Development of a Student-Centered Hands-on Laboratory Experiment of Chemical Detection using Micro-cantilever Sensor and Optical Lever Amplification Technique *

Geon S. Seo
Department of Aerospace and Mechaical Engineering
University of Arizona

Eniko T. Enikov
Department of Aerospace and Mechaical Engineering
University of Arizona

ABSTRACT

The development of an undergraduate experiment in micro- and nanotechnology based on the detection of chemicals via microcantilever sensors is described. The modified process allows the use of a simple wet-etching station to produce the cantilevers using commercially available substrates, which allows schools without access to clean room facilities to implement the experiment. Simple data analyses demonstrating first-order adsorption kinetics and Langmuir isotherm have also been included to assist in the interpretation of the data. Assessment of the educational impact of the experiment has shown a significant increase in domain knowledge and total engineering design experience of the students. Comparison between groups that have participated in design-only version vs. full-scale hands-on experimentation show increased appreciation of the field of nanotechnology, as well as in the students' perceptions of their marketability.

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INTRODUCTION

The field of engineering involves the application of known scientific principles to harvest the three basic resources of human kind—energy, materials, and information—in order to create useful tools and technologies. Consequently, engineering education has the objective of not only presenting the scientific principles, i.e., engineering science, but also of teaching students how to apply these to real problems. It is not surprising, therefore, that hands-on laboratories have been an integral part of the engineering curriculum since its inception [1]. Their importance has been recognized by the Accreditation Board of Engineering Education (ABET) and its predecessors by creation of criteria requiring adequate laboratory practice for students [2-6]. Unfortunately, during the last several decades, engineering laboratories have become highly complex and expensive, with multiple simulation tools and computer-controlled test and measurement equipment [7-8]. Moreover, ironically enough, the high cost of fabrication and testing of MEMS/NEMS devices often leads to very superficial, demonstration-style laboratory courses, even though this new engineering field can be a unique opportunity to foster important skills by exposing students to disciplines outside of their major. As a result, the students are mere observers in these high-tech laboratories.

One possible solution to the critical need for laboratory experience is to develop a student-centered learning environment based on the use of inexpensive portable micro-experiments. This paper reports on our experience in developing such a low-cost, hands-on exercise in the design and use of microcantilever sensors for chemical detection. While the first demonstrations were primarily focused on the detection of vapors alkanethiols [9], more recent applications have included detection of heavy metal ions (Hg, Cu, As) in liquids [10] and organic molecules [11]. The later are particularly well-suited for classroom exercises, as many issues associated with safe handling of organic vapors have been eliminated. Cantilever beams and their deformation under mechanical loads is a classical undergraduate topic covered in all mechanical engineering programs. The most common method of measuring the deflection of a cantilever is the optical lever technique as shown in Fig 1. The technique works with a focused laser diode beam at the free end of the cantilever, which is reflected onto a position-sensitive detector (PSD). When the cantilever bends due to adsorption-induced surface stress, the reflected light moves on the photo-detector surface, and this movement is proportional to the cantilever deflection. The Stoney formula, which relates the bending of cantilevers to the surface stress given by Eq. (1), is now widely used in surface stress measurements and in the design of cantilever sensors,

$$R = \frac{Et^2}{6\Delta\sigma(1-\nu)}, \qquad (1)$$

where E is elasticity of the cantilever, t is the thickness of cantilever, ν is the Poisson ratio, and $\Delta\sigma$ is difference in the surface stresses between the top and the bottom sides of the cantilever.

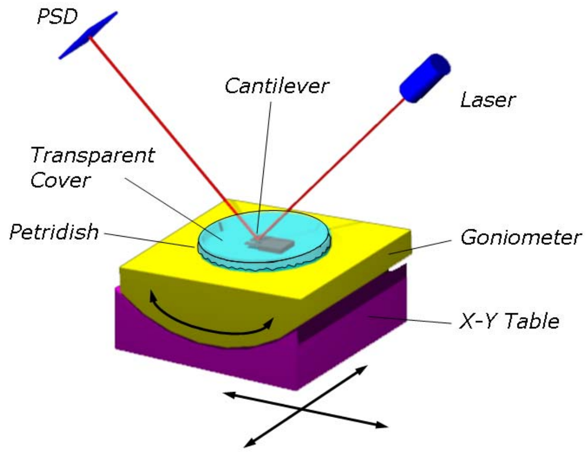


Fig 1. Schematic of cantilever sensor using the optical lever technique.

