Interdisciplinary and experiential approach towards the teaching of materials science and engineering

Judy Schneider, Associate Professor, Mechanical Engineering Keisha Walters, Assistant Professor, Chemical Engineering Bagley College of Engineering (BCoE) Mississippi State University (MSU)

Abstract

In industry engineers and scientists with varying backgrounds are expected to form effective project or product teams in order to solve problems and advance technology. This stands in stark contrast with engineering students' academic experience where they are typically exposed only to discipline-specific courses within their major department. Too often a student's first experience in interdisciplinary teamwork is at their first job where they are required to work well within a team that capitalizes upon the complementary strengths of team members from various backgrounds. These gaps can be addressed through college experiences where interdisciplinary teaming is encouraged.

At Mississippi State University, a split-level 4-credit hour course entitled "Experimental Methods in Materials Research" was recently taught to students from 4 different engineering and science disciplines. In addition to traditional lectures, the course contained a weekly laboratory where interdisciplinary teams of students were able to utilize their varied backgrounds and the lecture knowledge to solve problems. A common set of materials, virgin and recycled high density polyethylene were used repeatedly throughout the semester to illustrate concepts and serve as a common test material when learning new experimental testing methods.

Introduction

Many colleges are exploring interdisciplinary approaches to course offerings, a topic promoted by ABET EC2000. A review of recent ASEE publications reflects this growing interest. A good first step is the integration of materials and engineering in capstone design courses [1-2]. Indeed there is an inseparable relationship between the art and science of design as reflected in applied engineering mechanics and materials science [3]. In the pursuit of interdisciplinary approaches much has been learned about effectively teaming together across departments as well as colleges [4]. As with all materials, the bonds between individuals as well as the materials help to keep the course relevant.

Within the Bagley College of Engineering (BCoE) at Mississippi State University (MSU), the lack of a formal MSE department was addressed by the formation of an interdisciplinary Materials Working Group (MWG) in 1995 to bring together faculty from across the university with an interest in materials research. In addition to fostering collaborative research, the MWG also teams faculty for co-teaching materials-relevant courses, such as the interdisciplinary course "Experimental Methods in Materials Research" (ChE/ME 4624/6624). At MSU, this course is taught every two years and has proved successful at bringing together undergraduate and graduate students from different engineering and science disciplines to spend a semester examining material systems at different length scales and correlating atomic or molecular structure with observed macroscale behavior. This approach contrasts with an experimentally-base, traditional MSE course taught from a MSE department to a multidisciplinary group of engineering students which may be also cross-listed, and involve multidisciplinary instructors. In the approach discussed in this manuscript, there is no home department ownership.

Course Content

ChE/ME 4624/6624, Experimental Methods for Materials Research, is a four credit hour course co-taught between the Chemical Engineering and Mechanical Engineering departments, with 3 lecture credit hours and 1 laboratory credit hour. The split-level designation (4xxx/6xxx) indicates the course can be taken either for upper class undergraduate or graduate credit. The class has historically been composed of approximately 90% graduate students, with the enrolled undergraduates considering graduate school. Typically about 12-18 students enroll in this course offered every other year. Offering the course across multiple departments without departmental ownership not only helps satisfy program of study requirements, provide some student contact hour credits for co-teaching departments (a factor in tight budgetary environments), but can also alleviate student fears about taking a course outside their home department.

The Experimental Methods for Materials Research course is concerned with how to approach the testing and characterization of a wide range of materials. Pros and cons of various techniques (e.g., physical limitations, statistical significance, cost -- time and monetary) are taught in an effort to illustrate how various testing and characterization methods can be selected and employed to provide data that will hopefully fit together like pieces of a puzzle upon which conclusions can be drawn. As more pieces of the puzzle are obtained, the more confident one can be in the conclusions drawn. The specific course objectives include:

- 1. Expose students to basic and advanced techniques for characterizing materials.
- 2. Enhance the students' understanding of structure-property relationships.
- 3. Develop an understanding of the limitations and/or suitability of different characterization techniques for particular types of materials.
- 4. Provide students with some experience in collecting, analyzing and interpreting data by different materials characterization techniques.

