Video laboratory manuals and kits that explain nanoscale science and engineering concepts

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Abstract

The Interdisciplinary Education Group of the Materials Research Science and Engineering Center (MRSEC) on Nanostructured Materials and Interfaces at the University of Wisconsin-Madison, in conjunction with the Institute for Chemical Education (ICE), has created a number of student-centered, nanotechnology-focused educational kits. Designed for high school and college level instructors and students, these kits provide the tools and background information on numerous experiments that can be used to explore nanoscale science and engineering concepts in the classroom. The kits include an exploration of the nanoworld, optical transforms, amorphous metals, solid-state models, nanocrystalline solar cells, and light emitting diodes (LEDs). To provide teachers and students with a safe, inexpensive, easily accessible introduction to nanotechnology, several laboratory modules have been also developed by the MRSEC, including a new module on the synthesis of nickel nanowires using a nanoporous template. After the synthesis procedure is completed, release of the nanowires then allows their properties to be investigated. The module has been used in a number of different types of courses ranging from a nanotechnology course for non-scientists, to a graduate-level special topics physics course. This laboratory experiment is described in more detail by Bentley *et al*.[1] and has been incorporated into the UW-MRSEC Laboratory Manual for Nanoscale Science and Technology at (http://www.mrsec.wisc.edu/edetc/nanolab/index.html). In this presentation we will focus on the nickel nanowire video lab and two kits highly relevant to materials education at the undergraduate level: the optical transform kit[2] and LED color strip kit.[3] We will demonstrate how these relatively inexpensive educational materials can be used in the classroom to help students learn nanoscale material properties.

Introduction

The emerging field of nanoscale science and engineering has tremendous potential to allow scientists and engineers to design materials with unique properties that can improve existing products or form the basis of enabling technologies for new applications. In order to realize this potential, the workforce needs to be literate in nanoscale science and technology.[4] For this reason, there is a large effort underway to incorporate nanoscale science and technology into existing undergraduate and graduate, and precollege curricula.[5] Collaborators in the Interdisciplinary Education Group of the MRSEC on Nanostructured Materials and Interfaces at the University of Wisconsin – Madison[6] have developed a suite of hands-on and video-based tools for teaching nanoscale science and engineering in the undergraduate and precollege (both middle and high school levels) curricula. Here we summarize the resources available and highlight a few examples.

Hands-on Kits

A significant component of any nanoscale science and technology curriculum is the hands-on laboratory experience. In order to address the issues of reproducibility, dissemination, and familiarity, self-contained kits have been developed to improve both the success rate of nanoscale laboratory modules and the adaptability of the lab modules. The kits come with the necessary components and instructions.

Hands-on kits are a good means of disseminating nanoscale demonstrations and experiments. The UW-MRSEC has worked with the ICE[7] to make student-centered, nanotechnology-enriched kits that are readily available and sold at cost. Designed for high school and college level instructors, these kits provide the tools and background information on numerous experiments

Table 1. A list of some hands-on nanoscale science and engineering kits available through ICE

Kit	**Activities**
Solid-State Model Kit[8]	Build crystal structures. Explore the role of atom sizes in determining structure. Visualize physical origin of mechanical properties.
Optical Transform Kit[2]	Observe optical diffraction patterns. Connect to X-ray diffraction as a tool for determining crystal structures.
DNA Optical Transform Kit[9]	Observe diffraction patterns that illustrate the determination of the structure of DNA.
LED Color Strip Kit[3]	Characterize properties of light emitting diodes (LED). Correlate color with photon energy and wavelength.
Amorphous Metal Demonstration Kit[10]	Observe effect of nanoscale structure on mechanical properties.
Exploring the Nanoworld Kit[11]	Become familiar with principles of nanotechnology. The kit is intended for general audiences and includes materials for hands-on experiments.
Nanocrystalline Solar Cell Kit[12, 13]	Build a working solar cell using plant anthocyanins and nanocrystalline titanium dioxide.

that can be used to explore nanoscale science and engineering concepts in the classroom and laboratory. The kits have been designed to contain the essential materials for a set of demonstrations and activities, and usually include a booklet describing the activities and providing some additional background and references for the instructor. However, each kit has also been designed with the flexibility necessary to allow each instructor to adapt and modify the materials to fit into their curricular and classroom or laboratory settings. A list of some relevant kits available from the Institute for Chemical Education is provided in Table 1.

