

## **Undergraduate Educational Components for Nanoscale Issues in Manufacturing**

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### **Abstract**

Engineering designers during the next fifty years will work intimately with tools and applications made feasible by nanotechnology. Therefore, engineering undergraduates must be able to integrate concepts and principles of nanotechnology into their knowledge bases as soon as possible. The project “Nanoscale Issues in Manufacturing” will transfer knowledge gained through nanoscale research into undergraduate engineering curricula at Texas A&M University through four components. The level of detail and sophistication of the material taught will increase as the scientific understanding of the students increases through their undergraduate career. For first-year engineering students, a company involved in nanotechnology research will develop and offer a case study on nanoscale applications. This approach builds on the successful case study program that has been offered for five years. For sophomore engineering students taking ENGR 213 Principals of Materials Engineering the nanomanufacturing faculty team has developed two modules. The first focuses on two fundamental ideas in nanotechnology: scaling and granularity. The second module focuses on two approaches to manufacturing macroscale systems using nanoscale technologies: top-down and bottom-up. The third component consists of two one-hour modules that will be integrated into MEEN 360 Materials and Manufacturing Selection in Design, a junior-level course offered by the Mechanical Engineering department but available to all students who have taken the prerequisites. The module expands on the top-down and bottom-up approaches to nanoscale manufacturing and provides students with hands-on laboratory experience. The fourth component will be a new elective course that will be available to all engineering and science students who have completed the prerequisite courses. The elective course, which will be taught by three faculty members, is comprised of three elements: methods and techniques for nanostructure fabrication using nanolithography, fabrication of bulk materials through nanoparticle consolidation, and design and fabrication of active micro-devices using nanocomponents. The four components will be described.

### **Introduction**

Nanotechnology, construction of structures at the nanoscale and application of nanoscale structures and processes to create innovative solutions, holds singular promise to revolutionize science, engineering and technology, and in the process to transform our society. Its enormous potential to transform our world demands both that science and engineering graduates understand the technology and that society in general understands concepts and potential applications of nanotechnology. In addition, the excitement and dramatic potential of nanoscience and nanotechnology can be used as powerful tools to stimulate interest in science and technology. Given the potential, Texas A&M University (TAMU) has initiated a project with support from

the Nanotechnology in Undergraduate Education (NUE) program at the National Science Foundation (NSF) to address issues in manufacturing at the nanoscale. The project has two overarching goals: 1) to give students the knowledge and understanding they will need to work with nanotechnology in the future, and 2) to recruit and retain students in science and engineering by using nanotechnology as an example of the excitement and importance of scientific and engineering innovations.

Engineering designers are using and will increasingly continue to use options made available by nanotechnology. Therefore, engineering undergraduates must be able to integrate concepts and principles of nanotechnology into their knowledge bases as soon as possible. The proposed project will introduce elements of nanotechnology throughout the undergraduate curriculum. The level of detail and sophistication of the material taught will increase as the scientific understanding of the students increases through their undergraduate career. In addition to content changes, curriculum changes will use pedagogical innovations advocated by the NSF-funded Foundation Coalition (FC), one of eight engineering education coalitions: active/cooperative learning, technology-enabled learning and student teams. Further, the principal investigators will use the lessons about processes of curricular change gained from the FC experiences. As a result, many engineering students will become better acquainted with the possibilities offered by nanotechnology, and some engineering students will gain an in-depth understanding of nanoscale manufacturing processes.

The envisioned curricular change has four components corresponding to the four years of undergraduate engineering curricula. At each level, concepts related to manufacturing at the nanoscale will be introduced at the appropriate technical level and with special attention to the pedagogical approaches promoted by the FC. Further, changes in the first two years are facilitated by the curricular structure which TAMU implemented as a FC partner. The first component is an industry case study on nanoscale manufacturing into the first-year engineering course sequence. Case studies are already an integral part of the first-year engineering sequence and a case study on nanoscale manufacturing will promote awareness and interest in engineering and nanotechnology, in particular. For the sophomore year, the project team has prepared two one-hour modules on nanotechnology for a required introduction to materials engineering course. The third component consists of two one-hour modules that have been integrated into a required, junior-level course offered by the Mechanical Engineering department but available to all students who have taken the prerequisites. The fourth component is a new course that will be developed under this program and will be offered as an elective under the interdisciplinary Mechanics and Materials (MEMA) program. Each of the four components is described in greater detail in the following sections of the paper.