The course is divided into 5 major parts as summarized in Table I with the formal lecture portion of the course divided into a review of material systems, mechanical testing, imaging and diffraction techniques, and spectroscopy. Lectures cover the internal micro structure, corresponding mechanical behavior, and applications for various material systems; in addition, techniques that can be used to probe the chemical or microstructural

composition are discussed. Laboratories allow hands-on access to the application of theory and data collection using different types of equipment housed in either individual researcher laboratories or in the Electron Microscopy Center (EMC), a centralized user facility on the MSU campus. In this interdisciplinary environment, teams of students from different disciplines work together to obtain experimental data that are analyzed and discussed in the lectures. In addition to the technical instruction, the student researchers gain knowledge of the types, locations, and responsible faculty member for equipment available on campus.

As part of the introductory portion of the class, students and faculty each give an impromptu overview of their research projects. Projects with similar themes, either by end application or overall material system, are used to form teams that will work together for the class duration. As the class progresses, each student team develop a project proposal designed around the different academic backgrounds and research strengths of the team members.

Section	Lecture	Laboratories and Demonstrations
1	Review of materials systems	
	Metals	Basic laboratory procedures, MSDS,
	Polymers	laboratory notebooks, and density
	Ceramics/glass	measurements
	Composites	
2	Testing Methods	
	Quasi Static Uniaxial	Unaxial tension testing
	Tension	Charpy impact testing
	Bend (3 and 4 pt)	
	Dynamic Impact Testing	
3	Imaging/Diffraction Methods	Optical microscopy (OM)
	OM/QM	and Quantitative microscopy (QM)
	SEM	Scanning Electron Microscopy (SEM)
	TEM	Transmission Electron Microscopy (TEM)
	Diffraction Techniques	X-ray Diffraction (XRD)
4	Spectroscopy Methods	
	EDS and EBSD ¹ /SEM	Energy Dispersive Spectroscopy (EDS)
	WDS^2	X-ray Photoelectron Spectroscopy (XPS)
	Auger	
	XPS	
5	Group Project	

Table I. Lecture and laboratory course structure.

¹Electron Backscattered Diffraction (EBSD)

²Wavelength Dispersive Spectroscopy (WDS)

Establishing Laboratory Relevancy via Lectures

To enable ideas and concepts to be reinforced, the lectures and laboratories are coordinated through the selection of certain material systems that can be used to illustrate a concept during the lecture and then also examined in the laboratory. Based on the interests of the instructors, a recent offering of the Experimental Methods for Materials Research course utilized a series of polyethylene (PE) samples as a representative polymer system with its $(CH_2CH_2)_n$ repeat units. This sample set was also used to highlight how collaboration between researchers with different specialties or interests can further the overall understanding of a particular material system. This message was also reinforced with the group projects.

The PE samples selected included commercially available low density polyethylene (LDPE), high density polyethylene (HDPE), and recycled/modified HDPE as summarized in Table II. Since these materials were commercially available, only generic information was provided on the modifier type as often these additives are proprietary. This provided the additional opportunity to utilize characterization techniques to 'reverse engineer' materials and discuss limitations of this approach.

In reviewing material types, the lectures on polymers included a discussion of the chemical similarity and branching structure differences between LDPE and HDPE, as illustrated in Figure 1 [5]. The differences in branching were used to discuss related differences in crystallinities, molecular weight, etc. and also as the basis for thought experiments on how the different polymer structures might respond differently to an applied load. In addition, these PE samples provided an opportunity to discuss potential applications of various PEs based on molecular structure, physical properties, processing costs, and overall material performance. As the lectures moved to mechanical testing, this selection of PEs was discussed in terms of how molecular structure affects mechanical behavior and why polymers might be modified (i.e., enhanced properties) and what the concerns might be regarding selection of modifying agents (i.e. loss of desired properties, cost).

Sample	Polymer	Supplier	Recycled	Thickness	Color and
				(in)	Additional Info
1	HDPE	Sandhill	Yes	1/8 & 1/2	White;
		Plastics			Published additives:
					TiO_2
2	HDPE	Sandhill	Yes	1/8 & 1/2	Black;
		Plastics			Published additives:
					antioxidants
3	HDPE	McMaster	No	1/8	White opaque
		Carr			
7	LDPE	McMaster	No	1/8	Translucent natural
		Carr			

Table II. Commercially available PE samples used as a demonstration sample set throughout the course.



Figure 1. Illustrative schematic of the branching structure of HDPE and LDPE [5].

