AC 2011-2797: LEAN SIX SIGMA NANOMANUFACTURING COURSE FOR ENGINEERING AND ENGINEERING TECHNOLOGY PROGRAMS

Michael G Mauk, Drexel University
Vladimir Genis, Drexel University (Tech.)

Dr. Vladimir GenisProfessor and Engineering Technology Program Director in the School of Technology and Professional Studies, Drexel University, has developed and taught graduate and undergraduate courses in physics, electronics, nanotechnology, biomedical engineering, nondestructive testing, and acoustics. His research interests include ultrasound wave propagation and scattering, ultrasound imaging, nondestructive testing, electronic instrumentation, piezoelectric transducers, and engineering education. Results of his research work were published in scientific journals and presented at the national and international conferences. Dr. Genis has five U.S. patents.

Dhruv Sakalley, Drexel University

Master's in Biomedical Engineering, Drexel University, Philadelphia, PA, 2010

Areas of concentration: Computational Neuroengineeing, Nanotechnology, Six Sigma

Software Developer- Tata Consultancy Services, India 2006-2008

B.E. Biomedical Engineering, Shri Govindram Sekseria Institute of Technology and Science, Indore, MP, India 2006

Holly Burnside, Drexel University

Lean Six Sigma Nanomanufacturing Course for Undergraduate Engineering Technology and Engineering Programs

Abstract. We have developed a laboratory- and project-based course to instruct Engineering and Engineering Technology students in *Lean Six Sigma* methodologies for nanomanufacturing. The experiments include synthesis and characterization of quantum dots and magnetic nickel nanowires, and fabrication and testing of organic LEDs and nanocrystalline solar cells. Additional experiments related to ferrofluids, soft lithography, nanocrystalline phosphors, and nanofilters are under development. The broad objective is to impart the knowledge and skills needed to translate laboratory discoveries in nanoscience to the production of commercial nanotechnology-based products using Lean Six Sigma principles and methodologies. Many aspects of the experiments are amenable to image capture and image processing with inexpensive CCD cameras (e.g., cell phones or webcams), as well as the quantification of image features to generate the sufficiently-large and diagnostic data sets needed for Six Sigma analysis. The image capture/analysis component provides students with exposure to machine vision for process control and automation, materials characterization, and quality assurance and inspection, as practiced in modern manufacturing. By themselves, Nanotechnology, Nanomanufacturing, Lean, Six Sigma, and machine vision (image capture /image processing and analysis) are important and timely subjects for engineering and engineering technology students. Their integration in a single laboratory course provides an effective and leveraging means for gaining exposure, insights, and practical experience in the subtle and pervasive issues and challenges of nanomanufacturing. The resulting synergism enhances the instruction of each subject and an appreciation of their broader relevance. The laboratory projects are in a modular format, and their materials, protocols, equipment, and time requirements are appropriate for semester- or quarter-based programs. The modules will be made available to other educational institutions.

Introduction. We are developing a laboratory- and project-based course in *Lean Six Sigma* nanomanufacturing. The target students are Engineering and Technology majors, and the primary application is for process and product development of nano-scale materials and devices. The broad objective is to teach the knowledge and skills needed to translate laboratory discoveries in nanoscience to the commercial production of nanotechnology-based products using Lean Six Sigma principles and methodologies. Lean Six Sigma is a systematic, rational approach to organize and manage an industrial or business enterprise in order to maximize efficiency and value added, minimize waste, and achieve world-class quality and customer-driven focus[1-11]. Elements of Lean Six Sigma originated with the classic works of Taylor, Ford, Gilbreth, Deming, Juran, Shewart, Ishikawa and others, but its primary source is the Toyota Production System (Ohno, Shingo) and Six Sigma (Smith). Lean Six Sigma integrates Just-in-Time, Kaizen (continuous improvement), Total Productive Maintenance, Poke-Yoke (mistake-proofing), set-up time reduction by Single-Minute Exchange of Die (SMED), single-unit flow, inventory reduction, Pull Production, balanced work flow, and Kanban (visual controls/visual factory), among others, with Six Sigma Quality Assurance. Lean Six Sigma offers a battery of procedural, operational, and analysis techniques including Quality Function Deployment (QFD), Value Stream mapping, Process Capability, Measurement Systems Analysis, Critical-to-Quality, Critical-to-Cost and Value-Added Analysis; Design of Experiments (DOE), Failure Modes and Effects Analysis (FMEA); and other tools that provide explicit and quantitative means to develop and sustain processes to make high-quality products. Lean Six Sigma has been adopted by many companies the world over, and is proving crucial to technology firms that need flexible, low-volume, high-product-mix (i.e., highly variegated or customized) production to serve their markets. Lean Six Sigma applied to nanotechnology is challenging due to its novelty, especially in relation to Lean Sigma developments in more traditional industries such as automobiles, aerospace, and electronics. The application of Lean Six Sigma to nanotechnology is part of a trend in extending the scope of Lean Six Sigma beyond the factory for use in hospitals[12-13], research and service laboratories[14-17], schools[18-24], governments, military, financial institutions, and other service providers.

To teach Lean Six Sigma, the laboratory facility is organized and operated on Lean Principles. To establish an educational laboratory for purposes of Lean pilot-scale production studies, we incorporate recent approaches used to implement Lean in machine/fabrication shops[25-27] and Lean analytical/clinical laboratories[13-17] in addition to mainstream Lean Six Sigma developed for manufacturing. Adaptation of Lean for services, hospitals, and financial institutions also provides useful examples. One advantage of Lean is that it will reduce costs for educational institutions in terms of materials, floor space, equipment utilization, and waste disposal[19,23,24]. As discussed below, we place great emphasis on sustainability to promote accessibility to as many educational institutions as possible.