The purpose is to describe the modifications that have been made to sections of required courses in the freshman, sophomore, and junior years as well as the new senior elective course. Faculty members who are interested in one or more of the changes can download materials from the web site or contact one or more of the authors for additional information. Efforts to assess effects of the modifications or the new course are continuing and will be provided in future publications generated by the project. Suggested improvements to the efforts to integrate nanotechnology throughout four-year engineering curricula are welcome.

## First-year Case Study

The project will work with one or more companies to offer case studies on nanotechnology to first-year engineering students in the required sequence ENGR 111/112 Foundations of Engineering I/II. Case studies demonstrate “real world” engineering to currently enrolled engineering students [1]. Companies usually send a team of engineers that range from 2 to 8 members to model teamwork and diversity. A background reading packet [and homework] for the students often precedes visits. The industry team typically presents a 15-20 minute overview of a problem encountered in their company or industry. Students then break into assigned teams, generate possible solutions to the problem, and then present their team solutions to the class. The engineering team then leads a discussion that reveals the actual solution and reviews the major contributing factors.

## Required Sophomore Materials Course

ENGR 213 Principles of Materials Engineering is a sophomore-level course required for the following majors: aerospace engineering, chemical engineering, civil engineering, mechanical engineering, and petroleum engineering. The goal of the NUE project was to generate two modules for the course, each corresponding to one hour of lecture material. Given the breadth of nanotechnology material and the very limited time, the project team faced a difficult challenge of deciding which topics to include. One of the guiding principles was to focus on topics that were likely to continue to be relevant and applicable for several years in this rapidly changing area. After much discussion, the team decided to focus on why behavior of materials may be so different at the nanoscale and approaches to manufacturing at the nanoscale. In the first module two basic ideas underlie differences at the nanoscale: scaling and granularity. The second module features two different approaches to nanoscale manufacturing: top-down (use existing techniques to fabricate smaller and smaller features) and bottom-up (start with atoms or molecules and assemble larger units). Both topics mesh well with the overall conception of an introductory course on materials and both provide students with a foundation for future study of nanotechnology.

**Scaling:** The core idea of scaling is that a large number of material properties as well as other characteristics depend on ratio of lengths. For example, heat loss for warm-blooded living organisms is roughly proportional to surface area that is related to the square of a length (e.g., for a cube the surface area is proportional to the square of the edge length and for a sphere the surface area is proportional to the square of the radius). On the other hand, the weight of the organism and amount of heat energy that the warm-blooded organism might generate are roughly proportional to the volume that is related to the cube of the length. As the length scale of an organism decreases, its capability to generate heat decreases more rapidly (being dependent on the cube of the length scale) than its heat loss (being dependent on the square of the length scale). Therefore, smaller organisms face a greater challenge to keep warm; consequently, they must eat more often than larger organisms. Small organisms, such as insects, address the challenge by being cold-blooded. This example is used to introduce the concept of scaling and the dependence of characteristics on length.

A more quantitative example is the Hall-Petch (H-P) Relation that for metals relates yield stress, the inherent strength of the metal, and grain size:

$$\sigma_y = \sigma_o + k_y d^{-\frac{1}{2}}$$

where  $\sigma_y$  is yield stress,  $\sigma_o$  is a friction stress or can be called materials' inherent strength,  $k_y$  is a constant, and  $d$  is grain size. As a result a fine-grained material is harder and stronger than one that is coarse grained, since the former has a greater total grain boundary area to impede dislocation motion. If H-P equation were valid down to grain sizes in the nanometer range with the same value of  $k_y$  found at conventional grain sizes, remarkable increases in strength would be realized. Reducing  $d$  from 10 micron to 10 nm would increase the strength by a factor of about 30 for most common metals. However, the relationship usually breaks down for  $d < 20$  nm because the deformation mechanisms such as dislocation formation or stress induced phase transformation that take place at large scales do not operate at nanoscales. Change in the major deformation mechanisms occurs mainly because of the significant increase in the ratio of interfacial area/volume of the building blocks (grains) at nanoscales. At nanoscales, other mechanisms such as grain boundary sliding or deformation twinning become more effective and they do not follow the H-P relationship.

