Micro-/Nano- Characterization of Material Surfaces

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Abstract - **This paper describes the development of a 4-quarter credit hour upper-division technical elective course on Micro- and Nano- Characterization of Material Surfaces with a laboratory component that is being offered in the standard format of 3 hours/week of lectures and a 2 hours/week hands-on laboratory segment. Offered for the first time this Spring quarter, the course has attracted students from mechanical engineering, microelectronic engineering, materials science and engineering as well as doctoral students in Microsystems Engineering. It 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. It covers three families of experimental techniques: scanning probe microscopy, x-ray diffraction, and quantitative imaging. Each student pair is working on two experimental projects characterizing materials surfaces of their choice. The course has bi-weekly homework assignments and two written exams.**

Index Terms – Characterization, Quantitative Imaging, Scanning Probe Microscopy, X-Ray Diffraction.

INTRODUCTION

In the last three years, the Advanced Materials Lab (AML; 09-2170) 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 the collected signals, common artifacts, the engineering tradeoffs made in constructing actual instruments, and the fundamental detection limits for each method.

This paper describes the development of a 4-quarter credit hour upper-division technical elective course on Micro- and Nano- Characterization of Material Surfaces with a laboratory component that is being offered in the standard format of 3 hours/week of lectures and a 2 hours/week hands-on laboratory segment.

COURSE AUDIENCE AND PRE-REQUISITES

Offered for the first time this Spring quarter of AY 2005-6, the course has 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 programs 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	F, W, Sp
BS or MS in Microelectronic Engineering	0305-460: Semiconductor Devices I	F, Sp
MS in Materials Science and Engineering	1028-701: Introductory Materials Science	F
PhD in Microsystems Engineering	1028-701: Introductory Materials Science	F

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 will soon have 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 will allow the lab to acquire a scanning probe microscope with additional electronic and magnetic 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-electro-mechanical systems (MEMS) being developed with support from the DLR grant.

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 images, image acquisition and processing steps, and 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 three families of experimental techniques: scanning probe microscopy, x-ray diffraction, and quantitative imaging. Table II lists the lecture topics, companion lab experiments, and reference books (a single textbook is not available for such a course).

GRADING AND LABS

The course has bi-weekly homework assignments that constitute 30% of the grade. A closed-book mid-term test and a comprehensive final exam will each count for 20% of the course grade. Each student pair will work on two experimental projects to characterize material surfaces of their choice. The two lab reports will constitute the remaining 30% 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) Equipment and Specimen Details, (ii) Experimental Principles, (iii) Step-by-step Laboratory Instructions, and (iv) Experimental Data and Analysis.

CONCLUDING REMARKS

At the time of the writing of this manuscript, we are at the mid-point of the Spring quarter. The remarks below summarize facts and some observations by the course instructor:

1. Fourteen students enrolled in the course including one taking it for audit. Majors represented include mechanical engineering, microelectronic engineering, physics, chemistry, and materials science and engineering. One professor of microelectronic engineering is also attending the course.
2. Of the 14 students, one graduate student withdrew from the course due to inadequate background in physics and mathematics, and another graduate student withdrew due to prolonged illness.
3. Students' expertise in algebra and basic calculus ranges from poor to adequate. This diversity is a challenge for the instructor, and a meaningful solution is not easily evident.
4. In the first set of lab projects, a few students chose material surfaces that do not deserve characterization by a high resolution equipment such as the atomic force microscope. This will be remedied in the second set of lab projects by specifying clearly the kinds of material surfaces suitable for sophisticated characterization.
5. The instructor underestimated the time required out of the class to help students do the lab projects. Better scheduling during the first week of classes will alleviate this problem.
6. The use of wireless connection from the equipment PCs to the video projection system has worked exceptionally well. In future, incorporating a video camera to display different parts of an instrument may further improve the experimental demonstrations.

TABLE II
LECTURE TOPICS AND LAB EXPERIMENTS

Class	**Topic**	**Reference**
1	Course Policies & Introduction	
2	Two Particle Interaction	[1]
3	Static Deflection of a beam	[2]
Lab 1	*Contact Mode SPM; F-D Curves*	[3]
4	Undamped Free Vibrations	[4]
5	Undamped Forced Vibrations	[4]
6	Damped Free Vibrations	[4]
Lab 2	*Tapping Mode Atomic Force Microscopy (AFM)*	[3]
7	Damped Forced Vibrations	[4]
8	Intermittent & Non-contact AFM	[5]
9	Other Modes of SPM	[5]
Lab 3	*Phase Imaging using AFM*	[3]
10	Resolution and Limits of SPM	[5]
11	Image Artifacts and SPM Tip Shape	[5]
12	Introduction to Bravais Lattices	[6]
Lab 4	*Magnetic Force Microscopy (MFM)*	[3]
13	Miller Indices of Directions & Planes	[6]
14	Production/Properties of X-rays	[6]
15	Mid-term Exam	
Lab 5	*XRD Powder Pattern & Indexing*	[7]
16	X-Ray Diffraction	[6]
17	Intensity of Diffracted Beam	[6]
18	XRD Alignment & Calibration	[6]
Lab 6	*Particle Size Broadening*	[7]
19	Stresses and Strains	[2]
20	Residual Stress	[6]
21	Diffraction Peak Profile	[6]
Lab 7	*Residual Stress Measurement by XRD*	[6]
22	Developments in XRD	
23	Microscopy & Imaging	[8,9]
24	Point Operations in Image Processing	[8,9]
Lab 8	*Image Capture and Processing*	[9]
25	Spatial Operations in Image Processing	[9]
26	Edge Detection	[9]
27	Morphological Operations	[9]
Lab 9	*Segmentation and Image Analysis*	[9]
28	Shape Identification	[8,9]
29	Image Parameters	[8,9]
30	Skeletonization	[8,9]
Lab 10	*New Developments and Conclusions*	
31	Comprehensive Final Exam	

ACKNOWLEDGMENT

Financial support from the National Science Foundation through grants # EEC-0530575 (PI: Dr. Santosh Kurinec) and # ECS-0521341 (PI: Gupta) is gratefully acknowledged.

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