Published in final edited form as:

J Nano Educ. 2013 June 1; 5(1): . doi:10.1166/jne.2013.1033.

Cal Poly Pomona NUE Project: Implementing Microscale and Nanoscale Investigations Throughout the Undergraduate Curriculum

Kurt Vandervoort^{1,*} and Graciela Brelles-Mariño^{2,3}

¹Physics and Astronomy Department, California State Polytechnic University, Pomona, CA 91786

²Biological Sciences Department, California State Polytechnic University, Pomona, CA 91786

³CINDEFI, Facultad de Ciencias Exactas, Universidad Nacional de La Plata, Argentina

Abstract

NUE funded work at California State Polytechnic University involved development and implementation of nanotechnology modules for physics courses spanning all levels of the undergraduate curriculum, from freshman service courses to senior level laboratories and independent research projects. These modules demonstrate the application of fundamental physics at the nanoscale that complement macroscopic investigations. The introductory level and some of the advanced level modules have been described previously in journal papers and will be outlined briefly here. The main focus of this article, however, is to describe some newer work involving nanoscale experiments that have been developed for senior level laboratories and independent research. These experiments involve applications as diverse as tunneling diodes, gas discharge plasmas for biofilm inactivation, and quantized conductance in gold nanowires.

Keywords

Nanotechnology Undergraduate Education; Atomic Force Microscopy; Tunneling Diodes; Bacterial Remediation; Quantized Conductance; Junior/Senior-Level Experiments

In 2004, California State Polytechnic University, Pomona received an NUE grant (#0406533), "NUE: Linking Introductory Physics Laboratories with Modern Technology by Restructuring Service Courses Around Investigations at the Nanoscale." The activities for the grant consisted of the development and implementation of a number of nanotechnology modules into a variety of physics laboratories. These modules demonstrated the application of fundamental physics at the nanoscale that complemented existing macroscopic investigations. The goals behind these curricular changes were to (1) introduce students to research grade instrumentation at an early level that they may likely encounter in their future careers or in graduate school; (2) create a hierarchy of courses, where students are introduced to advanced instrumentation and experimental techniques at successive stages of complexity from freshman to senior level; (3) provide cross-disciplinary activities that demonstrate the utility and relevance of physics for majors in other disciplines; (4) demonstrate the importance and relevance of nanotechnology in all aspects of modern science. The nanoscale investigations were facilitated through the use of an atomic force microscope (AFM), a Quesant Instruments Universal Scanning Probe Microscope. This

microscope was used to develop eight primary modules for each quarter of the four-quarter introductory calculus-based physics sequence. In addition, these primary modules were modified and adapted for more advanced courses, ranging through senior-level physics. These primary modules and their original spin-offs have been described in more detail elsewhere (Vandervoort et al., 2006a) and are briefly summarized below. Following this summary, we describe our more recent nanoscale projects.

1. ORIGINAL NUE WORK

1.1. Primary Modules

In our first experiment in the introductory physics sequence, students measure the volumes and masses of various shaped objects using rulers, calipers and mass balances and then calculate the densities of these objects with their propagated uncertainties. To expose students to a much wider range of length scales, we included a supplement to this lab that includes measurements at the nanoscale. Students image a non-recordable compact disk (CD) using the AFM and observe rows of bumps separated by $\sim 1.7 \mu\text{m}$. By measuring the spacing and bump size, they ultimately determine the approximate number of bumps that could fit on an entire CD and obtain a value of the same order of magnitude as the storage capacity of a typical CD, ~ 700 megabytes. In addition, students examine an image of copper atoms (obtained by others using a scanning tunneling microscope) and from the spacing of these atoms, and their calculated mass from a periodical table, they obtain the density of copper, and compare to the density they determined from their macroscopic copper objects. The activities provided in the nanoscale supplement reveal to the students the wide range of length scales available to present day and future technology. In addition, students gain important practice in unit conversions over several orders of magnitude.

In an introductory mechanics experiment, students measure the acceleration of masses on an Atwood machine and measure both gravitational and frictional forces on the system. To examine friction on a microscopic scale, students image the surface of highly oriented pyrolytic graphite (HOPG) using the AFM. Because HOPG is atomically flat over most of its surface, defects are easily discernible and normally consist of monolayer steps. Along with the surface topography data, the AFM stores data for the lateral forces on the tip, which are associated with friction, and students compare this frictional image to the image of the surface topography. While most of the features in the frictional force image correspond to changes in surface topography, there are many that do not. The experiment serves to expand student perceptions of the origins of friction as more than solely a consequence of surface irregularities.