As another example, the melting point of a gold sphere decreases as the radius decreases. Therefore, smaller gold spheres melt at a lower temperature than large gold spheres. This is being used in a project to create small gold structures. Two sizes of gold spheres (5 nm radius and 3 nm radius) are mixed uniformly. Then, the temperature of the mixtures is raised to a temperature at which the smaller spheres will sinter while the larger spheres will remain separate. Various patterns can be created using this process. A presentation on scaling will be available at the project home page, <http://fc1.tamu.edu/resources/nano>. The presentation incorporates in-class team activities to provide a more active/cooperative learning environment for the students.

**Granularity:** Nanotechnology presents a major change in conceptualization because it destroys the concept of the continuum. We are all familiar with macroscale events that we interact with and model down to the microscale. When undergraduates learn concepts such as stress (force/area) and temperature (a representation of the thermal state of a large number of particles) they are not prepared to adjust for the failing of these concepts on the nanoscale. Stress calculations work because we assume that between any two points in an area we can define an infinite number of connecting points that make the area continuous. Applying this concept to a carbon nanotube brings us up short; how does one define the area of a single bond? Current literature shows a broad range of prediction and measure of carbon nanotube modulus—each researcher may have her or his own definition of the tube's cross sectional area. In a similar fashion, can we describe the 'temperature' of a small number of atoms? Finally, models that describe the flow of a fluid in the microscale cannot apply to nanochannel flow device that makes each molecule of the fluid separate from the others.

**Approaches to manufacturing:** The goal of the second module in ENGR 213 is to help students understand the two basic approaches to manufacturing at the nanoscale: top-down and bottom-up. The top-down approach uses lithographic processes for printing a pattern on a surface and

using either deposition to lay down material or etching to remove material using the pattern. The top-down approach is so named because it starts with large bulk material and creates smaller features by miniaturizing using existing larger-scale techniques. The second approach is bottom-up that starts with atoms or molecules and assembles larger units. The top-down approach builds on silicon fabrication techniques that have been refined for chip manufacturing during the last fifty years while the bottom-up approach uses synthesis concepts from chemistry. A presentation on approaches to manufacturing at the nanoscale will be available at the project home page, <http://fc1.tamu.edu/resources/nano>.

The modules were used in the ENGR 213 when it was taught in the fall semester of the 2003-04 academic year. Approximately 85 students worked with the two modules.

### **Required Mechanical Engineering Course on Materials and Design**

MEEN 360 Materials and Manufacturing Selection in Design is a required course for mechanical engineering majors. It is taught both semesters, offered to about 200 students each year, and includes a laboratory component. Like ENGR 213 the project introduced two one-hour modules on nanoscale manufacturing: one on micro and nanoscale lithography and another on nanoparticle processes for bulk materials. In addition, the project introduced one laboratory experiment that would be performed by all students.

The two modules in MEEN 360 have three goals:

- Introduce nanoscale manufacturing as an emerging field that might affect your career
- Visualize nanoscale issues in manufacturing
- Provide background information for the new senior elective course MEMA 489 Nanoscale Issues in Manufacturing to be offered in the spring semester of the 2003-04 academic year.

The module on micro and nanoscale lithography begins by providing background information on development of lithographic processes. The history highlights the work on Alois Senefelder who in 1789 decided that that his plays were losing money because it costs too much to print them. In response, he wanted to devise a way to print them at home for just pennies a page [2]! He tried carving mirror images into soft copper sheet with a steel tool and failed. So he practiced writing in reverse on limestone. He used limestone as a substrate (stone-lithos), used waxy ink as a mask/resist, etched the mask with acid, coat the upper (planarized) surface with ink, and carefully(!) placed paper on the inked plane. With an understanding of the historical development of lithography, the module introduced modern lithographic processes.

The module describes the components of a modern lithographer's toolkit:

- Oxidation
- Masking
- Implantation
- Etching
- Metallization
- Lift-Off

Up to this point in the presentation, the scale of the lithography processes has not been mentioned. Then, discussion moves to diffraction limit as a primary barrier for nanoscale lithography. The diffraction limit is described and alternatives to overcoming the diffraction limit are described:

- UV 365 nm
- Deep UV 248 nm
- Extreme UV 10-20 nm
- X rays 0.01-1 nm
- E beam “40 nm”

The nanoscale lithographic module provides background for the laboratory experiment.

