MSE 498 Modern Methods in Materials Characterization

Preliminary schedule – Spring 2021, Tuesdays and Thursdays 9:30 am to 10:50 am

	Dates:	**Topics:**
1	January 26	Comparative overview of analytical methods. Fundamental aspects of materials interactions with photons, electrons and ion probing species. General measurement strategies and concepts of resolution, energy, detection limits, accuracy, precision, noise, and data fitting and deconvolution.
2	January 28	X-ray scattering, powder x-ray diffraction and structure determination
3	February 2	X-ray analysis applied to texture, stress and specific methods for thin films, x-ray reflectometry
4	February 4	X-ray reflectometry and high-resolution reciprocal space mapping, x-ray fluorescence and small-angle x-ray scattering (SAXS)
5	February 9	*Live demo and data acquisition using a modern x-ray diffractometer at the MRL*
6	February 11	X-ray photoelectron spectroscopy and comparison with Auger electron spectroscopy and ultraviolet photoelectron spectroscopy
7	February 16	X-ray photoelectron spectroscopy: modern data analysis
8	February 18	Thermal analysis methods: thermogravimetric analysis and differential scanning calorimetry (DSC)
9	February 23	Particle size analysis, including dynamical light scattering and comparison with SAXS
10	February 25	*Live demo and data acquisition using a modern particle zeta sizer and DSC at the MRL*
11	March 2	Ellipsometry
12	March 4	Raman spectroscopy including tip-enhanced methods
13	March 16	*Live demo and data acquisition using a modern confocal Raman spectrometer at the MRL*
14	March 18	Optical profilometry and general imaging processing methods
15	March 23	Atomic force microscopy: topography and basic imaging analysis strategies
16	March 25	Atomic force microscopy: advanced measurement methods
17	March 30	*Live demo and data acquisition using a modern atomic force microscope and an optical profiler at the MRL*
18	April 1	Electron microscopy: general concepts, instrumentation. Scanning electron microscopy.
19	April 6	Advanced analytical methods using scanning electron microscopy: energy dispersive spectroscopy (EDS), electron backscatter diffraction, cathode luminescence, etc.
20	April 8	*Live demo and data acquisition using a modern scanning electron microscope at the MRL*
21	April 13	***Campus break - No instructions day***
22	April 15	Transmission electron microscopy (TEM): imaging and aberration-corrected high-resolution mode
23	April 20	Advanced analytical spectroscopy applied to TEM including EDS, energy electron loss spectroscopy and in-situ thermo and nanomechanical analysis methods
24	April 22	Cryo-electron microscopy including overall sample preparation of biological materials
25	April 27	*Live demo and data acquisition using a modern high-resolution aberration-corrected analytical transmission electron microscope at the MRL*
26	April 29	Summary of ion-based characterization techniques
27	May 4	Course closing with comparison of the various techniques with applications
	May 6	***Reading day - no classes, no finals***
	May 11	***Finals***

Course Syllabus

MSE 498
Modern methods in materials characterization

https://courses.illinois.edu/schedule/2021/spring/MSE/498

Instructor: Dr. Mauro Sardela, Materials Research Laboratory, Director of Central Research Facilities

Guest lecturers: Dr. CQ Chen, Dr. Kristen Flatt, Dr. Rick Haasch, Dr. Roddel Remy, Dr. Julio Soares, Dr. Kathy Walsh, and Dr. Jade Wang.

Course Schedule: Tuesdays, Thursdays 9:30 am to 10:50 am (starts on Jan 26, 2021)

A. Description:

This course provides an in-depth and critical review of the most common interdisciplinary materials characterization methods applied to materials in general (including polymeric and biological systems), with focus on ***modern methods*** related to the state-of-the-art instrumentation available on campus in the Materials Research Laboratory. We plan to take advantage of the MRL's decades-long experience in providing training and support to researchers with various levels of experience and expertise in various scientific fields. The classes will provide a direct correlation between the basic physical and chemical principles of each technique, the required mathematical formalism, and practical experience with instrumentation. We will be using textbooks in the area in addition to recent review papers available in the literature. A direct integration between the various concepts and methods discussed in class will be done with advanced, state-of-the-art instrumentation available in the MRL shared facilities. During the classes, live demonstrations of instrumentation operation and data collection will be provided using relevant samples in various areas of Engineering, Physical, Biological, and Chemical Sciences. The instructor and guest lecturers all have decades-long experience with the respective techniques.

B. Organization:

The course will start with the general fundamentals of analytical techniques, comparing the various interactions of materials with probing species such as photons (light, x-rays), electrons and ions, in addition to mechanical and thermal probes. A detailed review of metrology concepts such as accuracy, precision, resolution, detection limits, etc., will be presented in order to facilitate the discussions of specific techniques in later lectures. The course will proceed with lectures on selected mainstream analytical techniques with emphasis on modern methods – many of them just emerging in the literature and not necessarily available in textbooks. A considerable number of review papers on various techniques will be used as reference material. As the classes progresses throughout the various techniques, physical and chemical basis of the methods will be presented, in addition to the required mathematical formalism. Most of the techniques will include direct lab work using the state-of-the-art MRL shared facilities, with specific data being acquired for the students to analyze as part of the course evaluation metrics.