In one of the first experiments in electricity and magnetism, students investigate electric fields for various charge distributions by mapping equipotential lines on conducting paper. To complement these investigations, we have inserted a supplement that uses the AFM in force spectroscopy mode, to examine the attractive and repulsive forces between the AFM tip and a gold surface. Students record the maximum repulsive and attractive forces from the data and compare these nanoscale forces to more readily discernible forces, for example, the weight of an ant. The experiment serves to increase student awareness of the complexities associated with interatomic forces. In addition, it demonstrates that charge configurations with zero net charge may exhibit both attractive and repulsive interactions, due to polarization or due even to quantum effects, such as overlapping electron clouds.

In one of the last experiments in electricity and magnetism, students investigate the magnetic field profile of a long solenoid and a short coil. To demonstrate the variation of magnetic fields on the nanoscale, a module was developed to image the magnetic domains on a zip disk, by using the AFM in magnetic force mode, with a special cantilever with a

ferromagnetic tip. The zip disk image contains magnetic domains with widths of about 1.5 microns and lengths of about 10 microns. From these results, students are able to determine the area associated with one bit of digital information (one domain), and from the 4.5 cm disk radius, the total storage area available. The calculation yields 400 megabytes of nominal storage capacity, the same order of magnitude as the designated disk capacity, 250 megabytes.

Students are first introduced to geometrical optics in an experiment that investigates images produced by lenses, involving ray-tracing diagrams, where light is assumed to reflect and refract in straight-line paths. We inserted a supplement to this lab that demonstrates the physical criteria that define the limits for the ray approximation. A smooth glass slide and rough glass slide were sputtered with equivalent thicknesses of gold, which were easily distinguishable by the specular reflective properties (shine) of the smooth slide and the diffuse reflective properties (dullness) of the rough slide. AFM images reveal that the average size of the largest features on the specularly reflective surface is significantly less than the wavelengths of visible light while the average size of the largest features on the diffuse reflective surface is significantly greater than the wavelengths of visible light. The experiment reveals the microscopic origin of the properties of light-surface interactions in a straightforward manner.

In the physical optics experiment, students measure patterns produced by single- and multiple-slit interference. For this experiment, we inserted an AFM supplement where students measure the distance between the rows of bumps on a compact disk and associate this measurement with the reflective interference pattern of the CD. They measure the angles of the two first-order interference peaks, of a laser beam reflected from the CD surface. They then calculate the bump row spacing using the standard Fraunhofer interference equation, compare to their AFM measurements, and obtain agreement typically within 5%. In addition to this technological application of interference, students use the AFM to image the surface of the iridescent morpho butterfly wing, demonstrating a biological application of physical optics.

In the spectroscopy experiment, students investigate the spectrum of hydrogen, using a spectrometer with a diffraction grating to measure the precise angles for the first- and second-order interference lines. In the new supplement to this experiment, students image the surface of the diffraction grating using the AFM and verify the groove spacing that is stamped on the side of the grating. In addition, the AFM image reveals the surface geometry, which is probably different than student perceptions of the grating, as a flat surface with a series of slits. The geometry of the surface is important in terms of the angle of the grooves, also known as the blaze angle.

The microwave optics experiment allows students to investigate the properties of electromagnetic radiation on length scales of a few centimeters. We included a supplement to investigate the purpose of the blaze angle of a diffraction grating that the students observed in their previous week's lab on spectroscopy. From the spectroscopy experiment, students encounter the reduced intensity of the higher-order spectral lines, whose origin is from the diffraction envelope of the pattern. Gratings are often blazed so that the center of the diffraction envelope (where the intensity is highest) is shifted from its normal location, at the center of the interference pattern (zeroth order maximum), to any desired higher order interference maxima. Students perform the microwave experiment using standard physics education equipment for a two-slit interference pattern with and without a Plexiglas blazed *macroscopic* diffraction grating, about 20 cm in length. The macroscopic diffraction grating is machined to the appropriate dimensions to shift the 2.9 cm wavelength microwaves so that the intensity of the central maximum is shifted to the first order maximum of the

diffraction pattern. The experiment provides a way for students to easily visualize the purpose of the blaze angle and exposes them to an interesting engineering application. Significant further details for this module can be found elsewhere (Vandervoort et al., 2006b).

1.2. Primary Module Adaptation to Advanced Courses

Several adaptations of these primary modules were made so that they could be included as stand-alone experiments in our upper level courses. For example, more detailed investigations of the topography of a CD surface were developed as an experiment for our senior level Advanced Laboratory Physics course. Using AFM, students measure the average height of the bumps on the CD surface, an important parameter for the proper operation of the CD. They compare this value to the known wavelength of the laser light used in a CD, 780 nm, and then determine the index of refraction of the polycarbonate material on top of the bumps. The calculation involves the knowledge that the interaction of the laser with a bump and surrounding area requires destructive interference, so that the height of the bumps is precisely 1/4 of the wavelength of the laser light in the polycarbonate material. The students calculate indices of refraction that are typically within 10% of stated values from the manufacturer.