The laboratory experiment utilizes a Cobilt UV mask aligner specifically purchased for educational purposes. The goal of the experiment is to allow students to fabricate lithographic structures and to employ them for basic electronic measurements. The minimum feature sizes of the student-created patterns will be in the single digit  $\mu\text{m}$  regime. UV lithography allows for efficient feedback, an important aspect in teaching students a conceptually new technique. Thus, while these patterns are strictly not nanotechnology, we think that they pedagogically introduce the students to the lithographic concept and thus allow them to easily generalize their understanding to similar nanolithography techniques.

The module on nanoparticles in bulk materials begins by motivating consideration of nanoparticles. It illustrates tensile strength of bulk, fiber and whisker SiC and the intriguing possibilities for very high tensile strength for nanoparticles:

- Bulk 250 MPa
- Fiber 3400 MPa
- Whisker 14,000 MPa
- Nanoparticle ???

Next, the module illustrates current applications of nanoparticles in bulk materials.

- The film industry has used nanoparticles of silver halide in film since 1930.
- Inkjet printers produce nanoparticle ink (1 drop is about 1 picoliter or a radius of about 6 microns assuming a spherical drop).
- Nanoparticle layers are used to make stable photo quality inkjet paper.
- Nanoparticles are used as catalysis in chemistry and as a fuel in space applications because of their large surface energies.

Then, the two major approaches to nanoparticle production are overviewed.

- In the top-down approach, the starting point is bulk materials either naturally occurring (minerals, clay) or synthesized by current processes (metals, ceramics). Then, the particles in the bulk material are reduced to the nanoscale.
- In the bottom-up approach, chemistry is used to synthesize nanoparticles.

Each of the two approaches is explored in greater detail. As an example of the top-down approach, production of nanoparticles from limestone is examined. Limestone is obtained by drilling and blasting a limestone deposit. About 1600 tons per hour can be obtained. Mined limestone is processed in a primary crusher to obtain particles between 50 and 150 cm in diameter. Processing in a secondary crusher yields particles between 19 and 60 cm in diameter.

Crushed materials are ground using either ball or rolling mills. Powders are classified according to the size of their particles.

- Very coarse: 1000  $\mu\text{m}$
- Coarse: 355 to 1000  $\mu\text{m}$
- Moderate Coarse: 180-355  $\mu\text{m}$
- Fine: 125-180  $\mu\text{m}$
- Very Fine: 90-125  $\mu\text{m}$

Nanoparticles must be 1000 times smaller than a fine powder. And the question is how they can be manufactured.

Several approaches are available. Grinding is one approach and the energy cost of grinding down a bulk material is usually less than that required to collect pure substances and synthesize the same material. Jet mills grind the material against itself. Gas drives the particles and cold nitrogen gas can chill the particles. Ceramic materials are by nature brittle and the ductile to brittle transition temperature of most metals is above liquid nitrogen temperatures. Therefore, cold nitrogen gas is usually sufficient to produce brittle particles that can easily be ground. The system can classify particles and let only acceptable particles exit the system. Another approach is thermal processing using either atomized melt or vaporizing. High temperatures create problems with contamination since reactions with the surrounding materials are more likely. Vaporizers subject the material to intense energy that can be generated from several different sources.

- **Laser pulse:** A laser pulse provides an energy level that can vaporize any material. Each 10 ns pulse frees  $10^{14}$  to  $10^{15}$  atoms from the target surface. This approach is used to create carbon nanotubes.
- **Thermal evaporation:** In thermal evaporation the material is vaporized and allowed to precipitate as nanoscale particles. Electrochemistry might control reactions and novel structures, e.g., dendrimers, might be possible.
- **Spark erosion:** Using a high frequency, high voltage spark to erode bulk materials into nanoparticles, in the presence of a suitable gaseous or a liquid dielectric medium. The method is flexible in terms of the selection of materials that can be produced as nanoparticles, including semiconductors, metals, intermetallics and alloys, nitrides, and carbides. The spark-erosion process, for example, can produce particles of refractory metals, which are very difficult to accomplish with conventional thermal processes. This method offers significant advantages in processing oxide-free, reactive materials in inert atmospheres. At the same time, the erosion environment can be tailored to accomplish a desired reaction product, such as nitrides, oxides, etc.
- **Electroexplosion of wires:** Metastable nanosize metal powders can be manufactured by the electroexplosion of metal wire (EEW process). The EEW process is done in an inert gas atmosphere. A continuous metal filament is fed into the inert gas reactor and a portion of the wire is superheated with a very large electrical pulse during a brief (microseconds) duration to cause the wire to explode into atom clusters. Extraordinarily rapid quenching occurs from peak temperatures of 15,000+ Kelvin. Any metal, which can be obtained in the form of ductile wire, can be used as a starting material.