Optical Transform Kit and DNA Optical Transform Kit-- The use of X-ray diffraction (XRD) in determining crystal structures has been one of the essential characterization methods used by all of the natural sciences to examine the nanostructure of crystalline materials. When discussing nanoscale science and engineering, as well as traditional topics such as solid-state chemistry and physics, it is important to explain the techniques, usefulness and significance of XRD. However, for a number of reasons, especially safety, the use of actual X-rays in a classroom or even a laboratory environment, presents a number of challenges to helping students understand and appreciate XRD.

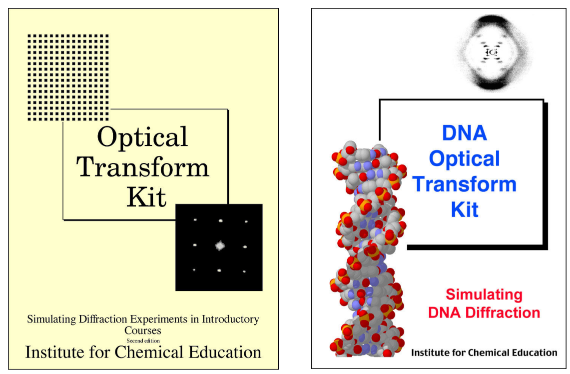

Figure 1. Optical Transform Kit and DNA Optical Transform Kit

Exploring diffraction using visible light and optical transforms designed to mimic crystalline structures is a simple and elegant solution to many of the challenges that using X-rays presents. Instead of using electromagnetic radiation with a wavelength on the order of 0.1 nm (X-rays) to observe diffraction patterns made by periodic features on the order of 0.1 nm (crystalline structures), electromagnetic radiation with a wavelength on the order of 650 nm (red light) is used to observe diffraction from periodic features on the order of 100 μm that are present on a 35 mm slide.[14, 15]

The optical transform kits, distributed by ICE, consist of 35-mm slides with various arrays printed on the slide and a booklet that guides the educator through the process of using the slides in a classroom or laboratory setting. By observing the diffraction patterns resulting from the different arrays on the transform slides, students can discover the fundamental relationships of diffraction based on Bragg's Law. The diffraction patterns can be observed either by shining a visible-light laser through the slide and projecting the diffraction patterns onto a wall or screen; or by holding the slide directly in front of the eye and looking at a point source of monochromatic visible light such as a light emitting diode. Safety issues involved in the use of a laser are described in the kits. Two kits are available for use, as shown in Figure 1.

The *Optical Transform Kit* demonstrates the basics of optical diffraction, leads students through the discovery of the fundamental principles governing diffraction, and makes the connection between optical diffraction and XRD. A more specialized kit, the *DNA Optical Transform Kit,* also discusses these concepts, but focuses primarily on the use of XRD to determine the structure of B-DNA through the optical diffraction analogy.

Figure 2. LED Color Strip Kit handbook with the device

LED Color Strip Kit-- Light emitting diodes (LEDs) are revolutionizing lighting and display technologies, because, relative to incandescent lights, LEDs are more compact, robust, energy efficient, and temporally responsive. Because the semiconducting solids used in LEDs can be grown a few atomic layers at a time, these materials represent an exciting application of nanotechnology that draws upon chemistry, physics, and engineering principles.

The *LED Color Strip Kit* provides teachers with creative, hands-on/minds-on ideas that illustrate the properties of light emitting diodes and semiconductor materials.[16, 17] It may be especially useful in a multidisciplinary science or engineering course that includes topics in materials and technology. The kit, for example, can be used to learn about periodic properties of elements and chemical bonding, electrical conductivity in solids, diode behavior, and properties of light. In addition, discussion of efficient light production and its impact on energy resources are natural extensions of the kit that can lead to better understanding of societal implications of technology.

Video Laboratory Manuals on Nanotechnology

Nanotechnology is an increasingly important crosscutting area of technology. Undergraduate science and engineering courses provide an excellent opportunity to expose students to this exciting interdisciplinary field. To provide teachers and students with a safe, inexpensive, easily accessible introduction to nanotechnology, we have developed several laboratory modules ranging in from basic to advanced. A short list is provided in Table 2.

Table 2. A list of nanoscale science video laboratory manuals. For the complete list please visit our website (mrsec.wisc.edu/edetc/nanolab)

Laboratory Module	**Activities**
Preparation of Cholesteryl Ester Liquid Crystals[18, 19]	Prepare temperature-sensitive cholesteric-nematic liquid crystals that change color.
Synthesis of Colloidal Gold[20]	Prepare colloidal gold nanoparticles. Explore the properties of colloids. Correlate how color of the colloidal gold changes as the distance between the particles changes.
Synthesis of Nickel Nanowires[1]	Synthesize magnetic nickel nanowires using alumina membrane filter as a template.
Microfluidic Nanofilter[21, 22]	Fabricate a simple microfluidic device where interfacial nylon membrane is polymerized *in situ* and used to filter gold nanoparticles.

