Hands-On Course on Materials Characterization at SBASSE-LUMS

Course Contents

A. FESEM & EDX: Surface Morphology and Composition analysis: Detailed Activities

1. Basic principle of FESEM

(i) Secondary electrons (ii) Backscattered electrons (iii) X-rays (iv) Image acquisition

2. Introduction to hardware and accessories

(i) FEG gun (ii) Column (iii) Chamber (iv) Vacuum, Pneumatics and chiller accessories

3. SEM sample preparation

(i) Mounting of samples (ii) Gold coating (iii) Sample mounting for STEM

4. Samples navigation

5. Software controls and precautions

(i) Detectors (ii) Focusing and astigmation (iii) Normal and high resolution modes (iv) Beam deceleration (v) Charge neutralization (vi) Aperture alignment (vii) Image acquisition and smart scanning (viii) Electron beam energy, spot size and other parameters selection (ix) Plasma cleaning of samples and chamber

7. Imaging with built-in ETD and TLD detector

(i) Imaging with ETD for normal resolutions (ii) Imaging with TLD for high resolutions (iii) Imaging with secondary electrons, backscattered electrons, and charge neutralization (iv) Imaging of polymers, conducting, insulating, magnetic, and thin film samples

8. Imaging with STEM detector

(i) Detector adjustment (ii) STEM modes (iii) Imaging of nanoparticles ranging between 1-20 nm

9. Imaging with low vacuum detector (LVD)

(i) Detector adjustment (ii) Pressure controls (iii) Imaging of biological or botanical samples

10. Imaging with concentric back scattered detector (CBS)

(i) Detector adjustment (ii) Stage bias and beam deceleration (iii) Selection of detector quadrants for acquisition of backscattered electrons of various energies (iv) Imaging of composites

11. Basic principle of EDX for elemental composition analysis

(i) Detector adjustments (ii) Electron beam energy, spot size and other parameters selection for obtaining of sufficient X-ray counts (iii) EDX modes – spot, line and map scans (iv) Aperture adjustment

12. Data analysis & Interpretation

B. Vibrating Sample Magnetometer (VSM): Physical Properties: Detailed Activities

1. Measurement of magnetic moment by vibrating sample magnetometer

a. Introduction to Vibrating Sample Magnetometer (VSM)

b. Magnetic Measurement Types with VSM

i. Room temperature hysteresis (0 to ±7T)

ii. Low temperature hysteresis (0 to ±7T)

iii. ZFC Temperature Scan (4k – 300K)

iv. FC Temperature Scan (300 - 5k)

c. VSM Sample Requirements & Measurement Ranges

d. VSM Functional understanding

e. Cryogen Free VSM Measurement System Overview

f. Sequence for Measurements

g. How to Characterize Magnetic Materials under Applied Magnetic Field and Temperature.

2. AC-Susceptibility (ACS) Probe

a. ACS Measurements.

b. Configuring the ACS Probe

c. Sample Mounting and Loading and Initializing ACS Probe

d. Detection of +ve or –ve Susceptibility Sample

e. How to Characterize Magnetic Materials with ACS under Applied Magnetic Field and

3. Resistivity, Hall effect measurement and magneto-transport

a. Resistivity Measurements

b. The sample holder and conductive epoxy

c. The sample probe electrical connections and wiring.

d. Sample platform, Break-out box Temperature Controller

e. Loading probe and switch to Resistivity Probe

f. Measurements by Van Der Pauw

4. Troubleshooting, Services and Maintenance

C. Magnetron Sputtering – Thin film deposition - Detailed Activities

1. Basic of deposition techniques

(i) CVD, PVD, ALD

(ii) Types of PVD

(iii) Magnetron sputtering

(iv) DC/RF sputtering

2. Introduction to Vacuum pumps

(i) Chamber (main, loadlock)

(ii) Vacuum accessories

(iii) Pneumatics and chiller accessories

(iv) Ultra High Vacuum (UHV)

(v) Plasma (Ar. O2. N2)

3. Substrates/ sample preparation

(i) Mounting of samples on holder

(ii) Coating for insulating/conducting and semiconductor samples

(iii) Coating for magnetic/ non-magnetic samples

(iv) Deposition Rate

4. Magnetic Measurements with MOKE/VSM

D. Photolithography - Microfabrication

1. Introduction to clean room, mask aligner and spin coater set-up
2. Basic Principle
3. Applications
4. Photoresist Coat- Spin Coater
5. Prebaking- Hot Plate
6. Pattern Transfer- Mask Aligner
7. Post Baking (Depends on Photoresist Type)

8. Develop Pattern
9. Inspection under Microscope
10. Trouble shooting and maintenance

E. Atomic Force Microscopy (AFM) - Surface Analysis

Basic principle of AFM

1. Introduction to hardware and accessories
2. Tip loading and alignment
3. Software controls and precautions
4. Image acquisition
5. Data analysis & Interpretation

F. Ellipsometry – Optical Properties Measurements

1. Basic Principle
2. Operation
3. Applications
4. Thickness estimation of thin films
5. Extraction of optical parameters (Refraction, Extinction, Reflectivity, Optical Conductivity, Epsilon)
6. Band gap measurements

G. X-ray Diffraction (XRD) analysis – Structural analysis

1. Basic Principle
2. Operation
3. Applications
4. Introduction to SmartLab SE Rigaku
5. Hardware and Optic Alignments
6. Thin Film/Powder measurements

H. Inductively-Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

1. Introduction to ICP-OES: General Overview
2. Basic Principle and Instrumentation
3. Applications of ICP-OES
4. Sample Preparation and Analysis
5. Data Interpretation
6. Practical Session and Laboratory Work
7. Instrument Setup and Operation

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J. Profilometry – Thickness Measurement

1. Basic Principle
2. Operation
3. Applications
4. Thickness estimation of thin films

K. Nuclear Magnetic Resonance (NMR)

1- Introduction to NMR Instrumentation

- Overview
- Introduction to NMR Spectroscopy
- NMR Instrumentation

2- Practice on Data Analysis with Bruker NMR Software TopSpin

- Introduction to TopSpin Interface
- Basic Data Analysis Techniques
- Advanced Data Analysis Techniques
- Hands-on Practice Sessions

3- Practical Applications and Wrap-Up

- Case Studies and Applications
- Review and Discussion
- Assessment and Feedback

Brief Introduction and Visit to other Experimental facilities

1. Raman Spectroscopy

2. Four Probe Measurements
3. TGA
4. UV-Vis spectrophotometry
5. FTIR, Zeta Sizer etc.