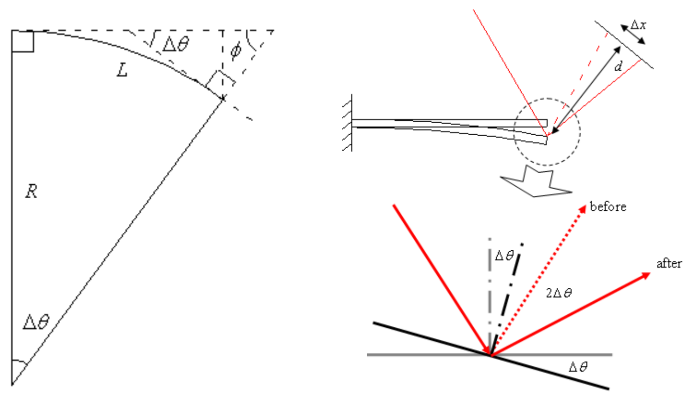
As illustrated in Fig 2(a), assuming constant curvature, the deflection angle $\Delta\theta$ at a distance L from the base of the cantilever is given by

$$\Delta\theta = \frac{L}{R}. \qquad (2)$$

The deflection angle is then amplified using the "optical lever" technique, as shown in Fig 2(b), i.e. by monitoring the position of the reflected laser beam illuminating a position sensitive detector at a distance d from the cantilever

$$\Delta x = 2d\Delta\theta = \frac{12dL\Delta\sigma(1-\nu)}{Et^2}. \qquad (3)$$

Therefore, by monitoring the deflection of the reflected beam, one can determine the surface stress, provided that the elastic and geometric constants of the cantilever are known. This simple geometry and the principle of operation of cantilever sensors make them a good educational tool in nanotechnology. Although many groups reported experiments using cantilever micro sensors, the actual procedures are complicated and some steps demand special and complex equipment, not commonly available at most educational institutions. In this paper, we describe our effort to develop a hands-on laboratory experiment with reduced complexity utilizing inexpensive fabrication process.



(a) (b)

Fig 2. Geometry of beam bending (a), and Schematic of the "optical lever" technique (b).

EXPERIMENTAL SECTION

Preparation of Cantilevers and Chemicals

The microcantilevers used in this work were fabricated by a traditional microfabrication process with dimensions of 300 μm length, 100 μm width, and 560 nm thickness, with a 50 nm thick Au layer. The fabrication process and resulting array of cantilevers are shown in Fig 3. The films are patterned by photolithography and the cantilever itself is formed by wet etching with CsOH. The large lateral etch rate of convex corners results in undercutting and produces freestanding cantilevers. Surface modification with thiolated ligand is performed by immersion of the microcantilever into a 10 mL solution of 1 mM cysteine in ethanol for 2 hours [10]. Upon removal from the ligand solution, microcentilevers are rinsed with ethanol and DI water, and then stored at room temperature in 1 M acetate buffer (AB) with pH 5.

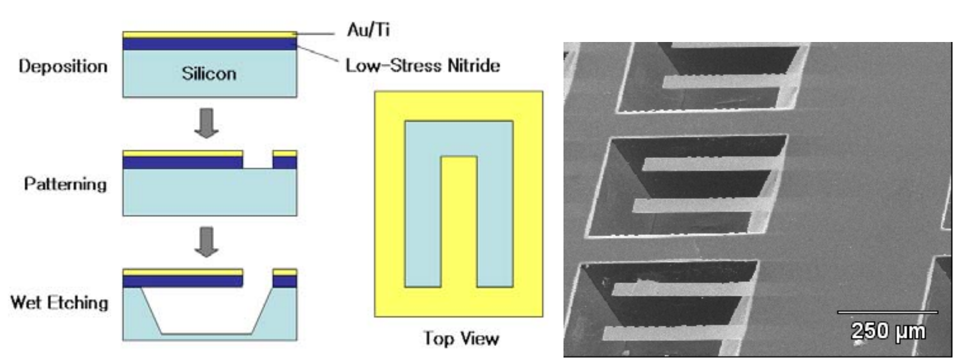


Fig 3. Fabrication process and microcantilevers.