Grinding or thermal processes can be used to produce nanoparticles, but the next question is how macroscale materials can be produced from the nanoparticles.

One approach is to add nanoparticles to other bulk materials. One concern is how to keep ‘nano’ character during processing. Different approaches include consolidation of metals in which case processing temperatures must stay below recrystallization temperatures and mixing with polymers in which nanoparticles are blended with a resin or melted polymer. After mixing nanoparticles with a polymer the resulting bulk material may be formed through extrusion or injection molding.

## **Senior Elective Course**

The senior elective course, MEMA 489 Nanoscale Issues in Manufacturing, is open to both science and engineering students who have the necessary prerequisites. The 489 number is used because it is an experimental course. If student and faculty interest are sufficient for the course to be offered on a regular basis, it will be assigned a different course number. It has both lecture and laboratory components. Since it covers a broad sweep of topics on nanoscale manufacturing, three members of the project team, Ibrahim Karaman (Mechanical Engineering), Winfried Teizer (Physics) and Terry Creasy (Mechanical Engineering), are team teaching the course. Each faculty member will develop lecture materials and laboratory experiments for about one-third of the course.

### *Theme 1: Methods and Techniques for Nanostructure Fabrication*

This theme will cover various methods for direct fabrication of nanostructures. Planned topics include: electron beam lithography, self-assembly, functional nanomolecules, patterned thin-film devices and neurons. Initially this class will focus on techniques for fabricating metallic nanostructures, and later will discuss molecular self-assembly techniques. This module will emphasize hands-on experience in a series of laboratory experiments.

While bottom-up nanomanufacturing techniques will be discussed, Theme 1 focuses more heavily on top-down nanomanufacturing technology. After introducing lithographic techniques to students, they will have lab time in which they can acquire hands-on experience with optical and electron beam lithography in general use facilities that are available on campus. Dr. Teizer will present the introductory lectures, and the lab time will be interactively supervised by Teizer and an assistant who has prior experience with the involved techniques.

The theme will start with an introduction to optical lithography. Didactically, it is important that students learn lithographic techniques in a setting where they can get rather direct feedback about their progress. The comparably straightforward evaluation of optical lithography devices under a microscope allows for this. After students have mastered this technique they will go through the entire process of electron beam lithography. Starting with designing their own devices, they will then write and develop them on a silicon wafer. After several iterations, they will continue with a successful device and evaporate a thin metallic film into it (using a so called lift-off technique). They will then evaluate the resulting pattern in detail under a Scanning Electron Microscope and take a picture of their device.

At the completion of this module students will assemble the results into a framed plaque, which includes:

- the computer based design of the students' nanodevice,
- the original silicon wafer on which the students designed and manufactured the nanodevice , and
- the SEM picture, which the students have taken, to show the details of the nanodevice.

*Theme 2: Fabrication of Bulk Materials through Nanoparticle Consolidation*

Theme 2 will provide an understanding of the principles governing a range of preparative techniques and their capabilities for the production of nanoparticles and bulk nanocrystalline materials. Upon successful completion of this theme, students should be able to apply the physical principles underlying microstructural control on the nanometer scale to design an appropriate synthesis route for nanomaterials. The following topics will be discussed in Theme 2:

- A. Production techniques for nanoparticles and nanomaterials
  - Nanoparticle synthesis in Gas, Liquid, and Solid State Phases
  - Bulk nanocrystalline materials: Consolidation of nanoparticles; Vapor deposition; Controlled crystallization of glasses; Mechanical alloying and mechanical milling.
- B. Mechanical properties of nanomaterials and microstructure-property relationships
  - Principles, properties and their measurement in traditional and nanostructured materials.
  - Metallic nanomaterials.
  - Ceramic nanomaterials.
- C. Microstructural stability in nanomaterials