There is a broad consensus that manufacturability, quality assurance, and scale-up will be crucial issues in realizing the immense potential of nanotechology in the coming decades[28-33]. This course will help produce students competent in using modern methods of manufacturing operations, experimental design, machine vision, and statistical analysis for production of nanomaterials and nanodevices. To this end, we incorporated extensive use of image capture and image processing for monitoring the manufacturing process and subsequent analysis, diagnostics, and statistical process control for quality assurance.

We have developed and validated laboratory experiments and projects that can simulate nanotechnology manufacturing processes. We adapted a number of nano experiments disseminated by the University of Wisconsin-Madison (mrsec.wisc.edu/Edetc) in order to study pilot-scale nanomanufacturing. The experiments include synthesis and characterization of quantum dots and magnetic nickel nanowires, and fabrication and testing of nanofilm organic LEDs and nanocrystalline solar cells. Additional experiments related to ferrofluids, soft lithography, nanocrystalline phosphors, and nanofilters are under development. Many aspects of the experiments are amenable to image capture with inexpensive CCD cameras (e.g., cell phone or webcams, and low-cost attachment cameras to microscopes) and subsequent image processing with MATLAB, or other widely-available packages and freeware (e.g., Image J). Image features can be extracted and quantified to generate the sufficiently-large data sets that are needed for statistical significance in Six Sigma analysis, and that are diagnostic of process and device performance. The image capture/analysis component provides students with exposure to machine vision for process control, automation, materials characterization, and quality assurance and inspection, as practiced in modern manufacturing. By themselves, Nanotechnology, Lean, Six Sigma, and machine vision (image capture /image processing and analysis) are important and timely subjects for engineering and engineering technology students. Their integration in a laboratory course provides an effective and leveraging means for gaining exposure, insights, and practical experience in the subtle and pervasive issues and challenges of nanomanufacturing. The resulting synergism enhances the instruction of each subject and an appreciation of their broader relevance.

The laboratory projects are in a modular format, and their materials, protocols, equipment, and time requirements are appropriate for semester- or quarter-based programs. The modules will be made available to other educational institutions. During the past school year, the laboratory course was offered at two Drexel campuses (West Philadelphia and Mt. Laurel, New Jersey). The laboratory modules were also used in workshops for high school and college science teachers. Our ultimate goal is to develop a suite of supported experiments and projects that can be used at a wide arrange of institutions to give students hands-on experience with nanotechnology, nanomanufacturing, machine vision, and Lean Six Sigma.

Approach and Aims. The main themes of our effort may be summarized as follows.

- The course introduces students to the science and engineering of nanoscale materials and devices. The Lean Six Sigma Nanomanufacturing course developed here would normally accompany lectures on nanotechnology, or follow an *Introduction to Nanotechnology* lecture and demonstration course. Students perform experiments exploring various aspects of nanotechnology involving quantum dots, nanoparticles, nanotubes, nanofibers, nano-thick films, and nanostructured surfaces, as described in more detail below. This hands-on-work serves to reinforce their classroom learning about nanotechnology and gives the students experience in instrumentation and measurements used for nanotechnology.
- We are aiming the course mainly at engineering and technology students who, upon entering tomorrow's workforce, will be tasked with process scale-up, prototyping, manufacturability, quality, reliability, process instrumentation, control and diagnostics. This group of students has been somewhat neglected in much of the nano education efforts of the past decade. It will do the United States no good to lead the world in nanotechnology discoveries if it does not recoup this public and private investment by manufacturing competitive, high-quality nano products for the global market.
- The experiments are adapted from the University of Wisconsin-Madison program that offers a set of highly-regarded and popular nanotechnology experiments for secondary school and undergraduate education under funding from the National Science Foundation (DMR Grant 0520527). We want to utilize and leverage previous efforts supported by NSF as much as possible. The advantage of using these experiments are numerous. They are well-documented and well supported with protocols, bills of materials, and demonstration videos. They are safe, do not use highly toxic substances nor create serious waste disposal problems. They do not require expensive or specialized equipment. They can be completed in a several-hour time frame, and can be done in a typical high school or undergraduate chemistry lab setting. The experiments cover a wide range of nanotechnology areas including biomedical applications, renewable energy, and information technology. In all cases, the total cost of the readily available materials (for 10-12 students) is around $100 per lab, and in many cases kits are available

for purchase. Although the laboratory exercises do not require sophisticated analytical instrumentation, the experiments can be supplemented with a wide range of analytical instrumentation (e.g., electron microscopy, dynamic light scattering, fluorescence spectroscopy) for schools that want to incorporate these more advanced techniques.