Copper chloride solutions were prepared in pH 5 AB (1 M), which was also used as a carrier solution. An example test protocol consists of a 0.5 mL solution of 2.5 mM copper chloride added into 24.5 mL of AB to make a 25 mL solution of 0.05 mM copper chloride, and a 1 mL solution of 2.5 mM copper chloride added into 24 mL of AB, for 0.1 mM.

Hardware

The optical measurement system has three components: laser source, positioning stage, and a detector, and all subsystems are installed on a portable optical bench as shown in Fig 4. The system was connected to a PC through an RS232 interface; the PC drives the system and collects the experimental data using MatLab. The PSD was integrated into a PCB (PCB1) with a micro-controller (PIC 16F684) and an OP-Amp (AD8554), and another PCB (PCB2) consisting of a dual driver/receiver (Max232) and a micropower regulator (LT1763), as shown in Fig 5. The schematics of the electric circuit, the layout of the PCBs, the associated assembly code and MatLab codes can be found on our laboratory web site [12].

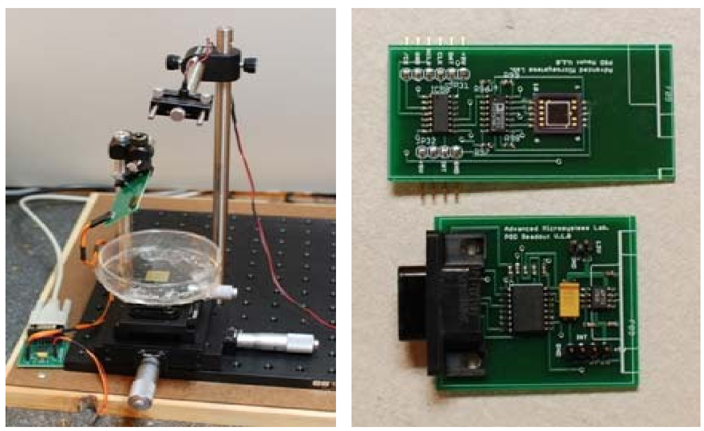


Fig 4. Experimental setup and photodetector PCB.

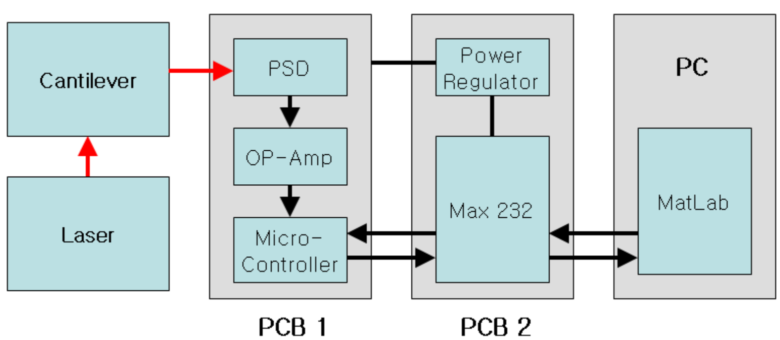


Fig 5. Block diagram of system.

Deflection Measurements

The modified cantilever was mounted inside a Petri dish and soaked in pH 5 AB. The Petri dish was wrapped in a transparent plastic cover that has a small hole through which the copper solution is injected, and it was then placed on the stage. The next steps are to focus a laser beam onto the smallest spot size and to set PSD position and angle. Prior to a detection experiment, the distance between the cantilever and the PSD has to be calculated to allow determination of the curvature or surface stress. The displacement of the reflected beam on the PSD was measured by inducing a known change of angle using the goniometer. The distance between the cantilever and the PSD, d, can then be calculated from Eq. (3). Using a low-power microscope, the laser beam was positioned at the end of one of the cantilevers via the X and Y translation stages. Finally, a copper chloride solution was added to AB in the Petri dish using a pipette, and the resultant deflection was stored in the PC.

