



# Techniques of Materials Characterization

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**INTENDED AUDIENCE :** Final year UG and PG students and PhD research scholars from various disciplines like Materials and Metallurgical Engineering, Ceramic Engineering, Nanoscience and Nanotechnology, Physics, Chemistry, Materials Science etc.

**PRE-REQUISITES :** Any introductory courses on Materials Science and Engineering

**INDUSTRY SUPPORT :** Industries dealing with metal making and processing (e.g. steel or Aluminum industries), semiconductor device making, biomedical applications etc.

## COURSE OUTLINE :

The objective of the course is to provide a broad overview about different techniques available for structural characterization of various materials systems. It is an amalgamation of the science behind these characterization techniques and their application in material systems. The course is divided into two segments dealing with two major aspects of material structures and characterization; initial part will focus on imaging the microstructure by various microscopy techniques while the later part will deal with understanding the internal structure by diffraction phenomena.

For this, the first set of lectures will introduce the fundamental issues of image formation and its inherent attributes and proceed towards details about specific imaging techniques e.g. light/optical microscopy and electron microscopy. Afterwards, the course will cover the basics of diffraction phenomena and related techniques using electron and X-ray sources. At all times, while dealing with these characterization techniques, their importance in materials research and application to real problem solving will be emphasized

## ABOUT INSTRUCTOR :

Prof. Shibayan Roy is currently an Assistant Professor in the Materials Science Center of Indian Institute of Technology IIT-Kharagpur in India. He has joined the institute from November 2015 and continuing till date. Previously, Dr. Roy was a post-doctoral research associate at Materials Science and Technology Division in Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA from November, 2013 to October 2015. Forewords, he worked as a post-doctoral researcher at Institute für Werkstoffwissenschaft und Werkstofftechnik (IWW), Fakultät für Maschinenbau, Chemnitz University of Technology, Chemnitz, Germany from February 2012 to September, 2013. Dr. Roy has obtained his PhD degree from the Department of Materials Engineering, Indian Institute of Science (IISc), Bangalore on November, 2011. Dr. Roy was graduated from Calcutta University with a Bachelor degree (B.Tech) in Ceramic Technology in July 2003. He subsequently completed his Master's at Department of Metallurgical and Materials Engineering (MME), Indian Institute of Technology (IIT Kanpur) between August 2003 to May 2005. He had worked afterwards in National Aerospace Laboratories (NAL), Bangalore as Scientist B from June 2005 to July 2006. Dr. Roy has published several papers in various international journals of scientific repute (including high-impact journals like Acta Materialia, Philosophical Magazine, Materials Science and Engineering A, Metallurgical and Materials Transaction A, Journal of Alloys and Compounds, Materials & Design etc.). He has also authored several book chapters and conference papers. His publication spans from the field of ceramic processing to Biomaterials and finally, to processing-microstructure-texture-property correlation in various metals and alloys. He presently serves as a reviewer to various journals from the field of materials science e.g. Materials Science and Engineering A, Metallurgical and Materials Transaction A, Materials Characterization, Scientific Reports (A Nature series open-access publication) etc.

## COURSE PLAN :

### Week 1:

Introduction to microscopy

- a. Basic principles of image formation
- b. General concepts of microscopy: resolution. Magnification, depth of field, depth of focus etc.

Optical microscopy

- a. Image formation, contrast development
- b. Basic components (light sources, specimen stage, lens system, optical train etc.)

### Week 2:

Various modes of optical microscopy

- a. Bright field mode (transmission vs. reflection)
- b. Contrast enhancing modes (dark field, polarized light, interference contrast, fluorescent microscopy etc.)

**Week 3:**

General concepts of electron microscopy

- a. Basic components of electron microscope (electron gun, electro-magnetic lenses etc.)
- b. Aberrations (chromatic, spherical, astigmatism etc.) and their corrections
- c. Electron-materials interaction (elastic vs. inelastic scattering, coherent vs. incoherent scattering, interaction volume)

**Week 4:**

1. Transmission electron microscopy (TEM)

- a. Image formation and contrast generation (mass-thickness contrast, atomic number contrast, diffraction contrast etc.)
- b. Modes of TEM (bright field, dark field, HAADF, STEM)

2. Electron diffraction in TEM

- a. Scattering of electrons in crystalline material (Braggs law, zone axis, order of diffraction etc.)

**Week 5:**

Electron diffraction in TEM

- a. Concept of reciprocal lattice, Ewald sphere, diffraction from finite crystal
- b. Diffraction pattern (Single crystal vs. polycrystalline diffraction, selected area diffraction etc.)
- c. Indexing of diffraction pattern (camera constant, structure
- d. Application of electron diffraction (DF imaging, dislocation contrast, phase identification etc.)

**Week 6:**

Scanning electron microscopy (SEM)

- a. Working principle in scanning mode
- b. Signal generation: Inelastic scattering (Secondary vs. backscattered electron, Auger electrons, characteristic X-ray emission etc.)

**Week 7:**

Basic components of SEM

- a. Detectors: SE (E-T detector), BSE (scintillator vs. solid state), in-lense detector
- b. Optics of SEM (magnification, pixel, resolution, depth of field)
- c. Resolution in SEM (minimum probe size, beam current etc.)

**Week 8:**

1. Chemical analysis in SEM

- a. EDS and WDS detectors
2. Imaging and contrast generation in SEM
- a. Topographic imaging (in SE & BSE mode)
  - b. Compositional imaging (BSE mode)

**Week 9:**

X-ray production

- a. Electromagnetic radiation, continuous spectrum, characteristic spectrum
- b. X-ray absorption (adsorption edge, excitation voltage, Auger effect etc.), X-ray filters

**Week 10:**

Intensities of diffracted beams

- a. Scattering by single electron (Thomson and Compton scattering)
- b. Scattering by single atom: atomic scattering factor

**Week 11:**

Intensities of diffracted beams

- a. Scattering from unit cell: structure factor calculation for various crystal systems
- b. Multiplicity factor and temperature factor

**Week 12:**

X-ray diffraction profile and analysis

- a. FWHM and line broadening
- b. Crystallite size effect and Scherrer formula
- c. Effect of strain (tensile vs compressive, uniform vs. non-uniform)
- d. Amorphous vs. crystalline materials