Nickel Nanowire Laboratory-- One of the more recent additions to the collection of video laboratory manuals is a module on the synthesis of nickel nanowires using a nanoporous template. After the synthesis procedure is completed, release of the nanowires then allows their properties to be investigated. The module has been used in a number of different types of courses at the University of Wisconsin – Madison, Milwaukee School of Engineering, and Beloit College. These courses ranged from a nanotechnology course for non-scientists, to a graduate-level special topics physics course. Lab development has relied upon formative feedback to increase the effectiveness of the experiment in subsequent uses of the module. This laboratory experiment is described in more detail by Bentley *et al.*[1]

1. Apply cathode to one face of membrane

2. Attach membrane/cathode to current collector and insulate with electrical tape

3. Connect AA battery to current collector and Ni-wire anode

4. Add nickel-plating solution to start electrodeposition

5. Dissolve cathode with 6 M HNO_3

6. Release nanowires by dissolving alumina membrane with 6 M NaOH

Figure 3. Experimental procedure

A schematic of the experimental procedure is presented in Figure 3. Anodisc® 25 alumina membrane filters (~50 μm thick, 25 mm diameter), consisting of hexagonal arrays of cylindrical pores approximately 200 nm in diameter, are used as templates to direct the formation of *ca*. 200 nm-diameter nickel nanowires by electrodeposition from aqueous nickel solution. A scanning electron micrograph of the cross-section of a filter is presented in Figure 4. Detailed inspection of the membrane reveals that the pore diameters are ~200 nm through the majority of the thickness, and narrow to ~20 nm on one side.

A cathode is constructed by painting GaIn eutectic on the small-pore side of the alumina filter (Figure 5a). A nickel wire is used as the anode. The coated membrane is placed on a copper current collector, which is then insulated with electrical tape (Figure 5b). The membrane/current-collector assembly and the nickel-wire anode are placed in a beaker, and a 1.5 V potential difference is applied between the copper current collector (negative) and the nickel wire (positive) by a AA alkaline battery. Addition of a commercially available nickel-plating solution completes the electrolytic circuit and results in the pores of the membrane being filled by electrodeposited nickel (Figure 5c). Over the course of 15-30 minutes, students make nanowires, which are 15-30 mm long.

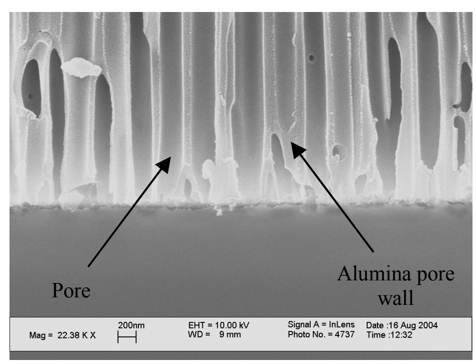

Figure 4. SEM micrograph of the cross-section of an Anodisc membrane filter. Pores with a diameter of ~ 200 nm can be observed.

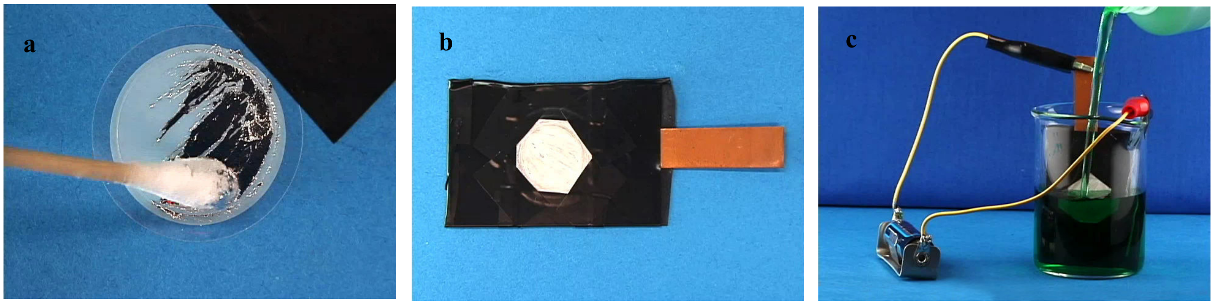

Figure 5. Steps in forming nickel nanowires. a) Ga-In eutectic is applied to the small-pore side of a membrane; b) the membrane is taped to a copper current collector (eutectic-coated side is face down) and the current collector is insulated from the electrolyte with electrical tape; and c) nickel-plating solution is added.