- We are adapting the experiments described above to serve as case studies of pilot-scale manufacturing processes. The students will treat the experimental protocol as the scientific basis for making a commercially-viable process for producing the nano-scale material or device. Essentially, we task the students as if a researcher is "handing off" the process to the students who then play the role of development engineers for implementation of a viable production process. The students will develop the process with Lean Six Sigma, and more specifically, work in the DMAIC framework (progressing through the stages of *Define*, *Measure*, *Analyze*, *Improve*, and *Control* in process development). The basic goal is robust process control to minimize product variation, maximize yields, and reduce wasted materials and effort.
- In order to provide meaningful case studies for Six Sigma, relatively large data sets must be generated. For instance, process capability measurements, hypothesis testing, design of experiments, gage and reproducibility studies, regression analysis, process control charts, and other Lean Six Sigma techniques require about 30 or more data points to achieve statistical significance. For instructional laboratories, this would be overly burdensome if not impractical using common instrumental methods such as analytical balances, electrical testing and probing, spectrophotometry, and other techniques typically available in school laboratories. We address this dilemma by using machine vision for data acquisition. There are many low-cost suitable CCD cameras (web cams, cell phone cameras, inexpensive microscope cameras) that can be used to image the nanomaterials and devices, or secondary effects closely related to nano phenomena. For materials analysis, instruments include optical and fluorescence microscopes, scanning electron microscopes, and atomic force microscopes. We note that for schools where these are not available, electronic image files can be provided showing typical results. These will be provided on a website developed in the proposed program. For nanodevices such as solar cells and light-emitting diodes, inexpensive optical instruments (inspection microscope, Webcam, laser pointer, CCD projector) will suffice for laboratory projects. Simple image processing software can extract and quantify features in the images, rapidly generating large data sets for Six Sigma analysis. Image processing functions available in MATLAB are well-suited for this analysis.
- The use of image capture and analysis not only provides sufficiently large data sets for Six Sigma analysis, but offers an opportunity to teach machine vision for process diagnostics and quality assurance. Machine vision is easily integrated into both the development phase and production phase of manufacturing[34] for both *in situ* and *in line* testing. The techniques can also include interferometry, spectroscopy, surface mapping, and ellipsometry.
- Six Sigma methods that can be readily adapted for this work include analysis of variance (ANOVA), attribute sampling plans, autocorrelation, cross-tabulation methods, fractional and full factorial designs, main effects plots, mixture designs, Pareto analysis, and Measurement Systems Evaluation. These methods can use the raw data (in spread sheet form) provided by the image processing software. Six Sigma statistical analyses can be performed with EXCEL®, Minitab®, or MATLAB®. We also used Design of

Experiments (DOE) software from Stat-Ease (Minneapolis, MN) provided with a free 45-day trial for educational institutions.

- Six Sigma methods provide an interesting collateral opportunity for assessment of student learning, since data collected on the process will also indicate the quality of the students work. For instance Gage Reproducibility and Repeatability[35] methods allow one to separate and assess variation due to the process, instrument, and operator (student), and to compare skills of different operators. Incidents of defects, process variation as indicated by control charts, and other Six Sigma methods provide metrics quantifying students competency and progress. Also, the National Association of Job Shops and Small Manufacturers (NAJS) provides a Lean Assessment for benchmarking and assessment in over 50 categories that can be applied to laboratory and pilot-line production[27].
- Lean manufacturing is taught in several ways. First, the laboratory is organized on Lean principles such as the 5 S's (sort, straighten, shine, standardize, and sustain)[20], cellular design to save floor space and minimize workpiece flow between work stations, and visual controls such as Kanban, a scheduling system, using visual cues, that signals what to produce, when to produce it, and how much to produce.
- It might be argued that a school is not a factory, and implementation of Lean in a laboratory setting is not practical nor a realistic representation of actual practice. On the contrary, Lean is being adapted in many venues outside of factories including hospitals, clinics, doctors' offices, schools, providers of various financial services, machine shops, clinical laboratories, analytical and test labs.[/]
- Lean Simulation will be used to accelerate and optimize the development of the experiments. These simulations can also be used by the students to supplement laboratory work. MEHTA[36] and others[37] introduced hands-on classroom simulations to demonstrate key metrics for process control using Lean and Six Sigma. Discrete event simulation models for Lean Six Sigma manufacturing are described in detail in a book by EL-HAIK and AL-AOMAR[38].
- Lean operation of a laboratory as developed, with its emphasis on waste reduction and doing more with less, provides inherent sustainability for educational institutions. This program is a step towards wider implementation of Lean in other laboratories and school operations. There are comprehensive information and tools for implementing Lean available from the Lean Advancement Inst. of MIT (www.lean.mit.edu), American Soc. For Quality- Learning Inst., (www.asq.org/lean-manufacturing), the Lean Enterprise Inst. (www.lean.org), and the American Soc. for Manufacturing Engineers- Lean Manufacturing Enterprise Tech group (www.sme.org/lean), Lean Education Enterprises, Inc. (www.leaneducation.com). We will utilize these resources extensively in the course of the proposed work.

Motivation. The increasing importance and widening scope of nanotechnology is well recognized. Many educational institutions have developed nanotechnology courses and programs in the last decade. We believe the course and programs described in this proposal fill an as-yet unmet need, and will foster and accelerate nanotechnology adaptation and commercialization efforts by US industry. Our view is that much prior activity in nanotechnology education focuses on introducing students to the fundamentals and basic science of nanotechnology, and instruction in analytical skills is concentrated on tasks needed at the R&D stages of nanotechnology. Comparatively little effort has been devoted to developing students with the know-how for translating benchscale laboratory processes to the factory floor. There is a wealth of important discoveries in nanotechnology coming out of US Universities and companies. Many of these will stagnate or even fail unless they can be made commercially. The crucial issues relate to product and process development to commercialize these discoveries. Further, it is clear that many nanotechnologies will not be suitable for the scale-up approaches used in traditional manufacturing and the chemical process industries. Nanotechnology will fill niche markets and will require companies adopt low-volume, high-mix production runs with a high-mix of product types. Indeed, in many cases the line separating the industrial laboratory and the production line will be blurred. Some nanotechnologies may even require customization. These are precisely the issues that are addressed in Lean Manufacturing.

Table 1: Lean Six Sigma Tools in the DMAIC Framework

Stage	Define	Measure	Analyze	Improve	Control
Six Sigma Tools	• Work breakdown • Flow Charts • Quality Function Deployment • Benchmarking • Kano Analysis	• Data Collection • SIPOC • Sampling • Process capability	• Hypothesis testing • Correlation • ANOVA • Full factorial	• Screening experiments • Steepest ascent • Factorial analysis • Taguchi methods	• SPC • Control charts
Lean Tools	• Value stream mapping	• Lead time • Takt time • Inventory Level	• Work Analyses • Flow Analysis • Scheduling	• SMED • JIT-Kanban • Line balance	• Error proofing • Visual controls • Standard Work • Kaizen

Since 2001, the federal government has invested over \$8.3 billion in nanotechnology research and development with similar levels of funding in the private sector. As the development of many nanotechnology-based inventions is completed, the lack of access to scale-up and initial production facilities has become apparent. One consequence is delayed benefit to the marketplace and delayed return on investment for stakeholders.