The Spectroscopy module was modified to include a more detailed analysis of the blaze angle. For a transmission diffraction grating that is blazed to shift the center of the diffraction envelope to one of the first-order interference maxima, the relationship between the index of refraction of the glass, n , the blaze angle θ_B , and the first-order interference angle, θ_1 , is given by,

$$n = \frac{\sin(\theta_B + \theta_1)}{\sin \theta_B} \quad (1)$$

The gratings we employ are designed so that θ_1 is calculated for light in the middle of the visible spectrum, 500-nm wavelength. Using their measured blaze angle and equation (1), students calculate the index of refraction for their grating and compare this value to a value they obtain by measuring the index of refraction in the standard way; they shine a laser through the edge of the grating glass and measure angles for incident and refracted rays.

2. RECENT WORK

2.1. Tunneling Diode

The term tunneling concerns the quantum phenomenon that allows a particle, with an energy less than the energy of the top of a potential barrier, to pass through or “tunnel” through that barrier. Numerous experimental verifications of the tunneling phenomenon exist. For example, alpha decay experiments in the 1920's showed that alpha particles are emitted from the nucleus of an atom and are measured with energies less than the energy of the top of the nuclear potential barrier. Therefore, they must tunnel through this barrier before they can be emitted from the atom. More recent examples include the scanning tunneling microscope (STM), invented in 1982, where an electron tunneling current is established between the tip of the microscope and the sample under observation. Measurement of this tunneling current as a function of tip position allows reconstruction of the sample surface topography, often with atomic resolution.

The tunneling phenomenon is one of the most direct manifestations of quantum physics. Observation of this phenomenon requires length scales on the order of nanometers. For example, the STM only achieves a measurable tunneling current (~ 1 nA) when the tip of the instrument is moved to within about 3 nanometers of the surface of a sample. Therefore,

devices that exploit tunneling are textbook examples of nanotechnology and exposing students to these applications gives them important insight into the subtleties of quantum physics.

An experiment was developed for our senior-level Advanced Laboratory Physics course to examine the properties of a tunnel diode. Tunnel diodes were invented in the late 1950's and represented the first way to produce a junction that allowed reproducible measurements of the tunneling current as a function of applied voltage across the device. The invention was so important that the inventor, Leo Esaki, later won the Nobel prize in 1973 (Esaki, 1958). Tunnel diodes have been used in various electronic circuit applications, for example, where fast switching speeds are required. The experiment we developed involves measurements of moderate currents ($\sim$ mA's) and voltages ($\sim$ 100 mV's) for a small but macroscopic device ($\sim$ cm's). Relating these measurements to the few nanometer wide tunneling junctions further enhances our goal of linking macroscopic to microscopic phenomena.

General purpose PN junction and other common diodes are described in detail in introductory textbooks on semiconductor devices (Pierret, 1996). With a couple of exceptions, however, tunneling does not occur in these more common applications. A notable exception is for certain types of zener diodes, which exhibit a tunneling current in the reverse breakdown regime, that is, when a significant positive bias voltage is applied to the N material with respect to the voltage on the P material. This more common application of tunneling is treated in most semiconductor textbooks, while descriptions of the tunneling effect in the less commonly used tunnel diode is not as commonly described. Details of the origin of the unique current-voltage characteristics of a tunnel diode can be found in the following sources (Sze, 1969; Allison, 1971; Beeforth and Goldsmid, 1970; Kwok, 1997).

A tunnel diode is similar in construction to a general purpose PN junction diode. Like a general purpose diode, the tunnel diode consists of a more positively doped P side and a more negatively doped N side. The difference in these two devices is chiefly due to the concentration of doping; the doping concentrations for the P and N regions in a tunnel diode are significantly greater (typically by five orders of magnitude) than the doping concentrations for a general purpose diode. The higher doping concentrations shift the electronic band structure of the device which leads to unusual characteristics; for a certain region of the current vs. voltage curve the tunnel diode exhibits a negative dynamic resistance (defined as $R_{\text{dynamic}} = -V/I$), where an increasing voltage leads to a decreasing current. In addition, this doping decreases the effective width of the junction between the P and N regions known as the depletion zone (to 5–10 nm), a requirement to achieve sufficient tunneling, similar to the minimum width of the tunneling gap required for the operation of an STM.