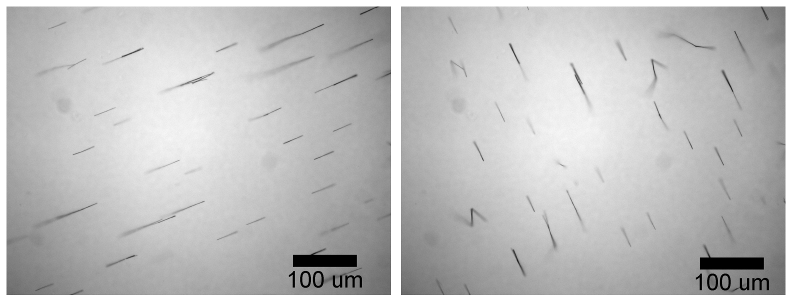

Figure 6. Manipulation of nanowires by application of a magnetic field. The applied magnetic field in (left) is rotated 90° from its orientation in (right).

The alumina membrane/In-Ga eutectic assembly is freed from the current collector by immersing it in acetone to remove the adhesive from the tape. The In-Ga eutectic is subsequently dissolved with nitric acid, and then the alumina membrane is dissolved in NaOH solution. After rinsing, the nanowires are suspended in ethylene glycol, a drop of which can be transferred to a microscope slide for optical observation with a 20X objective lens (Figure 6). The students are able to manipulate the nickel nanowires with a magnetic field. For visualization purposes, the nanowires in this lab are 200 nm in diameter, but much smaller diameter wires can be created with the same technique. The manipulation by magnetic field explored in the lab is related to an important new construction method for nanoscale devices.[23]

Assessment of the Nickel Nanowire Lab

The development of the lab was an iterative process, and assessment was used extensively throughout. This improvement with formative assessment can be seen in the data presented in Table 3. With the modifications and refinements that evolved out of the development stage, the laboratory can now be performed successfully by the vast majority of students on the first try. One exciting aspect of the high success rate of student performance in this lab is that it has opened up opportunities for student-initiated inquiry.

As mentioned above, this module has been utilized in several different types of courses. The module was adapted to address the needs of the instructor and the students in each of these courses. For example, in the intermediate-level chemistry course (Chemistry 311), the nickel nanowire synthesis was used as an example of electroplating techniques. In contrast, when the module was utilized in the graduate-level physics course on nanostructures, the focus of the module was on the fact that this synthesis technique is one that can be used to produce nanostructures, and the manipulation technique is one of several available for the assembly of nanoscale devices.

In addition to the traditional paper-based laboratory manuals, this laboratory has explored the use of a video-based format through the *Laboratory Manual for Nanoscale Science and Technology* (http://www.mrsec.wisc.edu/edetc/nanolab/index.html). In some cases, the video lab manual was used as a supplement to a traditional paper-based lab manual. In other cases the video lab manual replaced the printed lab manual entirely.

Table 3. Student success rates for the Nickel Nanowire Lab

Date	School	Course	Student Success Rate
Feb 2003	MSOE	Chemistry 200	2 of 12 pairs
April 2003	UW-Madison	MSE 361	5 of 9 pairs
May 2003	UW-Madison	Chemistry 311	23 of 24 pairs
Nov 2003	MSOE	Chemistry 200	14 of 14 pairs
Nov 2003	Beloit College	First Year Colloquium 100	8 of 13 students
Feb 2004	UW-Madison	MSE 361	10 of 10 pairs
April 2004	UW-Madison	Chemistry 311	31 of 33 pairs

Conclusion

The Interdisciplinary Education Group (IEG) of the Materials Research Science and Engineering Center on Nanostructured Materials and Interfaces at the University of Wisconsin-Madison, in conjunction with the Institute for Chemical Education (ICE), has created a number of student-centered, nanotechnology-focused educational kits. In addition, several video laboratory modules have been developed by IEG including the new module on the synthesis of nickel nanowires using a nanoporous template. Through these efforts, we have provided a number ways to teach and learn about nanoscale science and engineering concepts in and out of classrooms.

Acknowledgements

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References

1. Bentley, A.K., Farhoud, M., Ellis, A. B., Lisensky, G. C., Nickel, A-M. L., Crone, W. C., "Template Synthesis and Magnetic Manipulation of Nickel Nanowires," *J. Chem. Ed.* In press.
2. *Optical Transform Kit* by George C. Lisensky, Arthur B. Ellis, and Donald R. Neu. Institute for Chemical Education (ICE) Publication 90-002R.