Laboratory Experiments. Here we very briefly review the work of our NSF CCLI project, pertaining to four of the six modules we developed, and where nano experiments were adapted to simulate nanomanufacturing and where image processing was utilized to collect data amenable to six sigma analysis.

EXPERIMENT 1: SYNTHESIS OF QUANTUM DOTS. A relatively safe, easy, and fast synthesis of CdSe quantum dot nanocrystals as colloidal suspensions, which can be readily analyzed by absorption and emission spectroscopy, is described in Boatman *et al*.[39] This method avoids the use of highly toxic and/or pyrophoric compounds prescribed in other protocols for synthesizing quantum dots and does not require inert atmosphere working conditions, such as a glove box with controlled ambient. The CdSe QD nanocrystals are made from CdO and elemental Se using a kinetic growth method where QD size depends on reaction time. Students prepare a precursor stock solution of elemental Se in 1-octadecene and trioctylphosphine, and a Cd precursor solution of CdO in oleic acid (a surfactant) and octadecene. The Cd precursor solution is heated to 225°C in a round bottom flask clamped in a heating mantle, whereupon Se precursor solution is added to initiate the reaction that precipitates colloidal CdSe nanocrystals. Samples of 1-ml in volume are pipetted from the reaction mixture at frequent time intervals (~30 seconds) and quickly cooled to quench the reaction. About nine to ten samples are collected, and the reaction time is recorded for each aliquot. Each of the samples will exhibit a characteristic color (Figure 1), indicating a variable product range according to reaction time.

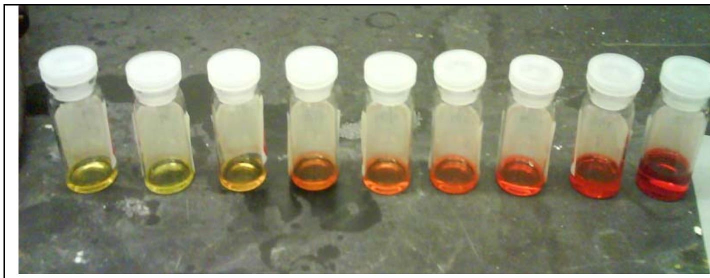


Figure 1: Suspensions of quantum dots showing different colors due to shift in absorption peak with quantum dot size.

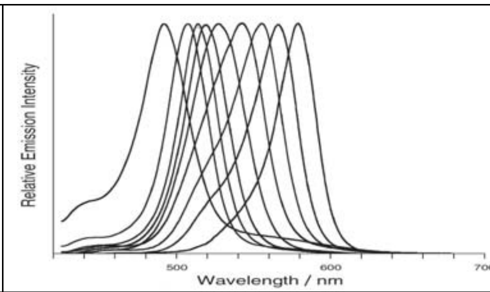


Figure 2: Emission intensity for different quantum dot suspensions.

For quantitative analysis, the emission and absorption spectra of the QD colloidal suspensions can be measured in a spectral photometer/fluorometer (Figure 2). A figure of merit for QDs is a narrow fluorescence emission spectra, and the FWHM (full-width at half maximum) can be regarded as a quality metric. This measurement is amenable to a Gage R&R study to assess operator and instrument repeatability and reproducibility. As an exercise in regression analysis, the students correlate absorption wavelength maxima with emission wavelength maxima. Another correlation is emission wavelength peak vs. reaction time, with the objective of showing control of a nanoscale property through a process parameter (reaction time), as well as inherent product variation (spectral emission width) which can be optimized with various response, DOE, and Taguchi methods.

Another laboratory experiment on the synthesis of silver nanoparticles is described in Solomon *et al.*[40]. Students make a colloidal suspension of 12-nm silver nanoparticles by reducing aqueous silver nitrate with excess sodium borohydride solution. Although metal nanoparticles do not fluoresce like semiconductor quantum dots, they do exhibit a characteristic optical absorption spectrum, which leads to a change in the color of the colloidal solution Transmission electron microscope (TEM) images can be used to measure the size of the silver nanoparticles, which exhibit an approximate normal distribution[40] with mean of 12 nm and standard deviation of 3.4 nm. Electron microscopy can be utilized by the students, who work from TEM images which are examined visually using a scale marker included in the image, or by using image processing software. This data can be used to analyze normally-distributed process variables.

EXPERIMENT 2: TEMPLATE SYNTHESIS OF NICKEL NANOWIRES. Nanowires (cylindrical structures with nanometer-sized cross-sections) have applications in electronic, optical, magnetic, and mechanical devices , see LAW et al.[41], Spanier[42] including solar cells and nanoelectronic power sources[43]. In this lab, students synthesize and characterize nickel nanowires which exhibit useful magnetic properties. A straightforward application of such nanowire suspensions as magneto-optical switches has also been described by Bentley et al.[44,45]. The nanowires are made by a simple electrochemical template synthesis method, see Rahman *et al.*[46] (Figures 3 and 4). Briefly, nanowires are fabricated by electrochemical deposition of nickel in nano-sized channels of an alumina porous membrane (Whatman Anodisc™) with 200 nm pores and 60 micrometers thickness. One side of the alumina membrane is painted with a gallium-indium eutectic to form a cathode contact. The nickel plating process is done in a 50-ml beaker with a commercial nickel plating solution, a nickel wire anode, and a battery or dc power supply as the voltage source. The electroplating area can be defined by partially masking the substrate with tape. After the deposition step, the membrane template is then dissolved in 6M NaOH (sodium hydroxide) solution at room temperature in five-ten minutes to yield a suspension of free nickel nanowires. The nanowires can be washed and resuspended in various solvents including water, ethylene glycol, and glycerol. The nanowire synthesis part of the lab takes about 90 minutes.

