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Undergraduate Engineering Ceramics Laboratory Development

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An Engineering Ceramics Laboratory for undergraduate education was established in Fall 1991. An undergraduate class in ceramics was converted from a lecture-only mode to a lecture-laboratory mode. In addition to purchasing new equipment, student design projects also contributed to the design and construction of laboratory equipment. A set of 12 laboratory exercises in engineering ceramics, designed with the undergraduate student in mind, have been developed. The role the Engineering Ceramics Laboratory will play in the overall curriculum development aspects of the department, and future developmental work, are also discussed.

AUTHOR QUESTIONNAIRE

1. New training tools or laboratory concepts
   This paper provides a method for development of a laboratory component to an existing lecture-only course. The paper describes the actual approach that was taken to bring about the development of the laboratory—both equipment-wise and also laboratory exerices.
2. New equipment
   The equipment described are all standard commercial items that can be purchased.
3. Level of students
   The anticipated level of students is undergraduate—junior and senior.
4. Aspects of contribution that are new.
   This paper describes, very briefly, new laboratory exercises that can be implemented in a ceramics course within a materials science and engineering curriculum. It also provides guidelines for developing the written communications skills of undergraduates.
5. Incorporation of material presented in engineering teaching.
   It is anticipated that readers of this paper can do at least two things. One, they can use this approach to convert their own lecture-only classes into lecture-laboratory classes. Two, they can utilize the laboratory exercises used within their own curriculum.
6. Texts of documentation accompanying the presented materials.
   None accompanying this paper. A complete laboratory manual has been developed, and is available separately.
7. Have the concepts been presented in the classroom?
   Yes, this course has been implemented for the past three years. Undergraduate students in the materials engineering curriculum have a much stronger background in ceramics processing and characterization.
8. Other comments.
   None.

INTRODUCTION

SAN JOSE State University is one of California State University's 22 campuses, and is located in the heart of the technologically vibrant Silicon Valley. Within California it is known for being the state's oldest institution of higher learning, having started out as California Normal School in 1857. It became California State College in 1934, and San Jose State University in 1972.

The university has eight colleges, one of them being the College of Engineering which has eight engineering departments. The Materials Engineering (MatE) Department has the distinction of being the only one of its kind within the California State University system.

The student body within the university and the college is extremely diverse ethnically, with a large proportion of both minorities and women. The MatE Department shares this feature with the university. Approximately 80% of the College of Engineering graduates stay and work within the community.

The MatE Department, though relatively small within the College of Engineering, is reputed for its extremely high standards at both the undergraduate and graduate levels. Approximately 25% of the BS graduates continue onto graduate school. The department has also had the distinction of being awarded the university's Undergraduate and/or Graduate Research Award for the past five consecutive years. The department has six full-time faculty, and 15 participating faculty from other departments within the College of Engineering and from local industry.

MOTIVATION FOR LABORATORY DEVELOPMENT

It has always been College of Engineering policy to encourage laboratory development for instructional purposes. The emphasis has been on providing students with a 'hands-on' education so that they will be adequately prepared to contribute when they start working in industry. Laboratories are considered crucial to the process of relating theory to practice and physical reality. Within the MatE Department, 50% of the required undergraduate classes are taught in a lecture-laboratory mode to ensure that the theoretical principles covered during the lectures are reinforced by practical experiences in the laboratory. The overall purpose of all laboratory exercises or experiments is to demonstrate the relationship between structure, property and processing of materials.

A secondary objective achieved via student-oriented laboratories is the development of written communications skills. Students are required to write laboratory reports that are an integral part of the grading process. In this manner, the technical writing skills are improved while providing the technical training.

ENGINEERING CERAMICS WITHIN THE CURRICULUM

In Fall 1989 the MatE Department targeted engineering ceramics as an area for development, in view of the increasing technological significance of this field. It was decided that the first step in this development would be to convert the existing undergraduate class in ceramics, Course Number MatE 185A—Nonmetallic Solids I: Ceramics, to a lecture-laboratory mode. This would provide the nucleus for continuous improvement, and development, of the ceramics laboratory within the department. The term 'engineering ceramics' was intentionally chosen so as to avoid confusion with ceramics for other applications, such as architectural and aesthetic applications.