C. Course Objectives:

The course's main goal is to offer our students a robust, critical and comparative overview of the main analytical techniques applied to Science and Engineering, with a clear focus on modern methods that can be immediately applied to the students future career work in industry, academy or research centers. The students should be able to independently identify and apply the best technique or set of techniques for specific research problems. It is imperative that the students acquire a clear understanding of the advantages and limitations of each technique. Detailed understanding of possible measurement artifacts intrinsic to each technique is also a priority.

D. Course Topics Outline:

Topics Covered	**Contact Hours**
Comparative overview of analytical methods. Fundamental aspects of materials interactions with photons, electrons and ion probing species, in addition to the mechanical and thermal probes.	1
General measurement strategies and concepts of resolution, energy, detection limits, accuracy, precision, noise, and data fitting and deconvolution.	1
X-ray scattering, powder x-ray diffraction and structure determination	2
X-ray analysis applied to texture, stress and specific methods for thin films	2
X-ray reflectometry and high-resolution reciprocal space mapping	2
X-ray fluorescence, small-angle x-ray scattering (SAXS)	2
Live demo and data acquisition using a modern x-ray diffractometer at the MRL	1
X-ray photoelectron spectroscopy and comparison with Auger electron spectroscopy and ultraviolet photoelectron spectroscopy	2
X-ray photoelectron spectroscopy: modern data analysis and deconvolution methods	2
Thermal analysis methods: thermogravimetric analysis and differential scanning calorimetry (DSC)	2
Particle size analysis, including dynamical light scattering and comparison with SAXS	1
Live demo and data acquisition using a modern particle zeta sizer and DSC at the MRL	1
Ellipsometry	2
Raman spectroscopy including tip-enhanced methods	2
Live demo and data acquisition using a modern confocal Raman spectrometer at the MRL	1
Optical profilometry and general imaging processing methods	1
Live demo and data acquisition using a modern confocal laser optical profiler at the MRL	1
Atomic force microscopy: topography and basic imaging analysis strategies	2
Atomic force microscopy: advanced measurement methods	2
Live demo and data acquisition using a modern atomic force microscope at the MRL	1
Electron microscopy: general concepts, instrumentation. Scanning electron microscopy.	2
Advanced analytical methods using scanning electron microscopy: energy dispersive spectroscopy (EDS), electron backscatter diffraction, cathode luminescence, etc.	2
Live demo and data acquisition using a modern scanning electron microscope at the MRL	1
Transmission electron microscopy: imaging and aberration-corrected high-resolution mode	2
Advanced analytical spectroscopy applied to transmission electron microscopy including EDS, energy electron loss spectroscopy and in-situ thermo and nanomechanical analysis methods	2
Cryo-electron microscopy including overall sample preparation of biological materials	2
Live demo and data acquisition using a modern high-resolution aberration-corrected analytical transmission electron microscope at the MRL	1
Summary of ion-based characterization techniques	1
Course closing with comparison of the various techniques with applications	1
Total	*45*

E. Required text and supplies:

All required literature will be made available through Compass or university library.

Several review and application papers from recent scientific publications in related fields will also be provided.

Selected chapters from:

- *Transmission Electron Microscopy: a textbook for materials science,* by David Williams, Springer.
- *Scanning Electron Microscopy and X-Ray Microanalysis*, by Joseph Goldstein, 4^{th} edition, Springer.
- *An introduction to sample preparation and imaging by cryo-electron microscopy for structural biology*, by Rebecca Thompson et al, Methods **100** (2016), 3-15, doi: 10.1016/j.ymeth.2016.02.017
- *Practical Raman spectroscopy: an introduction*, by Peter Vandenabeele, Wiley.
- *Spectroscopic ellipsometry: principles and applications,* by Fujiwara, Hiroyuki, Wiley.
- *Fundamentals of Nanoscale Film Analysis*, by T. Alford, L. Feldman and J. Mayer, Springer.
- *Scanning Probe Microscopy in Industrial Applications: Nanomechanical Characterization*, ed. Dalia Yablon, Wiley.
- *Introduction to x-ray photoelectron spectroscopy,* by Fred Stevie and Carrie Donley, J Vac Sci Technol A38 (2020) 063204, https://doi.org/10.1116/6.0000412
- *Principles and Applications of Thermal Analysis*, by Paul Gabbott, Wiley.
- *Fundamentals of Powder Diffraction and Structural Characterization of Materials,* by Pecharsky and Zavalij, Springer.
- *Practical Materials Characterization*, ed. M. Sardela, Springer.
- *Materials Characterization*, by Yang Leng, Wiley.

F. Grading Plan:

60% Final assessment (exam and/or project)

30% Homework (approximately 8)

10% In-class participation and attendance.

Grading scale:

A+: 100% points

A: 90% to 99%

A-: 80% to 89%

B+: 70% to 79%

B: 60% to 69%

B-: 50% to 59%

C: 40% to 49%

D: 30% to 39%

F: 29% and below

G. Credit:

3 undergraduate hours or 3 graduate hours

H. Meeting Schedule and Contact Hours:

Two 80-minute online lectures per week