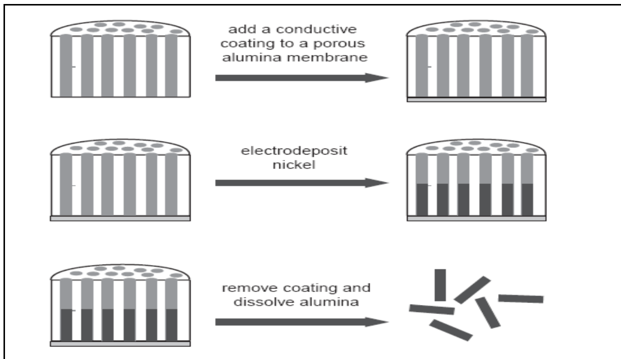


Figure 3: Schematic description of template synthesis technique for nickel nanowires. From [Bentley *et al.*[45]].

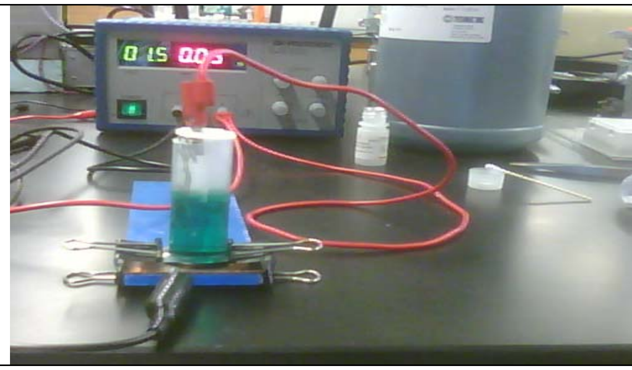


Figure 4: Electroplating setup for template synthesis of nickel nanowires.

The nanowires are examined by drying out a drop of nanowire suspension on a glass microscope slide. The wires are visible under a low power optical microscrope, and their images are captured with a CCD camera mounted on the microscope for subsequent analysis (Figure 5).

Figure 5: Nickel nanowires on a glass microscope slide.

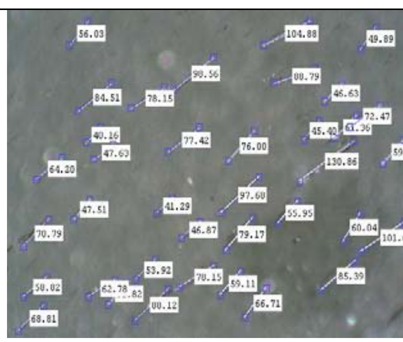


Figure 6: Assignment of lengths to nanowires using MATLAB®-based image processing.

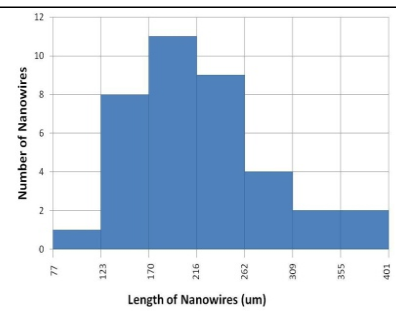


Figure 7: Distribution of nanowire lengths for sample shown in Figure 5.

The analysis of the nanowires is facilitated by using an external magnet to align the nanowires during the drying step. Scanning electron microscope images provide greater detail and resolution. To find the distribution of nanowire lengths, a simple image processing step is performed in MATLAB® using the Image toolbox functions to assign a pixel length to each wire (Figure 6). Figure 7 shows a distribution of wire lengths for a given sample. Measured lengths comprise single wires, two wires connected end-to-end, and chains of three and longer. In this lab, we regard the primary output process variables as the distribution of nanowire lengths (mean length, range, standard deviation) as controlled by the process input variable plating time. Examples of optical microscope and SEM images of the nanowires, and the relationship between nanowire length and deposition time, as well as additional characterization by X-ray diffraction of the wires still embedded in the template membrane, indicating a preferential orientation of the nanowire growth direction, have also been described by Bentley *et al*[45,46]. This experiments lends itself to a host of Six Sigma process parameter optimization methods. A more detailed study of nickel electroplating variables (plating solution composition, plating temperature, pH) is feasible[46]. This laboratory project can be extended to demonstrate applications of the nanowires as magneto-optical switches[46]. In this case, switch device performance (modulation depth, switching speed) could be used for process improvement.

EXPERIMENT 3: ORGANIC LIGHT-EMITTING DIODES. Solid-state electroluminescent devices are an area of considerable technical and commercial interest for applications to lighting, consumer products, and displays. Light-emitting diodes (LEDs) can be made from thin films of organic materials that are 60- to 100-molecules thick, see MANESS *et al.*[47]Gao and BARD[48], RUDMAN and RUBNER[49], SEVIAN *et al.*[50]. The operation of the LED is based on charge injection under voltage bias, charge transport by electron hopping and ion migration, and decay of an energetic ruthenium complex that emits red-orange light at 630 nm wavelength. This laboratory project has several aspects of interest to the course objectives :

- it utilizes nano-scale structures
- it involves several sequential fabrication steps each with several controlling process variables
- its end product is a device with well-known consumer applications and straightforward figures of merit quantitating its performance
- the devices have some stability or reliability issues characteristic of many emerging nanotechnologies.