It was recognized at the outset that engineering ceramics should really be an integral part of the overall curriculum. The objective was therefore to integrate engineering ceramics into the existing curriculum, and to utilize existing laboratory facilities as much as possible. Ceramics-related laboratory experiments would be developed within the framework of MatE 185A, Ceramics, first. After successful implementation and debugging, these would be handed over to other courses while new laboratory experiments are developed. The instructional philosophy was to demonstrate the application of fundamental principles of materials science and engineering to ceramics, while emphasizing areas where ceramics have similarities with, or differences from, metals, polymers and composites.

Beginning in Fall 1991 the department established an Engineering Ceramics Laboratory. Part of the funds for the equipment purchased for this laboratory was provided by a NSF-ILI grant. Establishment of this laboratory enabled conversion of the lecture-only undergraduate class in ceramics to a lecture-laboratory mode.

LABORATORY DEVELOPMENT IN ENGINEERING CERAMICS

The objectives of this effort were as follows:

1. Develop a laboratory in engineering ceramics that would be complementary to existing laboratory facilities.
2. Develop laboratory exercises for students so that they can perform a number of experiments within the constraints of the academic time requirements, while still gaining meaningful knowledge.

Achievement of each of these objectives is discussed separately below.

Engineering Ceramics Laboratory facilities

Development of this laboratory presented several challenges. In order to obtain the maximum benefit for the lowest capital outlay, it was decided to use existing facilities to the maximum extent possible. The department's laboratories, with major equipment holdings, are listed in Table 1. This table does not contain the equipment located in the Engineering Ceramics Laboratory.

Establishing the Engineering Ceramics Laboratory invariably involved acquisition of new equipment, and the approach taken here was to acquire equipment that would provide the department with unique capabilities. Due to limited funds available to a state-funded school, it was also decided to pursue sources of external funding.

An application was made to the National Science Foundation (NSF), under its Instrumentation and Laboratory Improvement (ILI) program, with the author as the Principal Investigator, for funds to acquire new equipment so that the Engineering Ceramics Laboratory could be established. The approval of this grant application was crucial to the ability of the department to implement its decision. Approximately $32,000 was awarded for the purchase of a pycnometer, particle size analyzer, BET (Brunauer, Emmet and Teller) surface area analyzer, and a computer for data acquisition and analysis. The NSF-ILI grant was also instrumental in obtaining additional funding from the College of Engineering and the university for purchasing not only the equipment proposed in the grant, but other equipment as well.

Table 2 lists the equipment that has been procured for the Engineering Ceramics Laboratory. This table identifies equipment that was purchased with matching funds from NSF, and equipment that was purchased solely with internal funds. It should be stressed here that had the NSF-ILI grant not

Table 1. Materials Engineering Department laboratories and major equipment holdings

Materials Characterization Laboratory	**Corrosion Laboratory**
X-ray Diffractometer Scanning Electron Microscope with EDS Transmission Electron Microscope - Phillips Model 301 and Siemens Model 101 Debye-Scherrer Camera Laue Back-Reflection Camera X-ray Fluorescence Spectrometer Electron Probe Micro-Analyzer Dark Room	EG&G Potentiostat/Galvanostat AC Impedance Unit Singleton Corrosion Test Chamber Potentiodynamic Scanner **Composites Laboratory** Laminating Press Injection Molder, 60 Ton Capability Dielectrometer Curing Oven Refrigerators and Freezers
Mechanical Properties Laboratory MTS Universal Testing Machine Riehle Creep Tester Taber Abrasor Sebastian Five Desk Top Mechanical Tester Tensil Kut Machine Iosipescue Shear Testing Fixture	**Metallography Laboratory** Microtome Precision Cutoff Wheel Cutting, Grinding and Polishing Equipment Optical Microscopes - Zeiss ultraphot, Nikon Epiphot Microhardness Tester Stereomicroscope Tripod Polisher Gatan Dimple-Grinder
MatE 25 Laboratory Hardness Testers - Rockwell and Microhardness Box Furnaces, air atmosphere, 1200°C Charpy Impact Tester Arc and Gas Welding Equipment RF Vacuum Induction Furnace Riehle Universal Testing Machine	**Thin Films Laboratory** Ion Beam Test Facility DC Magnetron Sputter RF Sputterer Metal-Organic Chemical Vapor Deposition System Vacuum Evaporator

been approved, the probability of obtaining internal funding for purchasing other equipment would have been relatively remote. The fact that the NSF-ILI grant required matching funds, and the fact that at least 50% of the funds were guaranteed to be provided by NSF, provided significant leverage in obtaining internal funds.