Comparison of LDPE to HDPE

The lectures on mechanical testing methods included both quasi-static tensile testing and impact testing. The standards developed by ASTM and available through a program called "ASTM International Campus" [6], were used to guide the design of experiments, prepare specimens, establish testing conditions, select instrumentation, and reduce the mechanical property data. Prior to polymer testing, the students tested aluminum and steel specimens to gain familiarity with the uniaxial load frame and basic instrumentation. As part of the metals laboratory report, the students referred to ASTM Standard E111 [7] for modulus calculations and E8 [8] for mechanical property calculations. Students reduced data for their laboratory report and compared their values with published literature [9]. Fractured specimens were labeled and saved for later imaging and spectroscopy laboratories.

To compare the mechanical properties of the LDPE vs HDPE, tensile specimens were prepared in accordance with ASTM Standard D638 [10]. Differences were discussed in the experimental procedure between ASTM Standard E8 for metals [8] and D638 for polymers [10]. In lectures the concept of viscoelastic behavior was introduced to explain differences in expected response to an applied load for a crystalline metal versus that of a polymer chain.

Section 7 of ASTM Standard D638 [10] provided guidance in the number of specimens for testing, dependent on isotropic behavior. To test for isotropic properties, the specimens had been prepared in two orientations, 90 deg apart, relative to the sheet dimensions. The students reasoned the linear structure of the HDPE would be more likely to align during fabrication processes that involved extrusion or rolling thus displaying more anisotropic behavior than the LDPE.

The laboratory provided the opportunity to demonstrate the strain rate insensitivity of a crystalline metal versus the strongly sensitive response of polymers. In selecting the test conditions, ASTM Standard D638 [10] provided a table outlining the selection of cross head speed, or relative motion of the grips during the test, for various materials and specimen geometries. According to Section 8 of the ASTM Standard D638 [10], a speed was to be selected resulting in a specimen rupture within 0.5 to 5 minutes. The class

collectively tested a set of LDPE and HPDE specimens in the 0 deg orientation at cross head speeds of 0.2, 2, and 20 in/min to determine the appropriate rate of 2 in/min.

Thus the polymer testing laboratory approach provided an opportunity to explore the isotropic nature of the LDPE vs. the HDPE with respect to theorized behavior. The homework assignment required a laboratory report to compile all the reduced data. The instructor took responsibility for data reduction for the various cross head speeds evaluated for the specimen number in the 0 deg orientation at 0.2, 2, and 20 in/min. Each student had responsibility for one tensile specimen including testing and data reduction. The reduced data was posted and accessible by all other students. Thus, if the data reduced by one student appeared "out of family", they had the opportunity to interact to discuss any potential anomaly and include this discussion in their individual laboratory report.



Figure 2. Comparison of stress vs. strain plots for the 2 orientations of (a) HDPE and (b) LDPE tested.

Examples of the reduced stress versus strain plots are shown in Figure 2. The students were able to accurately predict that the linear arrangement of the HDPE specimens would require less load for chain alignment and straigthening, as indicated by the lower ultimate tensile stress (UTS). Once the UTS was reached in both PEs, a reduced flow stress or yield was observed which correlated with the sliding and straightening of chains as they aligned in the tensile direction. In LDPE sample, the longer side branching would be expected to interfere with sliding resulting in a lower strain to failure. In the HDPE, after the chains were fully extended, an increase in flow stress was expected as the load would be carried on the covalently bonded strands. However, this behavior was not observed and resulted in further discussion of what might have prevented this behavior. Figure 3 shows the post-test specimen, highlighting the variation in elongation and failure mode. These failed specimens were then tagged for further examination during the imaging laboratory and revealed air bubbles -- predominately in the HDPE -- that were theorized to be the primary cause of premature failure.



Figure 3. Overview of tensile specimens post uniaxial testing for the (a) HDPE and (b) LDPE. A large variation was observed in the strain to failure for the HDPE which was later correlated with air bubbles in the specimen.

Summarized data from the polymer mechanical testing laboratory report are presented in Table III. A 18% difference was observed between the two orientations of the HDPE as compared with a 7% difference in the LDPE. By comparing the standard deviations within each oriented group, the class noted that the HDPE variation between orientations was higher than the experimental standard deviation while it was similar for the LDPE. Thus the experimental data reaffirmed their earlier hypothesis regarding the strength of the two samples with similar chemical structure, but very different chain branching structures. The students compared their results using a freeware database [9] and found expected values for the HDPE. While most values agreed with the reference [9], a large discrepency was noted for the LDPE. However, when considering how the processing method affects the properties, this again reinforced the need for better documentation on the history of the specimen.