Accordingly, nano LEDs (and solar cells as discussed below) are good case studies for illustrating various aspects of nanomanufacturing and the application of Lean Six Sigma. Following the methods described by SEVIAN *et al.* [2004], and supplemented by the material presented on the website http://www.mrsec.wisc.edu/Edetc/ nanolab/oLED/, an organic LED can be made by spin-coating an aqueous ruthenium complex ($[Ru(bpy)_3]Cl_2$) mixed with a polymer (polyethylene glycol) on glass slides. Figures 8 and 9 show a few of the processing steps.

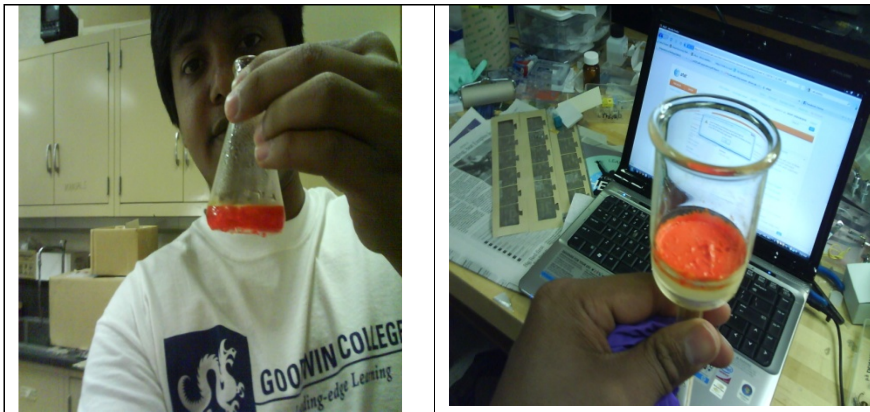


Figure 8: Synthesis of organic luminescent compounds.

Figure 9: Application of an organic LED material to slide.

The glass slides are pre-coated with a conducting, transparent indium tin oxide that serves as a cathodic electrode. Small droplets of indium-gallium (liquid) eutectic are dabbed onto the Ru-coated substrate to make an anodic contact (Figure 10). The deposition areas are defined by partial masking with tape. The spin coater used to deposit the Ru-containing solution is fashioned from a small electric cooling fan (such as used in desktop computers) run by a low-voltage dc power supply. LEDs are tested by probing with a dc power supply (Figure 11).

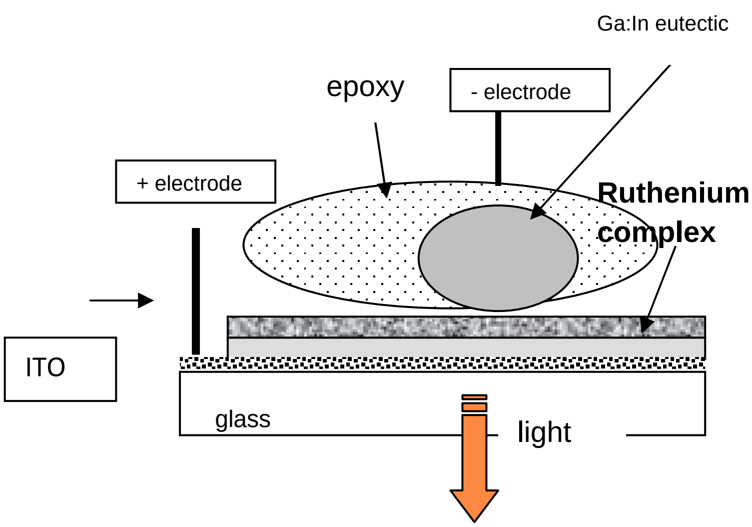


Figure 10: Cross section schematic of organic LED structure.

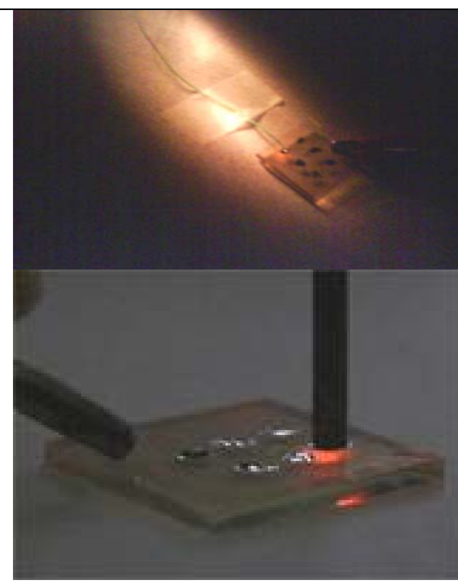
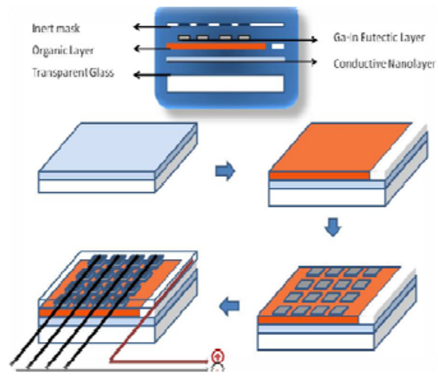
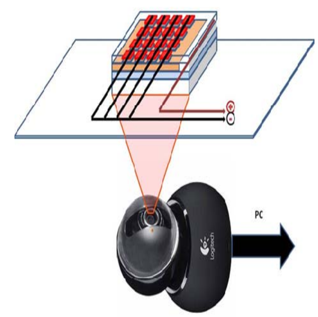


Figure 11: Testing of organic LEDs.

		
Figure 12: Printed circuit board patterns to test organic LED arrays.	**Figure 13:** making a test array of organic LEDs to study lifetimes.	**Figure 14:** Monitoring LED output with a webcam.

