Experiments in Micro-/Nano- Characterization of Material Surfaces

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Abstract

This paper describes eight experiments developed for a 4-quarter credit hour upper-division technical elective course on Micro- and Nano- Characterization of Material Surfaces. The course has 3 hours/week of lectures and a 2 hours/week laboratory segment. Offered for the second time last Spring quarter, the course has attracted students from mechanical engineering, microelectronic engineering, materials science and engineering as well a doctoral student in Microsystems Engineering. The course has become 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. The experiments cover two families of techniques: atomic force microscopy and x-ray diffraction. At the end of each experiment, each student pair is given a different experimental dataset to analyze. Students submit their analysis and results in a written lab report. The paper also describes the format of the lab reports.

Introduction

In the last three years, the Advanced Materials Lab (AML) has experienced a surge in demand for its characterization and testing services. This surge in demand is primarily due to greater participation of undergraduate students in research projects involving microelectronic thin films and devices, micro-electro-mechanical systems (MEMS), and nanocrystalline tribological coatings. 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. We train advanced undergraduate and graduate students in the use of these experimental tools to image and probe surface properties at micro- and nano- scales. However, in the one or two day hands-on training exercises, the students develop only a limited understanding of these powerful experimental techniques. Pedagogically, they should learn the physics of the interaction processes used for characterization, quantification and interpretation of collected signals, common artifacts, the engineering tradeoffs made in constructing actual instruments, and the fundamental limits for each method.

This paper describes eight experiments developed for a 4-quarter credit hour upper-division technical course on Micro- and Nan- Characterization of Material Surfaces. The course has 3 hours/week of lectures and a 2 hours/week laboratory segment.

Course Audience and Pre-requisites

Offered for the second time in Spring quarter of AY 2006-7, the course attracted students from mechanical engineering, microelectronic engineering, materials science and engineering, and Microsystems engineering. The only prerequisites required for this course are standard university-level Physics and Calculus courses as well as an introductory materials science course. The course is assigned a 700-level identification so that it can count as a technical elective for BS students and a graduate elective for MS or PhD students in science and engineering. These students are expected to have a reasonable background in Physics and Mathematics. Table I lists the materials science pre-requisite that is appropriate for students enrolling in this course.

TABLE I
ACADEMIC PROGRAMS AND PRE-REQUISITE

Academic Program	**Materials Science Pre-requisite**	**Quarter Offered**
BS or MS in Mechanical Engineering	0304-344: Introductory Materials Science	Fall, Winter and Spring
BS or MS in Microelectronic Engineering	0305-460: Semiconductor Devices I	Fall and Spring
MS in materials Science and Engineering	1028-701: Introductory Materials Science	Fall
PhD in Microsystems Engineering	1028-701: Introductory Materials Science	Fall

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 course is a part of a concentration program in nanotechnology and micro-electromechanical systems (MEMS) being developed with support from the DLR grant[1].

The lectures and 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 class/lab discussions.

Course Content

The course covers two families of experimental techniques: x-ray diffraction and scanning probe microscopy. Table II lists the lecture topics, companion lab experiments, textbook, and reference books. The topics, experiments and their order in the course were modified significantly from the first time the course was offered[2] in the Spring of AY 2005-6.

TABLE II
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	

Grading and Labs

The course has bi-weekly homework assignments that constitute 30% of the grade. Two closed book mid-term tests and a comprehensive final exam count for 50% of the course grade. At the end of each experiment, each student pair is given a different experimental dataset to analyze. Students submit their analysis and results in a written lab report. The eight lab reports constitute the remaining 20% of the grade. The lab report writing is intended to prepare students to manage a materials characterization and analysis lab in academia or industry. Each report has 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 alternates in writing either the experimental principles or the step-by-step instructions.

Concluding Remarks

Many changes in the format and the content of the course in its first offering[2] in Spring of AY 2005-6 were made based on the feedback from students in their end of the quarter course evaluations:

1. A textbook[4] was assigned for the x-ray diffraction portion of the course. Unfortunately, the third edition had many typesetting errors.
2. Scanning probe microscopy portion of the course was moved to the end.
3. Two classes introducing students to Bravais lattices and crystal structures were added at the beginning of the course.
4. Instead of lab projects of students' choice, each student pair was assigned a different experimental dataset for analysis and lab report.

These changes 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 is listed in Table III.

TABLE III
EVALUATION QUESTION AND AVERAGE NUMERICAL RESPONSE

Q#	Question	Average	
		20063	**20053**
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

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References

[1] S. K. Kurinec et al and S. K. Gupta, *Microelectronic Engineering Education for Emerging Frontiers*, Proceedings of the 9th International Conference on Engineering Education, San Juan, PR, 2006.

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[3] W. D. Callister, *Fundamentals of Materials Science and Engineering,* Seventh Edition, John Wiley, 2005.

[4] B. D. Cullity and S. R. Stock, *Elements of X-Ray Diffraction*, Third Edition, Prentice Hall, 2001.

[5] F. P. Beer, E. R. Johnston and J. T. DeWolf, *Mechanics of Materials,* 4th Edition, John Wiley, 2006.

[6] R. C. Hibbeler, *Dynamics,* 11th Edition, Pretice Hall, 2007.

[7] *A Practical Guide to Scanning Probe Microscopy*, Veeco Instruments, 2005.

Biographical Information

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"Vinnie" Gupta is a Professor of Mechanical Engineering and Materials Science & Engineering, and the recipient of the 2000 Eisenhart Award for Excellence in Teaching. At RIT, he teaches undergraduate and graduate courses in Applied Mechanics, Computational Techniques, and Materials Science.