated optical microscopy visualization system to precisely align specimens for uniaxial and biaxial testing and strain characterization (Figure 2)
b) force transducer system to measures microNewton loads using miniature load cells (Figure 2)
c) displacement transducer system capable with nanometer resolution using picomotors (Figure 2)
d) PXI data acquisition system for continuous load measurement and picomotor controls with VIC-GAUGE software for continuous strain measurement during testing (Figure 3)

A synopsis of the concepts learned in a a laboratory module that has been developed with the biaxial micro-tensile tester is given below in Table 1.
Table 1: Materials Testing Lab Module with concepts students are learning.

<table>
<thead>
<tr>
<th>Module Title</th>
<th>Concepts Learned</th>
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<tbody>
<tr>
<td>Multi-scale Mechanical Properties of Materials</td>
<td>1. Flexible thin films can wrinkle during uniaxial testing but not during biaxial testing, as revealed through optical microscopy measurements</td>
</tr>
<tr>
<td></td>
<td>2. Properties of thin films differ from standard macroscale systems because of lack of constraint on deformation mechanisms.</td>
</tr>
<tr>
<td></td>
<td>3. Relationship of microscale properties to nanostructure and nanoscale properties will be further explored using Nanoindentation/SPM testing system</td>
</tr>
</tbody>
</table>

Figure 1. Students engaged in biaxial micro-tensile testing of thin film specimen during undergraduate materials course in new MEMIL.

Figure 2. Integrated visualization system in biaxial micro-tensile tester with load and displacement transducers.
At the University of Maryland, the first exposure undergraduate students have to nanotechnology is in various materials courses in the different Departments. The primary focus of the curriculum is to draw a relationship between processing-structure-property from the nanoscale to the macroscale with an emphasis on mechanical properties. A laboratory module has been developed to characterize this relationship at multiple length scales using hardness measurements, seen in Figure 4. At the nanoscale, this module uses an integrated nanoindentation/SPM testing system for metrology.

The nanoindentation system is relatively easy for students to operate, as opposed to more complicated electron microscopy equipment, because the tip of the nanoindenter is also used as a scanning probe microscope (SPM) for imaging (Figure 5). The advantage of using the nanoindenter tip for imaging rather than a conventional AFM is a reduction in the failure of the tip. Because AFMs use microscale cantilevers that are micromachined from brittle materials, like Silicon, they are easily broken due to tensile stresses that evolve during flexure. The nanoindenter tips are under compression, and are made from very hard materials like diamond, whose compressive strength is an order of magnitude greater than its tensile strength. Consequently, these tips almost never fail, and are robust enough to be easily utilized by inexperienced undergraduate students. However, the sensitivity of the instrument requires that students use the instrument in groups of no more than 5 or 6 students.
Hardness Testing at Multiple Length Scales

Hardness is a measure of a material’s resistance to localized plastic deformation (e.g. a small dent or a scratch). Hardness testing is considered a non-destructive test, because it does not destroy the sample as tensile testing does, but rather leaves only a small indentation. This allows hardness to be measured on actual fielded components. For this reason, hardness testing is often used instead of tensile testing to provide a non-destructive measure of a component’s relative tensile strength, which is also a measure of the material’s resistance to plastic deformation.

The most common type of hardness testing for metals is Rockwell hardness testing. Rockwell hardness testing is conducted according to ASTM Standard E-18 “Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials,” which is available in the library. There are several different Rockwell scales which are given as Rockwell C (HRC), Rockwell Superficial 15T (HR-15T), etc. In this laboratory, you will test the hardness at different length scales using Rockwell hardness, Vickers microhardness, and Nanoindentation.

First, you will test your sample at the macroscale using the Rockwell hardness tester. The aluminum should be tested with the Rockwell 15T scale. Test five locations and take the average and standard deviation. Rockwell testing requires only that you set the scale, the major and minor loads, install the indenter, and push “Test” in order to have the hardness measurement displayed. Try another scale as well.

Second, you will repeat the tests at the microscale using a Vicker’s microhardness tester, and note the features that the indentations are obtained from. You will calculate the Vicker’s microhardness, $H_V$, by measuring the length of the diagonals for the indent.