The LEDs are generally short-lived (lifetime measured in minutes or hours), primarily due to the effects of humidity/oxidation. Thus, lifetime studies offer another opportunity for statistical analysis. Some relevant process variables include the concentration of the Ruthenium solution and the PEG (polyethylene glycol), the spin coater speed (rpm), the amount of solution applied in the spinning process, and the sheet conductivity of the indium tin oxide layer. Students use Design of Experiments (DOE) to optimize the process. This laboratory experiment was modified by making the LEDs on a custom printed circuit board[51] in order to produce an array of individually-addressable LEDs (Figure 12). This modification was done for generating a sufficient number of LED replicates (~16 to 20) for statistical analysis. The LED array output can be monitored with a CCD camera, and maps of pixel intensities can follow the variation in array elements and the reduction in light output over time.

To illustrate, we fabricated a 4 x 5 array of organic LEDs for testing purposes. The array elements (individual LEDs) were simulataneously energized with 5 volts by a common busbar and common ground on the test circuit board. The emissions of the LEDs are continually viewed with a 960 x 720 pixel CCD camera (Logitech Orbit AF webcam) fixed at about 5 cm over the array (Figures 13 and 14). The array and camera are placed inside a box to block ambient room light. Frames from the CCD camera video can be analyzed to discern: (1) the variation, i.e., non-uniformity, of emission from the individual LEDs; and (2) degradation of emission from each LED element over time (Figures 15-17). The LED array output can be analyzed on the basis of a simple assumption of LED element behavior over time as

$$L_{i,j}(t) = L_{i,j}\left(t = t_{start}\right) \cdot e^{-t/\tau_{i,j}} \quad [1]$$

where $L_{i,j}(t_{start})$ is the initial intensity of the LED element designated by row i, column j of the array at some arbitrary time t_{start}, t is the LED operating time measured from t_{start}, and $\tau_{i,j}$ is the to-be-determined decay time of the LED element. Using this scheme, the areal uniformity of LED arrays and variations in their lifetime (decay time) or reliability can be assessed. Large LED arrays or several arrays will generate sufficient data for Six Sigma analysis.

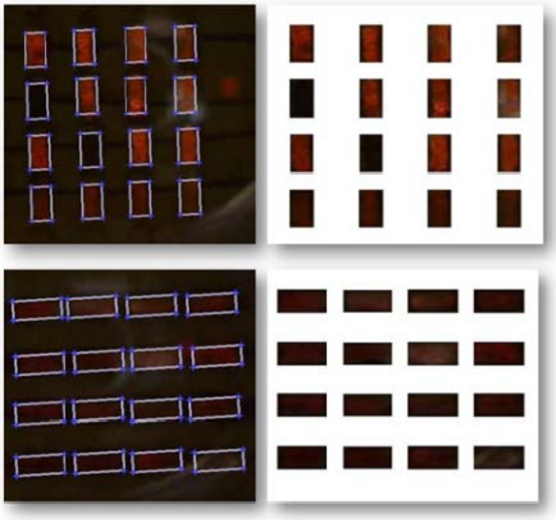


Figure 15: Images of LED array captured by webcam.

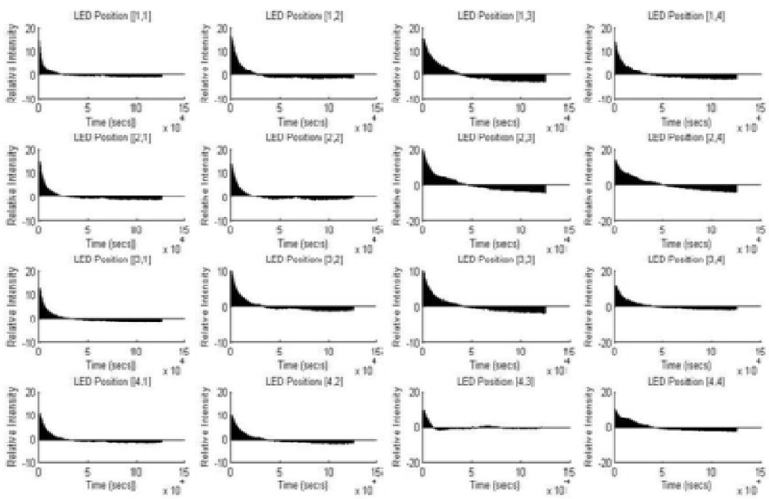


Figure 16: Light emission from a 4 x 5 organic LED array.

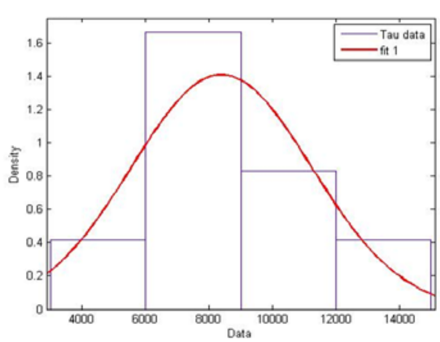


Figure 17: Distribution of LED lifetimes.

EXPERIMENT 4: TiO_2 NANOCRYSTALLINE SOLAR CELLS. Photovoltaic solar cells based on nanomaterials are an important application of nanotechnology to renewable energy[52] see Figure 18. As a specific example, organic dye-sensitized TiO_2 nanocrystalline solar cells demonstrate the utilization photosynthesis-like electrochemical processes to solar generation of electrical power, and are the focus of pure and applied research in nanomaterials and devices[53,54]. Commercialization of similar kinds of solar cells is under way[55]. This particular type of cell has been well developed for educational purposes[56-58].