2.2. Tunnel Diode Experiment

Although not available from most mainstream electronics suppliers, we acquired a suitable silicon tunnel diode from a commercial company (1N4397 silicon tunnel diode from American Microsemiconductor, 133 Kings Rd., Madison, NJ 07940). Students first measure the general current-voltage characteristics of the tunnel diode. For stability purposes, that is, to eliminate oscillations in the negative dynamic resistance regime of the I - V curve, it is best to measure the diode in parallel with a resistor (Bao and Wang, 2006). From the characteristics of our diode, a 68 ohm resistor was appropriate, and was soldered across the diode. The students construct a simple circuit with an adjustable power supply in series with the tunnel diode-68 ohm resistor parallel combination and in series with a 4800 ohm ballast resistor to limit the total current in the circuit to less than 13 mA when the power supply is at its maximum voltage of 60 volts. The power supply is varied so that the voltage across the diode slowly increases from 0 to $\sim$ 700 mV, an adequate range to witness regions of both

positive and negative dynamic resistance. An ammeter is used to measure the total current in the circuit, and the current through the diode is determined from,

$$I_{\text{diode}} = I_{\text{Total}} - \frac{V_{\text{diode}}}{R} \quad (2)$$

where R is the 68 ohm parallel resistance. Figure 1 shows typical I - V data obtained for our silicon diode. Students answer questions concerning the ranges of the curve exhibiting positive or negative dynamic resistance (from the slope in each region) and calculate resistance values at various points in the three regimes.

As an added bonus to the interesting features displayed by the basic shape of the current versus voltage curve of the tunnel diode, there is a secondary feature to these measurements that reveals important insight into the properties of the semiconductor comprising the device. By examining the I - V characteristics in much finer detail, it is possible to determine the phonon spectrum, that is, the energy levels of the quantized lattice vibrations for silicon. Phonons enhance the tunneling effect and by associating these current enhancements with their corresponding electric potentials (voltages), the electric potential energies of these phonons (or energy spectrum) can be ascertained (Chenoweth et al., 1962). To achieve this goal requires several additional instruments and measurements at reduced temperatures to alleviate some of the thermal background noise. The 25 meV thermal background noise at room temperature would mask the minute variations in tunnel current that occur at the phonon energy levels. To achieve the best results, measurements at liquid helium temperature (4.2 K) are required. However, due to cost constraints, we found it sufficient to obtain adequate results at liquid nitrogen temperature (77.4 K). We developed a modernized version of a frequently employed technique (Merrill, 1969) that extracts the first (dI/dV) and second (d^2I/dV^2) derivatives of the current-voltage curve by using a lock-in amplifier to measure the first and second harmonics of an ac voltage applied to the diode. The first and second derivative technique can extract very subtle variations in the slope and curvature, respectively, of the I - V curve and reveal changes due to phonon assisted tunneling not distinguishable in the original curve.

A separate permanent precision circuit was built in a shielded box at the top of a probe with the diode and 68 ohm resistor wired at the probe base, to facilitate easy immersion into a liquid nitrogen dewar. Figure 2 shows a diagram of this circuit and connections to the other necessary instrumentation for controlling the experiment and collecting the data. A laptop computer was employed to control the experiment through a LabVIEW program (National Instruments, 11500 N. Mopac Expwy, Austin, TX 78759) and was interfaced to each instrument (two voltmeters and the lock-in amplifier) through a USB to GPIB controller (General Purpose Interface Bus) and associated connector cables. As in the room temperature experiment, the students obtain a current-voltage curve for the diode, but, using the precision circuit, they obtain many more data points (~200) over a much narrower voltage range (0–70 mV). To facilitate this operation, one of the auxiliary outputs of the lock-in amplifier (Stanford Research model SR830) is connected to the “Ramp” input of the precision circuit and ramps the voltage across the diode, which is monitored by voltmeter 1, as shown in Figure 2. The current through the diode-68 ohm resistor parallel combination is transferred to a current to voltage converter (the op-amp circuit to the right of the diode in Fig. 2), that converts 1 mA to 1 volt, which is then monitored by voltmeter 2. To facilitate measurement of the first and second derivatives of the current-voltage curve, a 200 mV_{rms}, 3 kHz sine wave is used as the reference signal and delivered from the lock-in. This ac signal is attenuated by a factor of 100 and added to the dc ramp voltage through the summing amplifier (the op-amp circuit to the left of the diode in Fig. 2). The final output signal from the current to voltage converter ultimately connects to the input of the lock-in amplifier,

allowing measurement of the first and second harmonics. A prototyping board is used for the ± 12 volt op-amp power supplies.

Students wire the circuit to the external instruments according to the diagram and then take some preliminary measurements to verify that the circuit is connected properly and to establish the range for data collection. They access the LabVIEW program, enter the correct GPIB addresses for each instrument, and collect just a few data points over the desired range of the experiment. They then make adjustments to the desired range by adjusting the potentiometer at the input of the voltage follower, the left most op-amp circuit in Figure 2. Over the range of the experiment, the total current in the circuit will vary by only a few milliamps and the first and second harmonics vary by less than 100 mV and 100 μ V, respectively.