Students will learn methods of fabricating nanoparticles in the section on “nanoparticle synthesis”. The idea is that the building blocks of nanomaterials (metal, ceramic or polymer) are nanoparticles and properties of nanomaterials can be engineered by controlling the sizes of the building blocks and their assembly. The advantages and disadvantages of different nanoparticle fabrication methods will be discussed to select methods that are more suitable for different material groups. Then, methods for the fabrication of bulk materials from nanoparticles, such as consolidation and controlled crystallization of glasses, will be introduced. Theories behind the methods will be discussed together with the advantages and disadvantages of the methods. Examples of properties of the resulting bulk materials fabricated using different methods will be given in the light of recent literature. For each topic, homework problems will be developed.

For the laboratory part of the course, three different nanoparticle consolidation techniques will be demonstrated: hot compaction, equal channel angular extrusion and hot isostatic pressing. The resulting materials will be compared by evaluating their density, mechanical properties and particle-particle bonding characteristics using the nanoindenter. The initial nanoparticles (copper) will be acquired from Argonide Corporation, Aveka Inc. and DOE-Ames Laboratory. These organizations use different methods to synthesize nanoparticles that will allow students to compare the resulting powders. Experimental parameters of consolidation techniques will be discussed and students will be asked to decide on some of the processing parameters such as consolidation temperature, pressure, speed, etc. Samples of the consolidates will be given to the groups made out of four students for evaluation of density of the consolidates, mechanical

properties and interparticle bonding strength using a nanoindenter available at The College of Engineering.

### *Theme 3: Design and Assembly of Devices and Multifunctional Materials Using Nanoscale Components*

This theme will focus on assembling nanostructures into larger scale structures and devices. We will address understanding of nanoscale processes and scale-up of processing/fabrication methods for devices incorporating nanosystems, design concepts for manufacturing, simulation of the manufacturing methods at the nanoscale, and evaluation of the economic and environmental implications of manufacturing at the nanoscale. The goal of this theme is to have students explain scaling issues that make incorporation of nanostructures/nanosystems into larger scale structures a challenge. From this goal, the following objectives are constructed:

- Expand the student's awareness of design methodology to include materials design to the nanoscale as a significant component of the process.
- Provide tools for student assessment of processing effects of nanoparticle filled polymers: impact on rheology during processing and impact on mechanical properties after processing.
- Lead the students in a design exercise where they design microdevices based on functional properties provided by nanoparticles.
- Produce a laboratory sequence where students fabricate recyclable molds with features down to 60- $\mu\text{m}$  and mold multifunctional polymer matrix devices with nanoparticle activation.

Current and pending research projects that form the basis of this theme are aimed at creating multifunctional microdevices through filament extrusion or spinning processes. These processes will create micromachines with variable properties, e.g. Young's modulus, through the filament cross section as well as active components using particulates embedded in the polymer. One obstacle to wider use of emerging nanotechnologies is the difficulty of scaling 'bottom up' methods to high production rates [3]. However, processes that continuously produce polymer profile, filamentary and film structures can operate across the scale range from macro- to micro- and recently [4] to nanoscale production. Once running under steady conditions, extrusion processing is an efficient, continuous means of producing a great volume of material. This complex process can be applied to making microscale devices at a classroom laboratory level by mimicking the method through low-pressure injection molding of room temperature curing gels and elastomers. The molding process can combine multiple polymers into a single structure [5, 6]. Each polymer can contribute specific functions, e.g. flexibility or strength, as well as synergistic improvements such as diffusion control [5]. Magnetic nanoparticles provide activation of the microdevice and embedded conductors can produce a simple sensing mechanism.

## **Conclusions**

Nanoscale Issues in Manufacturing is an attempt to incorporate material on nanotechnology throughout four-year engineering curricula at Texas A&M University. In the first three years of most engineering curricula, there are so many required courses that new material can either be added by removing a required course or introducing modules into existing required courses. The

project team has chosen the second course to reduce potential resistance against introducing new topics and reach the largest number of students quickly. Work is continuing to assess the degree to which students comprehend the new material that is being introduced.

## **Acknowledgements**

The authors gratefully acknowledge the support of the Division of Engineering Education and Centers of the National Science Foundation under grant number EEC-0304049. Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation.

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