Table III. Summary of experimental and literature mechanical property data for LDPE and HDPE tested at two different orientations. A total of 6 tensile specimens were tested to obtain the values listed.

Sample	Initial modulus (ksi)	Overall average modulus (ksi)	UTS (ksi)	Overall average UTS (ksi)
3 HDPE – 0 deg	163 <u>+</u> 7	150 <u>+</u> 14	4.2 <u>+</u> 0.1	4.2 <u>+</u> 0.1
3 HDPE – 90 deg	134 <u>+</u> 7		4.0 <u>+</u> 0.1	
Reference values [9]	175		4.1	
7 LDPE – 0 deg	190 <u>+</u> 13	185 <u>+</u> 12	5.1 <u>+</u> 0.1	5.1 <u>+</u> 0.1
7 LDPE – 0 deg	176 <u>+</u> 11		5.2 <u>+</u> 0.1	
Reference values [9]	70		2	

Modified HDPE

Since the modified HDPE samples could be commercially obtained in 0.5" thick sheets, it was possible to machine impact tensile specimens to evaluate changes in mechanical behavior and fracture toughness. Figure 4 shows a comparison of the modulus and UTS

for the various HDPEs in the 1/8" thick sheets including reference data [9] for comparison. Students found that the "black" modified HDPE displaced showed a reduced stiffness (modulus) and strength compared to the unmodified specimens. In contrast, the "white" specimens displayed increased stiffness (modulus) and strength. Figure 5 shows an overview of the literature and experiment initial modulus and UTS values for the neat (unmodified) and modified HDPE specimens.



Figure 4. Comparison of literature [9] and experimental initial modulus and UTS properties for HDPE samples, including 6 specimens tested for neat (unmodified) and modified ("black" and "white").



(a) (b) **Figure 5.** Variation in fracture behavior between the tensile specimens of (a) "black" HDPE versus (b) "white" HDPE.

Dynamic impact testing was used to determine the material's resistance to a high-rate of loading. The samples were notched to promote a brittle rather than a ductile fracture in accordance with ASTM standards [11]. ASTM standard D6110 [11] for notched impact testing was used to guide the specimen geometry and establish the test procedures. The method was modified since the existing ASTM standard D6110 is designed for the pendulum style of impact testers. It should be noted that for the older pendulum style

impact tester the pendulum length and weight sets the velocity and energy of impact while the newer drop tower impact tester can only duplicate either the velocity or the energy [12, 13]. Figure 6 shows a comparison of the impact strength of the modified HDPE samples as compared to literature values. Reference values for the HDPE were included showing an approximately 50% decrease in toughness for the modified HDPEs.



Figure 6. Comparison of literature [9] and experimental impact strengths for the modified HDPE samples ("black" and "white").

As part of the imaging laboratory, the students used scanning electron microscopy (SEM) to collect images of the fractured surfaces. Electron microscopy captures a large depth of field, suitable for documenting the as-fracture surfaces. Figure 7 is a secondary electron image (SEI) of the two modified HDPE fracture surfaces. Similar fracture patterns were observed with notable non-uniformity regions corresponding with potential defects.

Figure 8 shows a magnification of the non-uniform area noted on the "white" specimen which revealed embedded spherical particles. The commercial information available for the "white" specimen had indicated the addition of TiO_2 particles. For the laboratory report, the students were able to obtain further information regarding the morphology and size of TiO_2 particles which corresponded to their observations. These specimens were also used in the spectroscopy portion of the class where the students could verify using EDS/SEM that the particle inclusions did contain Ti.



Figure 7. SEM SEI comparison of impact fracture surfaces of two modified HDPEs (a) "black" and (b) "white". Arrows show non-uniform regions of potential defects.



Figure 8. Magnified region from Figure 7b showing details of non-uniform region in the impact fracture surface of the "white" HDPE specimen.