Following the preliminary adjustments, students commence data collection for the archival run, which lasts about an hour. They ultimately transfer these data to a spreadsheet program for further analysis. Figure 3 shows typical results for the precision measurements of the silicon tunnel diode. All three curves, the I - V curve and the first and second harmonics have been plotted on the same graph for comparison. The units on the vertical axis are for the current curve and the other two curves have been scaled appropriately (multiplied by a constant) and shifted vertically (by an additive constant) to maintain the essential features of each curve but allow them to fit on the same plot. Comparison of the I - V curve from figure 3 to the first part of the general data curve from Figure 1, establishes that the qualitative properties of the diode, over this range, have not changed appreciably due to the measurements in liquid nitrogen, although the maximum current has dropped by about 1 mA. Although no obvious changes in curvature occur in the I - V curve, the first harmonic curve undergoes a few changes in curvature and the subsequent second harmonic curve reveals two peaks, a prominent one at 16.5 ± 1 mV and a more subtle peak at 55 ± 1 mV. These peak values in mV correspond to phonon energies in meV. Published results from some of the original work on tunneling diodes (Chenoweth et al., 1962) show very strong peaks for a transverse acoustic and a transverse optical phonon at 18.4 and 57.6 meV, respectively. Comparison to our measurements yields respective errors of 10% and 5%. It should be noted that in the older published data obtained at liquid helium temperatures, three other much weaker peaks were observed corresponding to other phonon energies (as also confirmed through neutron scattering experiments). However, due to the much larger thermal smearing at liquid nitrogen temperature, we do not observe these peaks but can still reproduce the strongest peaks observed at the lower temperatures.

The tunnel diode experiment addresses some of the main pedagogical goals of the Advanced Laboratory Physics course and the main theme of our NUE grant. A primary goal of the course is to introduce students to advanced level instrumentation and analysis relevant to future career preparation. The experiment does this by using a precision circuit, a top of the line dual-phase lock-in amplifier, and precision multimeters interfaced through GPIB. In this experiment, students get their second introduction to LabVIEW (in an earlier experiment in the course, they write a section of a program to control the temperature of a furnace), a software language widely used in industry and research.

Students are made aware of some of the important subtleties of the tunneling experiment through pre-lab quiz questions, directions in the lab manual and follow-up questions they must answer in their reports. For example, the advantages of the GPIB interfacing protocol are emphasized by comparison to one of their previous experiments that involves a 16 bit data acquisition (DAQ) board. The DAQ board has a maximum input range of 10 volts, and on the lowest range of the board (50 mV), the maximum sensitivity is 1.5 μ V. On the other hand, the Keithley 2000 multimeter has an input voltage range up to 1000 volts and a

maximum sensitivity (at its lowest range of 999 mV) of 0.1 μV giving it a much higher fidelity (ratio of maximum to minimum signal) of 9,990,000 than for the board of 33,3333. In addition, the multimeter can measure both current and resistance and can even perform a four point resistance measurement, a technique introduced in one other of the course experiments. Most importantly, the GPIB interface controls instruments for performing specialized tasks, like measuring higher harmonics on the lock-in amplifier.

The utility of the specialized instrumentation is further explored through a more thorough examination of experimental results. For example, from the directly measured current versus voltage data, one can obtain the first and second derivatives of these curves by analysis in a spread sheet program. Figure 4 shows the second harmonic curve, recorded from the lock-in, and shown in Figure 3, along with a curve of the second derivative of the current-voltage data as generated through the spread sheet program. While the peaks from the second derivative curve can be somewhat distinguished, it is apparent that the lock-in amplifier's direct measurement of the second harmonic of the sine wave signal gives a much smoother double peaked curve that can be more easily analyzed. This exercise helps give students insight into the necessity for the lock-in for this experiment and the subtleties of precision instrumentation.

Last but not least, the tunnel diode experiment further demonstrates the possibility of examining microscopic properties through macroscopic measurements. Ultimately, a 5–10 nm wide junction makes it possible for tunneling to occur and voltage and current of reasonable magnitude are readily measured by a simple circuit to reveal the general properties of this unusual device. In addition, using a somewhat more complicated circuit and more advanced equipment, the microscopic tunneling process makes it possible to measure the phonon structure of the core silicon itself, something unachievable for more common diodes.

2.3. Bacterial Biofilms

A significant amount of research has gone into the inactivation of bacterial biofilms. Biofilms form when planktonic bacteria encounter a surface, aggregate and produce a matrix mostly composed of exopolysaccharides and excreted DNA. The matrix encompasses bacteria, making the biofilm less susceptible to standard killing methods. Biofilms are quite common, most bacteria produce them; for example, dental plaque is a biofilm. Due to their prevalence and resistance to most methods of sterilization, many new methods have been proposed and used to remediate bacterial biofilms.