Once the nucleus of the laboratory was established, it became easier to continue the process of improvement of the laboratory. Again, due to limited funds, innovative approaches had to be taken. One such innovative approach was to utilize student talent in the design and construction of necessary laboratory equipment. This approach was especially suitable for smaller laboratory items that could be constructed in-house. Students earn 'individual project' or 'design project' credits for their design and construction effort. Thus, by providing the cost of materials, the department has been able to enhance its Engineering Ceramics Laboratory, with infrastructure-type equipment and fixtures. This approach not only reduced the capital investment necessary, but also provided students with valuable design experience. A list of equipment constructed and built by students within the department, under this approach, is provided in Table 3.

In order for the laboratory and the equipment therein to be effective teaching tools, laboratory exercises for students needed to be developed. The process followed in developing the laboratory exercises is described in the next section.

Engineering Ceramics Laboratory experiments

The laboratory experiments were designed to demonstrate to students the uniqueness and excitement of engineering ceramics. As mentioned before, wherever possible, similarities and differences with other engineering materials were demonstrated. It was also necessary to design each experiment such that it could be completed in approximately 2–2.5 hr, since the time allocated for each laboratory period is a maximum of 3 hr.

A list of laboratory experiments developed, including the objective for each, is shown in Table 4. Students were provided with handout materials,

Table 2. Engineering Ceramics Laboratory equipment

Name of Equipment	Purchase Date (Academic Year)
BET Surface Area Analyzer*	1990/91
Particle Size Analyzer*	1990/91
Multipycnometer*	1989/90
Dilatometer, max 1600 C, controlled atmosphere	1989/90
Cahn Microbalance	1989/90
Tap-Pak Volumeter	1990/91
Ball Mill	1990/91
Sieves	1989/90
Tube Furnace	1980/81
Ovens**	1990/91
Chemical and Electronic Balances	1990/91
Tape-Caster	1990/91
Viscometer	1990/91
Differential Scanning Calorimeter	1980/81
Controlled Atmosphere Box Furnace, 1600°C	1992/93

Note: * indicates equipment purchased with funds provided by NSF-ILI grant. ** indicates donation from local industry. All other equipment purchased with university funds.

Table 3. Engineering Ceramics Laboratory equipment designed by students and constructed in-house

1. Four point bend fixture for determining stress rupture modulus and Weibull statistics
2. Three point bend fixture for determining stress rupture modulus and Weibull statistics
3. Pellet press
4. Argon glove box for sample preparation
5. Controlled atmosphere test chamber: 0 to 100°C and 0 to 85% Relative Humidity
6. Equipment for measuring contact angle of liquid metals on ceramic substrates
7. Tape caster
8. Gas header for mixing reaction gases
9. Shear test fixture for shear testing

in the form of a laboratory manual, for each experiment.

The manual for each experiment was written with the specific purpose of enabling it to stand alone independently, and is typically 15–20 pages long. Each manual contains the following:

1. An *introduction* or *background* that explains the theory involved in the experiment.
2. A well-defined *objective* for the particular experiment.
3. A list of the *equipment and materials* needed for the experiment.

Table 4. Listing of laboratory experiments developed

Title	Objective	Equipment
Crystal Structures	Application of principles of crystallography to ceramic structures	Crystallographic models of five different ceramic structures
Porosity Measurement	Measurement of density of engineering materials, and identification of different types of porosity	Pycnometer
Alumina Pellet Pressing	Effect of pressing load on green density	MTS Universal Testing Machine, Pycnometer
Particle Size Analysis	Determination of particle size distribution, including mean, median and mode	Particle Size Analyzer, Sieves
Surface Area Measurement	Measurement of surface area of different ceramic powders	BET Surface Area Analyzer
Tap Density Measurement	Measurement of Tap Density of different ceramic powders	Tap Densitometer
Surface Tension Measurement	Measurement of surface tension of different solutions, demonstrating the effect of solutes on surface tension	Glass ware
3-point and 4-point bend testing	Measurement of modulus of rupture, using 3-point and 4-point methods	MTS Universal Tester, 3-point bend fixture, 4-point bend fixture
Moisture Content and Water of Crystallization	Determination of moisture content and water of crystallization in ceramic powders	Furnace, electronic balance
Sol-gel synthesis of ceramic compounds	Preparation of glasses using acid and basic routes	Glass ware
Synthesis of 123 Superconductors	Synthesis, by solid-state reaction, of 123 superconducting compounds	Furnace
Sintering of Pressed Alumina Pellets	Measurement of percentage shrinkage and sintered density	Dilatometer, pycnometer