Table V correlates the use of the HDPE samples with lecture topics in a recent offering of ChE/ME 4624/4424. Because the same specimens were used for various laboratories, this encouraged discussion in advance of expected behavior for the mechanical property testing and what to look for in subsequent imaging and spectroscopy laboratories. The students were able to verify the use of TiO_2 additives in the "white" modified HDPE specimens by subsequent elemental analysis. Evidence of clumping was associated with reduced impact strength or toughness. Little data was available on the "black" modified HDPE specimens and thus subsequent spectroscopy failed to identify the modifiers. The students reasoned that the "black" color may have been due to some type of carbon additive.

Торіс	HDPE vs. LDPE	Modified HDPE
Mechanical properties	Laboratory:	Laboratory:
	quasi-static behavior	dynamic impact behavior
Imaging		OM and SEM
Spectroscopy		EDS/SEM

Table V. Use of HDPE samples to reinforce lecture concepts through laboratory exercises.

The mechanical properties for the HDPE compared favorably with the literature. Based on discussion of the underlying molecular chains, little difference was expected in reported properties. However, the LDPE with the longer side branching is expected to demonstrate considerable differences in mechanical properties vs. the HDPE, even at comparable molecular weights. Due to time limitations, LDPE measurements were not made but may be added as an element in future classes. Overall, it was shown that while one modification system for HDPE increased the stiffness and strength, another additive system caused a decrease. Clumping of additives in the "white" modified HDPE was found in the fracture surfaces of the impacted specimens which would be expected to correspond with decreased fracture toughness. This was recognized as a manufacturing issue which was also observed in the measured tensile elongation as compared with literature values [9]. Overall the students gained a hands-on appreciation for how processing can affect overall mechanical properties.

Group Projects

As the students progressed through the formal lectures and accompanying laboratories, bonds were formed across departmental boundaries. Complimentary strengths among the class members were recognized from the laboratories. Using information obtained about various techniques and capabilities on the MSU campus, the student teams were tasked to propose a topic, submit an abstract, and develop a formal research proposal. Once the research proposal was accepted by the instructor, each laboratory facility supporting the class was notified regarding the student's request for instrument access, type of specimens, and estimated instrument and data analysis time for the proposed experiments. The students then had responsibility for scheduling their samples and obtaining their data. At the end of the semester, the student teams prepared a final report and orally presented their research results to various faculty across campus. Table IV summarizes some recent projects and team composition. Only one of the 4 teams included students from only one department. The others formed humanistic bonds on the basis of similar research interests.

Material System	Team Disciplines	Techniques Utilized
Ce-based Coating on	Chemistry, ChE	SEM, AFM, XRD, XPS
AA Substrates		
Carbon Nanofiber Polymer	ASE	Tensile testing, OM, SEM, QM
Composite		
NaCl Crystals	ASE, ChE	XRD (powder vs single crystal),
		SEM
Bio-oil Contact with Storage	ChE, ME	Tensile testing, SEM, XPS
Materials		_

Table IV. A summary of the team projects for the Fall 2009 Experimental Methods for Materials Research course at MSU.

Course Assessment

From student performance and feedback, the course content and structure, interdisciplinary nature of the instructors and student population, and use of a common sample set throughout the course positively affected student engagement. Along these lines, a fascinating occurrence was the level of discussion among the students in the laboratories. It remains unknown if the dramatically increased interaction level correlated with the time of day -- as the lectures were at 11 am on Tuesday/Thursday and the laboratory at 5 pm on Wednesday -- or some other factor. Perhaps the lecture on Tuesday, which set the stage for the laboratory on Wednesday, gave students time to digest the material and be prepared to raise questions. Another possibility may have been the impending homework assignment (received during the laboratory session) that encouraged questions and discussion. Or perhaps the informality of the laboratory setting put students at ease.

It is difficult to assess directly the impact of the interdisciplinary and team-based elements of the course on student learning and interest in materials research. This is primarily due to the lack of a comparable course that does not include these features, but also due to small class size and frequency of the course offering. However in future semesters, the instructors do plan to use a survey-style assessment to gauge the student's perceived impact on learning and interest based on different elements of the class.

Conclusions

Co-teaching of an interdisciplinary course to engineers and scientists can be a rewarding learning experience for all involved. Using a common set of materials, such as the PE described, in the majority of the laboratory exercises provided a common thread to link the various aspects of the science and engineering of materials. Selecting instructors from different departments, outside of a home department ownership, proved effective in removing perceived departmental ownership of the subject matter. Students who have completed this course also have a greater appreciation of the various equipment and capabilities available to them through.

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