Gas discharge plasmas have been shown to be an effective alternative method for treating biofilms (Brelles-Mariño, 2010). Gas discharge plasmas' mixture of charge particles, radicals and ultraviolet light, provide several constituents harmful to bacteria. At Cal Poly Pomona, students and faculty from the Physics and Biological Sciences departments have been involved in interdisciplinary research using gas discharge plasmas to treat biofilms. Some of this research has involved nanoscale AFM investigations that have provided independent senior thesis projects for our physics students. These student research projects are described below.

Our initial research involving AFM investigations sought to examine changes in bacteria morphology due to plasma applications. Room temperature helium-nitrogen plasmas were generated and applied to biofilms of *Chromobacterium violaceum*, a common organism in soil and water, for periods ranging from 0 (control) to 60 minutes. Biofilms were disaggregated, separating the bacterial cells from one another and from the mostly exopolysaccharic matrix. The detached bacteria were examined by AFM for the control and treated samples. To identify appropriate areas for imaging, an optical microscope (a useful

attachment to our scanning probe microscope) was first used to locate regions where the density of bacteria was low enough to avoid multilayered clusters of organisms. The sample slide was then adjusted with a micromechanical stage manipulator to position the AFM tip over the area of interest. In this way, individual bacteria could be imaged on the flat glass-slide background allowing unambiguous identification. For each slide, at least seven widely separated regions were imaged to obtain a representative sample and ensure reproducibility.

These studies showed that plasma treatments of 5 minute duration were sufficient to render 99% of bacterial cells non-culturable, as ascertained from counting colony forming units, although there was little damage to cell structure, as determined from the AFM images. Further analysis through ATP measurements and fluorescence microscopy showed that many of the non-culturable cells were still viable, that is, alive. After 60 minutes of plasma treatment, however, AFM images revealed major cell damage. In fact, no recognizable bacterial cells were ever observed from any of the 60-minute plasma-treated sample images. These findings demonstrated the utility of gas-discharge plasmas as an alternative sterilization method for bacterial biofilms. Significant further details along with AFM images from this work have been published elsewhere (Vandervoort et al., 2008; Joaquin et al., 2009).

In subsequent research, we studied plasma interactions with biofilms of the opportunistic pathogenic bacterium *Pseudomonas aeruginosa*. These studies involved AFM imaging of the biofilms themselves, after plasma treatments of 0 minutes (control), 5 minutes and 30 minutes. Examining $40 \times 40 \mu\text{m}^2$ scan regions (the largest scanning area possible for our AFM scanning head), the average thickness of the biofilms was sequentially reduced by 6% and 9% following 5 minutes and 30 minutes of plasma treatment, respectively. However, there was a wide variability in the average thicknesses measured for each of the samples and the reported mean thicknesses differed by less than one standard deviation.

In addition to imaging, the AFM was used to measure the micromechanical properties of the biofilm. Force distance curves were obtained, where the sample was moved upward relative to the AFM tip, while monitoring the displacement of the cantilever (or tip), with an associated increased flexure of the cantilever and consequent increased applied force on the sample. Figure 5 shows a diagram depicting this measurement. The piezoelectric transducer that adjusts the horizontal and vertical movement of the sample holder is displaced upward by a known amount, z . The corresponding upward movement of the tip is measured to be x , and the difference, $z-x$, is equal to the indentation of the sample, δ . Since the relative tip movement and corresponding increased flexure of the cantilever is proportional to the applied force through the cantilever spring constant, k , a measurement of x versus z is really a force-displacement curve. It can be shown (Zhao et al., 2005) that the bacterial stiffness, $S = dF/d\delta$, can be derived from the cantilever spring constant and the slope of the x versus z curve, m , through the expression,

$$S = k \frac{m}{1 - m} \quad (3)$$

Therefore, a cantilever displacement–sample displacement curve is directly related to the stiffness of the biofilm, and a higher-sloped curve corresponds to a stiffer sample.

Figure 6 shows a typical force-displacement curve obtained on the biofilm for the control sample. The curve displays the same general features that were exhibited in all of our measured force-displacement curves. Upon approach (black line), the tip encounters the sample surface at the origin of the graph, and deflects upward with a slope that increases. Upon retraction (red line), the tip roughly retraces the approaching curve with some hysteresis. Upon further retraction (in the third quadrant of the graph), the tip adheres to the

surface until it breaks free, and the points retrace the approaching data along the negative horizontal axis of the graph. For the purposes of analysis, two sections of the curve were considered. For approaching, only the initial slope of the curve for positive sample displacements up to 0.2 μm was determined. For significantly higher sample displacements, the data are less reliable, since the optical detection of the cantilever deflection becomes increasingly non-linear. For retracting, the height of the adhesive step, as indicated in Figure 6, was measured.