Finally, you will test your specimen at the nanoscale using an instrumented nanoindenter, the Tribolindenter from Hysitron. For the nanoindenter, you will calculate the Hardness, $H$, and Reduced Modulus $E_r$ by selecting a portion of the unloading curve to fit the power law relation, 

\[ P = A(h - h_i)^2 \]

then taking the derivative with respect to $h$ at the maximum $P$ to obtain $S$ and using the predetermined area function $A(h_c)$

\[ E_r = \frac{\sqrt{\pi}}{2\sqrt{A(h_c)}} S \quad \text{and} \quad H = \frac{P_{\text{max}}}{A(h_c)} \].

You will also be able to observe the indent and microstructure of the material using the integrated Scanning Probe Microscopy capability of the nanoindenter.

Questions:

1) What is the difference between the Rockwell Scale and the other scale you chose?

2) What was the standard deviation on the Rockwell scale? How does it compare with the average? If it is relatively high, why might that be the case?

3) How does the hardness of your specimen change with length scale (i.e., Rockwell to Vickers microhardness to Nanoindentation)? Why do you think it is changing? **IMPORTANT: This is asking you to compare measurements – to do that you may need to think of some way to convert the scales.**

Figure 4. Lab for multiscale hardness characterization utilizing the new integrated nanoindenter/SPM.
IV. Enhancement of Curriculum

The new MEMIL at the University of Maryland has had a broad and far reaching impact on the undergraduate education experience in the Engineering College. This facility has enhanced the undergraduate engineering curriculum by bringing it into the modern era of materials and nanotechnology. The two systems acquired in this effort have helped the MEMI Lab become a model for both interdisciplinary collaboration and for integrating research techniques into the curriculum for engineering colleges. Table 3 provides a current list of the courses that directly use the MEMIL on campus. The lab serves over 400 students per semester from various courses throughout the college.

<table>
<thead>
<tr>
<th>Course No.</th>
<th>Course Title</th>
<th>Course Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>ENES 102</td>
<td>Statics</td>
<td>Equilibrium of stationary bodies under the influence of forces. Topics include: vectors, forces, moments, equilibrium, structures, friction, stress, strain, material properties and behavior, etc. Lectures reinforced through lab demos.</td>
</tr>
<tr>
<td>ENES 220</td>
<td>Mechanics of Materials</td>
<td>Stress and deformation of solids - rods, beams, columns, and other structural, machine, and vehicle members, etc. Topics include: torsion, bending, statically indeterminate structures, etc. Lectures reinforced through lab demos.</td>
</tr>
<tr>
<td>ENMA 310</td>
<td>Materials Laboratory I</td>
<td>Characterization of the structure of materials including both single crystal and polycrystalline materials. This is a full semester lab course.</td>
</tr>
<tr>
<td>ENMA 311</td>
<td>Materials Laboratory II</td>
<td>Characterization of the electromagnetic properties of materials. This is a full semester lab course.</td>
</tr>
<tr>
<td>ENMA 362</td>
<td>Mechanical Properties of Materials (with Lab)</td>
<td>Mechanical Behavior, Elastic Behavior, Plastic Deformation, Fracture, and Experiments on Mechanical Properties including Hardness, Strength</td>
</tr>
</tbody>
</table>
Table 3. Current list of courses utilizing the MEMIL

V. Educational Contributions and Benefits

Through this effort, the following distinct contributions were made to motivate engineering students in multi-scale materials testing.

(1) Creation of a new Modern Engineering Materials Instructional (MEMI) Lab that provides students with a 21st century learning experience understanding structure/property relationships in modern engineering materials. This lab serves as a model for other undergraduate engineering education institutions.

(2) Development and installation of a micro-scale biaxial microtensile tester and an integrated nanoindenter/AFM testing system into the MEMIL to serve as the core for undergraduate students to learn about the structure/property relationships of modern engineering materials at multiple length scales.

(3) Development of laboratory modules to integrate the new testing system into the undergraduate engineering curriculum.

A significant benefit of this work is that micro- and nano-technology can now be more easily understood using these testing systems in terms of structure/property relationships in materials from the nanoscale to the macroscale, and the educational curriculum should enable the next generation of engineers to properly utilize these testing systems. We expect that in the long term our effort will lead to engineering students with the knowledge needed to develop micro- and nano-engineered products, which will make them aware of the exciting possibilities of careers.
and graduate study in the area of micro- and nano-technology and enable them to make unique scientific and technological contributions.

Acknowledgements

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References

3. http://mtil.tam.uiuc.edu/