ANALYSIS OF EXPERIMENT RESULTS

Figure 7 shows the displacement of the reflected beam on the PSD as a function of time of exposure to copper chloride solutions of three different concentrations. Since the surface stress is proportional to the number of adsorbed molecules [13] and the displacement of the reflected beam on the PSD is also proportional to the surface stress, we have fitted the experimental adsorption curves with the Langmuir adsorption model as follows:

$$\dot{\theta} = k_a cN(1-\theta) - k_d N\theta = A(1-\theta) - B\theta \,. \tag{4}$$

The first-order isotherm solution described as

$$\theta = \frac{A}{A+B}\left(1 - e^{-t(A+B)}\right), \tag{5}$$

where θ is the fraction of occupied sites, k_a and k_d are adsorption and desorption reaction rate constants respectively, c is the concentration of solution, N is the total number of sites on the surface, A is $k_a cN$, and B is $k_d N$. Table 1 shows the constants A and B as a result of the first-order Langmuir adsorption isotherm fitting. According to this result, the time constant and the steady-state value are proportional to the molar concentration of copper ions, and the adsorption and desorption reaction rate constants are also a function of the molar concentration.

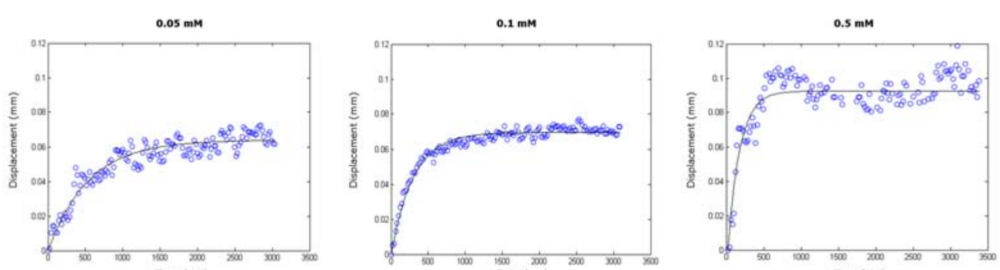


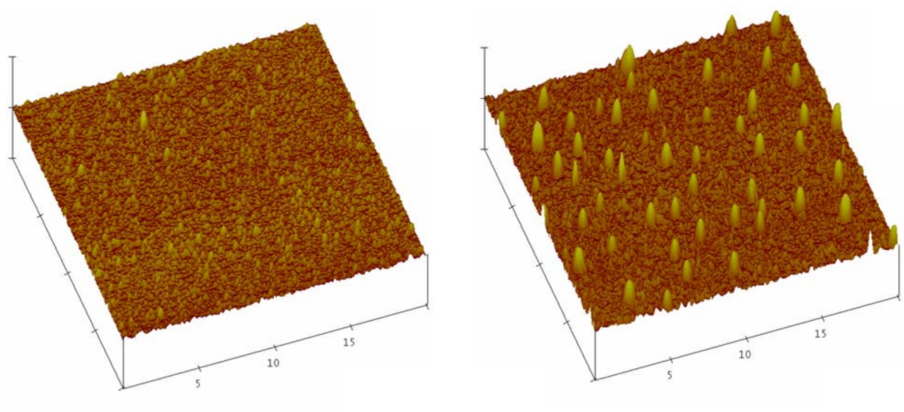
Fig 7. Displacement of reflected beam on PSD (0.05, 0.1, and 0.5 mM).

Table 1. The result of the first-order Langmuir adsorption isotherm fitting.

Concentration	$A = k_a cN$	$B = k_d N$.
0.05 mM	0.0001	0.0017
0.1 mM	0.0002	0.0031
0.5 mM	0.0006	0.0057

PROCESS OF MODIFICATIONS ENABLING USE IN THE CLASSROOM

In a typical fabrication process, the Au layer is deposited after the cantilevers have been etched and released. The rationale behind this is to prevent contamination during etching. However, in a classroom setting, it is convenient to have a stock of pre-deposited wafers so that students can quickly etch their cantilevers. Therefore, we have reversed the order of etching and Au deposition and tested the effect of surface contaminants during etching.



(a) (b)

Fig 8. Three-dimensional images of the gold surface before (a) and after (b) wet etching.

Figure 8 shows AFM scans of the Au surface before and after etching. The r.m.s. roughness value was increased from 3.4 nm to 8.5 nm (about 2.47 times), and the area covered by surface contaminants was approximately 9.8 %, Since the length of cystein is only a few angstroms [14], regions with contamination are most likely completely inactive. Although the roughness of the gold surface increased during the silicon wet etching process, it allows institutions that do not have a cleanroom facility to perform this experiment by purchasing pre-deposited silicon wafers.