Biofilms grown on different substrates were investigated. In our initial work, biofilms were grown on one centimeter diameter glass cylinders (coupons) and in later work, biofilms were grown on polished stainless steel coupons. The overall results were that the stiffness of the biofilm decreased after plasma treatment, for the glass coupons, while the stiffness increased for biofilms grown on stainless steel. However, in the adhesive step studies, the results showed that adhesion decreased with increasing plasma treatment, a universal finding for both glass and steel coupons. Average adhesive step heights decreased by 88% and 49% for the biofilms grown on the glass and steel coupons, respectively. This reduction with plasma treatment indicates that the biofilm would exhibit less adhesion to surfaces, deterring its retention. Further details regarding the results of these studies can be found elsewhere (Zelaya et al., 2010; Zelaya et al., 2012).

As in the *Chromobacterium violaceum* work, our studies on *Pseudomonas aeruginosa* included cell culturability measurements by counting colony forming units and showed that this biofilm could be inactivated through the application of gas discharge plasma. The additional information from the AFM measurements indicates that the architecture and the stability of the biofilm as a whole may be impacted by plasma treatment. Our results demonstrate the potential for gas discharge plasmas for bacterial remediation.

2.4. Quantized Conductance

Our most recent research involving nanoscale applications stemming from the NUE grant involves measurements of quantized conductance in gold nanowires. When a voltage is placed across two metal wires, quantized conductance has been shown to occur when the wires are vibrated against one another. As the wires come in and out of contact, nanometer size constrictions form and the conductance of these current paths tends to occur in integral multiples of the quantity, $G_0 = 2 e^2/h$, where h is Planck's constant and e is the charge on the electron. Because of the rapidity of the wire oscillations, data are often recorded on a digital storage oscilloscope, as the current lasts only for a millisecond during the brief wire contact. This physical phenomenon has been extensively studied; in fact, articles describing useful pedagogical methods for demonstrating this effect have even been written (Foley et al., 1999).

So far, at Cal Poly, one physics student has devoted his senior-level thesis project to measuring conductance quantization in gold nanowires. The project consisted of building a suitable circuit and writing a LabVIEW program that would store the conductance data for each measurement trial and accumulate the results of many trials into a histogram, showing the prevalence of data at the quantized values. Our circuit design and experimental procedure were similar to those described in Foley et al. Figure 7 shows a typical trace recorded on the oscilloscope illustrating the quantized conductance phenomenon as the two gold wires separate from one another. Conductance steps occur just below $1G_0$ and $2G_0$ and moderately below $4G_0$. Our ultimate goal for this project is to develop a stand-alone 3-hour experiment, illustrating and analyzing this effect, for the Advanced Physics laboratory, exposing many more of our students to this interesting phenomenon.

In our most recent work, we are developing a way to produce conductance quantization in a more controlled manner. We are using an STM with a gold tip and gold sample and producing the conductance quantization by pushing the tip into and retracting it away from the sample at a precise rate. The efficacy of this method has been demonstrated by numerous investigators and, for our project, stems from our earlier work in a related study (Lewis et al., 1999). Although our commercial scanning probe microscope is provided with an STM head, we found it somewhat prohibitive to alter the existing electronics to make the head suitable for the quantized conductance experiments. For example, because it is measuring a very small tunneling current, the current to voltage transfer function on the preamp of the STM head converts 1 nA to 0.1 volt, a far cry from the 1 mA to 0.1 volt conversion needed for the quantized conductance experiments. Because of this constraint, we are instead using a previously constructed home-built STM head and electronics, similar to an instrument described elsewhere (Vandervoort et al., 1993), that we easily altered to make more suitable for the quantized conductance experiments. Preliminary results look promising. We also find it beneficial to illustrate to students the ability of constructing and implementing their own equipment to do nanoscale investigations.

3. CONCLUSIONS

Eight introductory-level nanotechnology modules were developed to elucidate physical phenomena at the nanoscale that would complement experiments at the macroscopic scale. These original modules spawned further work and nanoscale experiments were developed for junior and senior level laboratories and for student independent research projects. Most recent work involves experiments that measure the phonon spectrum of a tunneling diode, the morphology and micromechanical properties of bacterial biofilms exposed to gas discharge plasmas, and quantized conductance of nanostructures that form between gold contacts. Future work will focus on developing more nanotechnology based experiments to expose students to further applications in this emerging field.

Acknowledgments

Funding was provided by the National Science Foundation Nanotechnology Undergraduate Education Program, award # 0406533, and by the U.S. National Institutes of Health under Grant SCORE SC3 # 1SC3GM088070-01. The authors acknowledge the participation of numerous undergraduate students in this work.