4. A *procedures* or *methodology* section that provides detailed instruction on how to prepare samples, connect equipment, operate equipment and run the experiment.
5. A *data collection* section, normally in the form of a table, identifying the data that are to be collected.
6. An *analysis of results* section that shows students how to reduce the raw data collected into processed results.
7. A list of *references* for further reading, especially for the interested or motivated student.
8. A *list of questions* that students are required to answer, so that the instructor can evaluate how much the students have learned.
9. A glossary that explains the key technical terms encountered in the course of the laboratory experiment.

STUDENT REPORTING REQUIREMENTS

Both the College of Engineering and the MatE Department place major emphasis on development of written and oral communications skills by its students. Writing of laboratory reports is therefore a normally expected part of the curriculum.

In view of the large number of laboratory exercises, it was decided that the written laboratory report part be kept to a minimum. Students are therefore required to submit laboratory reports containing the following:

1. A statement of the *objective* of the experiment.
2. *Results* of the experiment, in a neatly tabulated form.
3. *Discussion* of the results. This is viewed as the most important part of the laboratory report, where the students discuss the validity of their findings.
4. A *Conclusion* statement, which should be directly relatable to the objective statement.
5. Answers to the questions contained in the laboratory manual.

Field trips

As a normal part of the laboratory curriculum in this class, students go on two field trips to local ceramics industries. These field trips provide another valuable link between the classroom theory, laboratory hands-on experience and reality in industry. The ceramics industry in the San Francisco bay area has been more than willing to accommodate requests for field trips. Students are required to formulate and submit a flowchart of the production process that they visit. The flowchart is evaluated for the ability of the students to identify unit operations and quality control standards for each unit operation.

FUTURE PLANS

Plans for future development of the Engineering Ceramics Laboratory are based on the department's philosophy of continuous improvement. The Engineering Ceramics Laboratory will serve as the focal point for development of ceramics laboratory experiments. These will be tested within the framework of MatE 185A—Ceramics, and then incorporated into other courses, while new laboratory experiments continue to be developed.

The experiment on density measurement of a variety of materials, using the pycnometer, has already been 'transferred' to MatE 25—Introduction to Materials Engineering, effective Spring 1992. This course is taken by all students majoring in engineering. It is expected that by conducting this experiment they will not only have a better appreciation for density measurements, but will also realize that density is not always a number that can be looked up in a reference book, but rather is a property that can be processing dependent.

While the existing equipment holdings within the department are quite extensive, there is still a critical need for several other pieces of equipment. The process of utilizing undergraduate and graduate design projects to design and construct various pieces of equipment that would be necessary for the Engineering Ceramics Laboratory will continue.

The laboratory manual currently in use needs to be revised and expanded. A section on data analysis and data acquisition techniques is necessary. It is expected that this section will be written within the next academic year.

Curriculum development within the area of engineering ceramics is also planned. Classes focusing on electronic ceramics, bioceramics and structural ceramics are currently being developed for future offering.

Another long-term goal the department has is to incorporate ceramics-related concepts into courses taught by other departments. Notable among these is the class on engineering statistics, which currently does not include statistical concepts, such as the Weibull modulus, that are uniquely related to ceramics.

CONCLUSION

Since Fall 1991, the Materials Engineering Department at San Jose State University has established an Engineering Ceramics Laboratory. This has enabled conversion of a lecture-only undergraduate class in ceramics to a lecture-laboratory mode. A NSF-ILI grant was critical in the department's ability to establish this laboratory, and also to convert the undergraduate class to a lecture-laboratory mode. A laboratory manual for student laboratory experiments in engineering ceramics has also been developed.

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