ASSESSMENT OF EDUCATIONAL MERIT

In order to evaluate the educational impact of this hands-on laboratory experiment, we compared feedback surveys from two groups of students attending "ABE/AME 489 Engineering

Properties and Micro/Nano Technologies for Biological Systems" in Fall semesters 2005 and 2007 at the University of Arizona. In both semesters, students learned the same topics and performed the same design term project based on a microcantilever sensor. However, in 2005 they designed cantilever sensors and simulated their deflection using computer programs such as ANSYS, SolidWork, and MatLab, and in 2007 the students fabricated their own cantilever sensors and performed the experiment on deflection measurements with them.

Table 2. The results of surveys.

Questions (Answer: 1 - 10)	Fall 2005			Fall 2007		
	Aug. 26	Dec. 9	Diff.	Aug. 2	Nov. 29	Diff.
1. How much do you know about nanotechnology?	3.78	7.33	3.55	3.00	7.00	4.00
2. How much do you know about biosensors?	3.33	7.43	4.10	4.50	8.17	3.67
3. How much do you know about semiconductor processing?	4.44	6.14	1.70	2.00	6.17	4.17
4. Are you considering further studies in nanotechnology?	8.67	8.14	-0.53	5.83	6.00	0.17
5. Do you think nanotechnology provides valuable educational experience?	9.33	8.86	-0.47	8.67	9.33	0.67
6. Are you likely to recommend this class to your peers?	8.67	7.43	-1.24	5.83	7.00	1.17
7. Do you think this course will help you find a better job/grad school?	8.33	7.57	-0.76	5.67	7.50	1.83

We asked the same questions at the beginning and at the end of the class to assess the effect of the course. As shown in Table 2, students acquired a lot of knowledge about nanotechnology and biosensors and increased their total design experience through the lectures and the design portion the course. Also, they obtained knowledge about semiconductor processing during both semesters, but the increment in 2007 was much larger than that in the previous year. Furthermore, the interest in nanotechnology showed a small decrease as students' preferences shifted from a lecture and lab/design course to a lecture-only one. The lecture/simulation-based course seems to have fallen short of the expectations of students in 2005. On the other hand, students in 2007 showed more interest in nanotechnology and their feedback was positive in general. This survey result shows that a hands-on laboratory experiment is a more effective educational approach than a simple computer simulation, and it seems to provide more valuable experience and practical knowledge to students.

CONCLUSIONS

The modified experimental setup and method described here offer a simpler and more convenient hands-on laboratory experiment in nanotechnology. The reversal of gold deposition allows schools without access to clean room facilities to implement the experiment using commercially available substrates. The experimental data allow illustration of micro-technology, as well as classical theorems such as the Langmuir adsorption isotherm. The hands-on laboratory experiments have been shown to contribute to the total design experience of our students, which is required by ABET, and assessment of the educational impact of the experiment has shown a significant increase in domain knowledge and total engineering design experience of the students.

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Biographical Information

Geon S. Seo received his MS degree from Korea Advanced Institute of Science and Technology (KAIST) in 1996 and PhD degree from the University of Arizona in 2004. During his graduate studies in the Advanced Micro and Nanosystems Laboratory, he analyzed the mechanism of actuation and developed mathematical transport and deformation model of ion-exchange polymer/metal composite (IPMC) actuators. Currently, Dr. Seo is a Research Associate in the Department of Aerospace and Mechanical Engineering at the University of Arizona, and his research is focused on the design, fabrication and modeling of micro-electromechanical systems (MEMS) sensors, and the integration of MEMS sensors with biomedical applications.

Eniko T. Enikov is an Associate Professor of Aerospace and Mechanical Engineering at the University of Arizona. He received his PhD degree from the University of Illinois at Chicago in 1998. Since then he has worked as a post-doctoral associate at the University of Minnesota and subsequently as an Assistant and Associate Professor at the University of Arizona. There, he established the Advanced Micro and Nanosystems Laboratory with sponsorship from multiple federal agencies (AFOSR, NSF, DOE, DE) and the private sector. Prof Enikov has also led numerous educational programs including summer school in micro-systems design held in Udine, Italy, 2004, student exchange programs with ETH, Zurich, Budapest University of Technology and Economics, and Slovak Technical University of Bratislava. Prof Enikov is a recipient of several prestigious awards including NSF Career award (2001), US Dept. of State Fulbright Research Scholarship in Hungary (2007).