References and Notes

- Allison, J. Electronic Engineering Materials and Devices. McGraw-Hill; London: 1971.
- Bao M, Wang KL. Accurate measuring current–voltage characteristics of tunnel diodes. IEEE Trans. Elec. Dev. 2006; 53:2564–2568.
- Beeforth, TH.; Goldsmid, HJ. Physics of Solid State Devices. Pion Ltd.; London: 1970.
- Brelles-Mariño, G. Environmental and Biological Applications of Gas Discharge Plasmas. Nova Science; Hauppauge, NY: 2010.
- Chenoweth AG, Logan RA, Thomas DE. Phonon-assisted tunneling in silicon and germanium Esaki junctions. Phys. Rev. 1962; 125:877–881.
- Esaki L. New phenomenon in narrow germanium *p-n* junctions. Phys. Rev. 1958; 109:603–604.
- Foley EL, Candela D, Martini KM, Tuominen MT. An undergraduate laboratory experiment on quantized conductance in nanocontacts. Am. J. Phys. 1999; 67:389–393.
- Joaquin JC, Kwan C, Abramzon N, Vandervoort K, Brelles-Mariño G. Is gas-discharge plasma a new solution to the old problem of biofilm inactivation? Microbio. 2009; 155:724–732.
- Kwok, HL. Electronic Materials. PWS; Boston, MA: 1997.
- Lewis BB, Vandervoort KG, Foster RD. Measurements of quantized conductance in gallium as a function of temperature. Solid State Comm. 1999; 109:525–530.

- Merrill JR. An advanced laboratory experiment on tunneling in semiconductor diodes. *Am. J. Phys.* 1969; 37:269–272.
- Pierret, RF. *Semiconductor Device Fundamentals*. Addison-Wesley; New York, NY: 1996.
- Sze, SM. *Physics of Semiconductor Devices*. John Wiley & Sons; New York, NY: 1969.
- Vandervoort KG, Zasadzinski RK, Galicia GG, Crabtree GW. Calibration from 4 to 300 K of the voltage response of piezoelectric materials for use in scanning tunneling microscopes. *Rev. Sci. Instrum.* 1993; 64:896–899.
- Vandervoort K, Hyder A, Barker S, Torrico R. Including nanoscale investigations in undergraduate physics laboratories at all levels of the curriculum. *Mat. Res. Soc. Symp. Proc.* 2006a; 931E:0931–KK03-02.
- Vandervoort KG, Adams SL, Hyder AM. Revealing the blaze angle: A simple experiment for visualizing diffraction effects using microscopic and macroscopic gratings. *Am. J. Phys.* 2006b; 74:649–651.
- Vandervoort KG, Abramzon N, Brelles-Mariño G. Plasma interactions with bacterial biofilms as visualized through atomic force microscopy. *IEEE Trans. Plasma Sci.* 2008; 36:1296–1297.
- Zelaya A, Stough G, Rad N, Vandervoort K, Brelles-Mariño G. *Pseudomonas aeruginosa* biofilm inactivation: Decreased cell culturability, adhesiveness to surfaces, and biofilm thickness upon high-pressure non-thermal plasma treatment. *IEEE Trans. Plasma Sci.* 2010; 38:3398–3403.
- Zelaya, A.; Vandervoort, K.; Brelles-Mariño, G. Battling bacterial biofilms with gas discharge plasma. In: Machala, Z.; Hensel, K.; Akishev, Y., editors. *Plasma for Bio-Decontamination, Medicine and Food Security*, NATO Science for Peace and Security Series A: Chemistry and Biology. Springer; Frankfurt: 2012.
- Zhao L, Schaefer D, Marten MR. Assessment of elasticity and topography of *Aspergillus nidulans* spores via atomic force microscopy. *Appl. Environ. Microbiol.* 2005; 71:955–960. [PubMed: 15691953]

Fig. 1.
Current-voltage properties of the silicon tunnel diode.

Fig. 2. Precision measurement circuit for the tunnel diode. Each operational amplifier represents one-half of an OP227 dual precision amplifier. Circles with black dots in their centers represent BNC input connectors.

Fig. 3. Current-voltage properties of the silicon tunnel diode at 77.4 K, in addition to the first and second harmonic curves.

Fig. 4. Second harmonic data (equivalent to the curve from Fig. 3) and second derivative data of the I - V curve from Figure 3. Each curve has been scaled to portray similar shapes and has been shifted upward or downward for ease of comparison.

Fig. 5.

In a) the amount of sample indentation, δ , is equal to the sample displacement, z , minus the cantilever (or tip) displacement, x . The bacterial stiffness can be derived from the spring constant of the cantilever and the slope of the cantilever-sample displacement curve, shown in b).

Fig. 6. Typical AFM force-displacement curves for tip approaching (black) and tip retracting from (red) the *Pseudomonas aeruginosa* control sample.

Fig. 7.

Typical oscilloscope trace indicating quantized conductance in the gold nanowires. Considering our circuit components and the 25 mV bias voltage that was used, one unit of quantized conductance, $1G_0$, corresponds to 0.194 volts on the oscilloscope trace. The initial plateau at 1 volt corresponds to the maximum measurable voltage for the oscilloscope setting and does not represent a conductance step.