AC 2008-260: EXPERIMENTS IN MICRO-/NANO-CHRACTERIZATION OF MATERIALS SURFACES

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Experiments in Micro-/Nano- Characterization of Material Surfaces

Abstract

This paper describes major revisions made in Spring quarter of AY 2006-7 to a 4-quarter credit hour upper-division technical elective course on Micro- and Nano- Characterization of Material Surfaces. The course was first offered in the Spring quarter of AY 2005-6, and has 3 hours/week of lectures and a 2 hours/week laboratory segment. The course is now a part of a concentration program in Nanotechnology and MEMS being developed under a department-level reform grant from the National Science Foundation to the department of microelectronic engineering. To accommodate brief reviews of differential equations and materials science concepts, the course now covers only *two* families of experimental techniques: x-ray diffraction and scanning probe microscopy. At the end of each of the eight experiments, each student pair is given a different experimental dataset to analyze. Students submit their analyses and results in a written lab report. The paper describes the format of the lab reports, and compares course evaluations by students before and after the revisions were made.

Introduction

Rochester Institute of Technology (RIT) is a predominantly undergraduate teaching university with an emphasis on applied research in *five* focus areas: imaging, microsystems, astrophysics, manufacturing, and information technology. It offers PhD degrees in both Imaging Science and Microsystems Engineering. Multidisciplinary research in these two areas is supported by the Center of Imaging Science, Information Technology Collaboratory (a NY State designated Strategically Targeted Academic Research Center), and Semiconductor & Microsystems Fabrication Laboratories (SMFL). Materials characterization and testing needs in these research projects are partially met by equipment available in the Advanced Materials Lab (AML).

AML is the only facility at RIT that has equipment for scanning probe microscopy (SPM), x-ray diffraction (XRD), micro- and nano- indentation, and quantitative imaging. With greater participation of undergraduate students in research projects involving microelectronic thin films, photo-voltaic materials, MEMS devices and nano-crystalline tribological coatings, AML has experienced a surge in demand for its materials characterization and testing services in the last five years. To help undergraduate and graduate students learn the experimental techniques and underlying theoretical principles, a 4-quarter credit hour upper-division technical elective course titled *Micro- and Nano- Characterization of Material Surfaces* was developed and offered for the first time[1] in the Spring quarter of AY 2005-6 (Q20053). The course covered *three* families of materials characterization techniques: atomic force microscopy (AFM), x-ray diffraction, and optical microscopy. The course became a part of a concentration program in Nanotechnology and MEMS being developed under a department-level reform grant[2] from the National Science Foundation to the department of microelectronic engineering at RIT.

This paper describes the major revisions to the course based on feedback from students and observations of the instructor[1] when the course was offered for the second time in the Spring quarter of AY 2006-7 (Q20063). In Q20053, fourteen students majoring in mechanical engineering, microelectronic engineering, microsystems engineering, physics, chemistry, or materials science and engineering enrolled in the course including one taking it for audit. All of the students had taken standard university-level Physics and Calculus courses as well as another course covering introductory materials science, and therefore, were expected to have a reasonable background satisfying the pre-requisites for the course. Of the 14 students, two graduate students withdrew early in the quarter due to prolonged illness or inadequate background. Students' expertise in algebra and basic calculus ranged from poor to adequate. Of the remaining 12 students, seven more withdrew from the course later in the quarter because they were unlikely to obtain an "A" or "B" in the course.

In Q20053, students working in pairs were asked to work on two experimental projects to characterize material surfaces of their choice and submit project reports. However, several pairs chose material surfaces that did not deserve characterization by high resolution equipment such as the atomic force microscope or x-ray diffractometer. The instructor spent considerable time out of the class in helping students do the lab projects.

Lab Equipment and Class Size

AML's equipment includes a Rigaku DMAX-IIB X-Ray Diffractometer (XRD), a DI-3000 Scanning Probe Microscope (SPM), Mitutoyo Micro-hardness Tester, and Olympus Microscopes with Image Pro Plus for image acquisition, processing and analysis. With a recent Major Research Instrumentation award from the National Science Foundation (NSF), the lab acquired a high-resolution x-ray diffractometer with a general-area detector system (Bruker D8 with GADDS). D8 has capability for x-ray reflectometry and high-resolution x-ray diffractometry. The Department of Microelectronic Electronic was recently awarded a Department Level Reform (DLR) grant from the NSF that helped the lab to acquire a PSIA XE-150 scanning probe microscope with additional electronic, magnetic and thermal characterization capabilities. These equipment acquisitions will provide additional opportunities to significantly enhance this course next year.

The lectures are held in a standard classroom and the labs are held in 800 ft^2 AML that houses the XRD, SPM, hardness and microscopy equipment. The lab can seat 15 students around a very large conference table so the class size is limited to 15. AML has a ceiling-mounted video projection system that has wireless connection to all of the personal computers in the lab. The system is used to display patterns or images during the acquisition and processing steps as well as the equipment control software so that the entire class benefits from the enlarged view, and also participates in the lab discussions.

Revisions to the Course

To accommodate brief reviews of differential equations and materials science concepts, the course in Q20063 covered only *two* families of experimental techniques: x-ray diffraction and scanning probe microscopy. The portion on optical microscopy was eliminated. Scanning probe microcopy portion of the course was moved to the end. The course has 3 hours/week of lectures and a 2 hours/week laboratory segment. Table I lists the lecture topics, companion lab experiments, textbook, and reference books.