3. *LED Color Strip Kit* developed by Jonathan Breitzer, S. Michael Condren, George C.Lisensky, Karen J. Nordell, Cynthia G. Widstrand, and Arthur B. Ellis Institute for Chemical Education (ICE) Publication 02-001.
4. Roco, M. C. "Converging Science and Technology at the Nanoscale: Opportunities for Education and Training" *Nature Biotechnology* **2003**, *21*, 1247-1249.
5. Nanotechnology Research and Development Act of 2003 (Referred to Senate Committee after being Received from House), H.R.766.RFS. http://thomas.loc.gov/ (accessed December 2004).
6. Interdisciplinary Education Group, University of Wisconsin Materials Research Science and Engineering Center (MRSEC) on Nanostructured Materials and Interfaces. http://mrsec.wisc.edu/nano (accessed December 2004).
7. ICE, the Institute for Chemical Education, University of Wisconsin-Madison Department of Chemistry, 1101 University Avenue, Madison, WI 53706-1396. http://ice.chem.wisc.edu/ (accessed December 2004).
8. Solid-State Model Kit, the Institute for Chemical Education, http://ice.chem.wisc.edu/catalogitems/ScienceKits.htm#SolidState (accessed December 2004).
9. DNA Optical Transform Kit, the Institute for Chemical Education, http://ice.chem.wisc.edu/catalogitems/ScienceKits.htm#DNA (accessed December 2004).
10. Amorphous Metal Demonstration Kit, the Institute for Chemical Education, http://ice.chem.wisc.edu/catalogitems/ScienceKits.htm#Amorphous (accessed December 2004).
11. Exploring the Nanoworld Kit, the Institute for Chemical Education, http://ice.chem.wisc.edu/catalogitems/ScienceKits.htm#Nanoworld (accessed December 2004).
12. Smestad, G. P.; Gratzel, M. "Demonstrating Electron Transfer and Nanotechnology: A Natural Dye-Sensitized Nanocrystalline Energy Converter" *J. Chem. Educ.* **1998**, *75*, 752-756.
13. Nanocrystalline Solar Cell Kit, the Institute for Chemical Education, http://ice.chem.wisc.edu/catalogitems/ScienceKits.htm#SolarCell (accessed December 2004).
14. Lisensky, G. C.; Kelly, T. F.; Neu, D. R.; Ellis, A. B. "The Optical Transform: Simulating Diffraction Experiments in Introductory Courses" *J. Chem. Educ.* **1991**, *68*, 91-96.
15. Ellis, A. B.; Geselbracht, M. J.; Johnson, B. J.; Lisensky, G. C.; Robinson, W. R. *Teaching General Chemistry: A Materials Science Companion*; American Chemical Society: Washington, DC, 1993.
16. Lisensky, G. C.; Penn, R.; Geselbracht, M. J.; Ellis, A. B. Periodic properties in a family of common semiconductors—experiments with light-emitting-diodes. *J. Chem. Educ.* **1992**, 69, 151-156.
17. Condren, S. M.; Lisensky, G. C.; Ellis, A. B.; Nordell, K. J.; Kuech, T. F.; Stockman, S. A. "LEDs: new lamps for old and a paradigm for ongoing curriculum modernization" *J. Chem. Educ*. **2001**, 78, 1033-1040.
18. Brown G. H.; Wolken J. J. *Liquid Crystals and Biological Systems*, Academic Press, NY, 1979, pp. 165-167.
19. Elser W.; Ennulat R. D. *Adv. Liq. Cryst.* **1976**, 2, 73.
20. McFarland A. D.; Haynes C. L.; Mirkin C. A.; Van Duyne R. P.; Godwin H. A. "Color My Nanoworld," *J. Chem. Educ.* **2004**, 81, 544A.
21. Zhao B.; Moore J. S.; Beebe D. J. "Surface-Directed Liquid Flow Inside Microchannels", *Science*, **2001,** 291:1023 -1026.
22. Beebe D. J.; Moore J. S.; Bauer J. M.; Yu Q.; Liu R.H.; Devadoos C.; Jo B.H. "Functional Hydrogel Structures for Autonomous Flow Control Inside Microfluidic Channels," *Nature*, **2000,** 404:588-590.
23. Bentley, A.K., Trethewey, J.S., Ellis, A.B., Crone, W.C., "Magnetic Manipulation of Copper-Tin Nanowires Capped with Nickel Ends," *Nano Letters*, 4(3) 487-490 (2004).

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