In this experiment, and as with the previous organic LED laboratory, glass slides with a transparent conducting indium tin oxide coating are purchased or prepared by vacuum deposition. The slides are coated with a colloidal solution of TiO_2 nanoparticles, and then sintered in an oven at about 400°C. Next, the TiO_2 coating is stained with natural dyes, such as chlorophyl derived from plant material, by soaking the TiO_2-coated substrate in an aqueous dye solution. A second conductive oxide-coated glass slide is coated with carbon using a pencil or graphite rod. The two glass slides are then clamped together to form a solar cell. The solar cell area can be defined by masking the substrates with opaque tape (Figure 19).

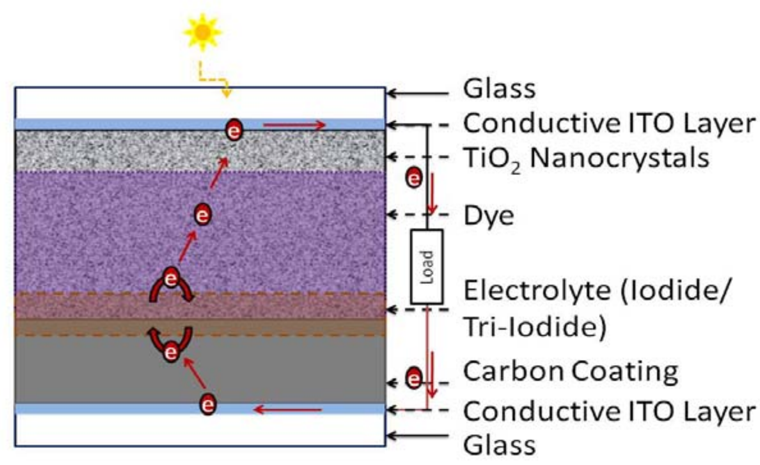


Figure 18: Cross section of nano solar cell structure.

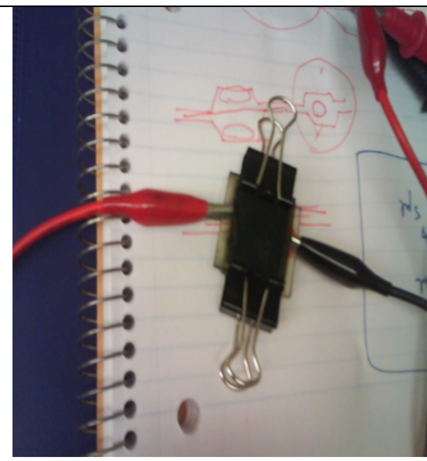
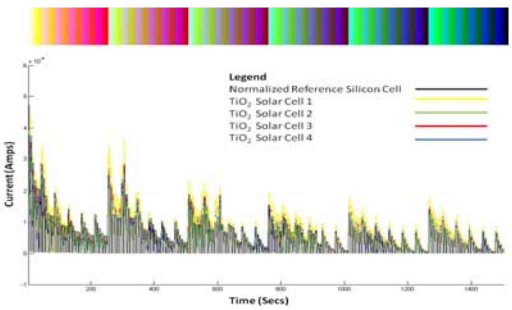


Figure 19: Nano solar cell sample ready for testing.

A solar cell provides many easily-measured performance metrics, several of which are diagnostic for specific device faults and material quality. These include open-circuit voltage, *I-V* curve shape or fill factor, photogenerated current, diode ideality factor, conversion efficiency, series resistance, shunt resistance, and most revealing spectral response or quantum efficiency, i.e., the solar cell photocurrent as a function of incident light wavelength. Most of these parameters can be measured with a multimeter or curve-tracer instrument. We used an educational robot trainer to scan red, green, and blue laser beams (using low-cost laser pointers) to map the current of nanosolar cells (Figure 20). A monochrometer, white light source with color filters, or different color LEDs can be used to generate approximate monochromatic light for spectral response measurements. We adapted a common CCD projector, as commonly used in lecture rooms, to produce multicolor illumination of solar cells. This provides low-cost, easily-accessible methods for schools to do spectral response of nanodevices (Figure 21). As with the organic LEDs of the experiment described previously, these solar cells also have stability issues that can be quantified and optimized using Six Sigma. As a Six-Sigma case study, this experiment provides a large parameter space to explore process optimization. These variables include the sheet resistivity of the conductive oxide layers coating the glass slides, the thickness of the TiO_2 layer, the concentration and soak time for the dye agent, and the thickness of the carbon layer.

<table>
<tr><td></td><td>
</td></tr>
<tr><td>Figure 20: Laser scanning of nanocrystalline solar cell with laser pointer mounted programmable robot arm.</td><td>Figure 21: Spectral response data of nanosolar cell collected from a CCD projector.</td></tr>
</table>

Sustainability. This course, in both its objectives and practice (laboratories, experiments, and materials), fulfills the aims of a sustainable curriculum on several levels. First, nanotechnology enables innovation in the areas of renewable energy, toxic waste remediation, more efficient use of materials, substitution for scarce materials, and affordable healthcare. Smith and Granqvist[59] provide a survey of sustainable Green nanotechnologies for electric lighting and daylighting (luminaries), heat and electricity via solar energy, cooling devices, air sensing and cleaning, thermal insulation, and electrical storage, using nanotechnology similar to the subjects of our proposed laboratory projects. Second, Lean approaches are predicated on waste reduction—"doing more with less." Such a Lean laboratory could serve as a model for other laboratories in colleges and secondary schools.

Conclusion. In this program, we have demonstrated the key steps in developing a hands-on undergraduate nanomanufacturing laboratory that teaches Lean Six Sigma and utilizes machine vision for data collection. Four modules have been 'student-tested' as labs for courses, and can be disseminated to other schools. There is considerable scope for developing more modules to broaden the coverage of nanotechnology topics and the depth and extent of of Lean Six Sigma methods applied. Image processing, from a variety of camera types and analytical instruments can be productively incorporated into our laboratories, and prove very useful in generating sufficiently-large (statistically significant) data for Lean Six Sigma analysis.

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