TABLE I
LECTURE TOPICS AND LAB EXPERIMENTS

Class	**Lecture Topics**	**Weekly Lab Experiments**
1 2 3	Course Policies & Introduction Lattice Points, Lines, Planes[3] Bravais Lattices, Crystal Structure	Lab Procedures & Safety Regulations
4 5 6	Properties of X-Rays[4] Filters & X-Ray Tube Bragg's Law & Laue Equations	#1: Powder Diffractometer #1: Acquiring & Indexing a Pattern
7 8 9	X-Ray Methods Scattering of X-Rays Structure Factor	#2: Intensity Calculations #2: Phase Identification using ICDD-PDF
10 11 12	Diffraction by Polycrystalline Material Summarize Part I of X-Ray Diffraction Test #1	#3: Alignment & Calibration #3: Precise Lattice Parameter
13 14 15	Effect of Crystallite Size Strain & Perfect Crystal Peak Shapes and Profiles	#4: Peak Profile Parameters #4: Grain Size Broadening
16 17 18	Grain Size in Polycrystalline Material Microstrain & Penetration Depth General Principles of Stress	#5: Peak Position Determination #5: Residual Stress Measurement
19 20 21	Residual Stress Summarize Part II of X-Ray Diffraction Test #2	#6: Scanning Probe Microscopy[7] #6: Contact Mode Atomic Force Microscopy
22 23 24	Two Particle Interaction Static Deflection of a Beam[5] Undamped Free Vibrations[6]	#7: Non- and Intermittent Contact #7: Tapping Mode Atomic Force Microscopy
25 26 27	Undamped Forced Vibrations Damped Free Vibrations Damped Forced Vibrations	#8: Interleave Mode of SPM #8: Magnetic Force Microscopy
28 29 30	Vibration Considerations in AFM Summarize Part III on SPM Concluding Remarks	Other Modes of SPM Image Processing & Analysis
31	Comprehensive Final Exam	

A textbook[4] was assigned for the x-ray diffraction portion of the course. Unfortunately, the third edition of the textbook had many typesetting errors. Two classes introducing students to Bravais lattices and crystal structures were added at the beginning of the course. Reference textbooks covering introductory concepts from materials science[3], cantilevered beam deflection[5], and vibrations of a cantilevered beam[6] were kept in the reserve section of the library for student use.

The course had bi-weekly homework assignments that constituted 30% of the grade. Two closed book mid-term tests and a comprehensive final exam constituted 50% of the grade. The remaining 20% of the grade was assigned to eight lab reports.

In the revised course, the laboratory segment was revamped. Lab projects of students' choice were eliminated. Each week in the laboratory, students collected experimental data on a well-characterized specimen, and completed the data analysis in the class under the guidance of the instructor. For the XRD experiments, students used either a calculator or spreadsheets. For SPM experiments, students were directed to use a freeware called WSxM. At the end of each experiment, each student pair was given a different experimental dataset to analyze. Students submitted their analyses and results in a written lab report. The lab report writing was intended to prepare students to manage a materials characterization lab in academic or industry. Each report had four major sections: (i) Abstract of Specimen Details and Experimental Results, (ii) Equipment and Specimen Details, (iiia) Experimental Principles or (iiib) Step-by-step Laboratory Instructions, and (iv) Experimental Data and Analysis. Each student pair alternated in writing either the experimental principles or the step-by-step instructions.

Concluding Remarks

In Q20063, eleven students enrolled in the course including one taking it for audit and two doctoral students in microsystems engineering. All ten students finished the course successfully earning either an "A" or "B" grade. The changes in the format and the content of the revised course also improved considerably the students' satisfaction and appreciation of the course as is evident in their course evaluations. On a scale of 1 to 5 (highest), the average numerical response to selected questions from the evaluation forms collected in each of the two quarters in the past when the course was offered is listed in Table II.

TABLE II
EVALUATION QUESTION AND AVERAGE NUMERICAL RESPONSE

Q#	**Question**	**Average**	
		20063	**20053**
		N=9	**N=4**
2	What is your present feeling about how much you learned in this course?	4.42	4.00
3	What is your opinion of the principal textbook in this course?	2.75	0.00
4	In general, how do you feel about the out of class assignments?	4.39	5.00
5	How was the instructor's presentation in helping you understand the material associated with the course?	4.72	4.00
9	How stimulating was the instructor?	4.14	3.50
16	Overall, how would you rate this course?	4.57	3.50
17	Overall, how would rate this instructor?	4.43	3.75

Table II shows significant improvement in student ratings for all items except for Q3 (textbook) and Q4 (homework) for revised course. The third edition of the textbook had many errors, and even though the instructor provided a hardcopy of the errata in the first class it was not satisfactory. In Q20053, students submitted only five homework sets and two lab reports whereas in Q20063, six homework sets and eight lab reports were required. This might explain the lower rating on Q4.

In their written comments, students unanimously found the instructor to be clear, systematic and well organized. They thought that the lab experiments complemented the lecture material very well. They suggested that SPM experimental datasets could have been more interesting and that the instructor should have provided more guidance in class in using the WSxM software for analysis.

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