

Department of Metallurgical Engineering, NIT Raipur Scanning Electron Facility

Introduction:

One of the primary objectives of National Institute of Technology Raipur is to provide best research and analytical services facilities, keeping in view the technological needs of the country, for all its undergraduate, postgraduate and research activities. Consequently, IIT Delhi has set up several facilities.

Scanning Electron Microscopy laboratory is located on the ground floor in the premises of Department of Metallurgical Engineering. ***All students, researchers and faculty members from various science and engineering departments of the institute are free to avail the services*** as per the guidelines laid by the SEM user Committee of the institute/department. These guidelines are regularly reviewed from time to time.

Whenever it is possible, outside users from Industries, Research & Development Organizations and other educational institutions are also allowed to avail the facility against payment. The charges finalized by the Departmental committee, are displayed on the SEM website of the institute and are non-negotiable under any circumstances.

What is Scanning Electron Microscopy?

The scanning electron microscope uses a focused electron beam which is scanned on the surface of the sample to produce high quality images of the surface topography. SEM essentially offers a very high magnification with very high resolution capabilities and a large depth of focus. This characteristic makes it an indispensable tool for analysis of a wide class of conducting, semi-conducting and insulating materials. A strong beam of electrons called primary electron beam is produced by thermionic emission using either a **Tungsten (W)** or a Lanthanum Hexaboride (LaB6) filament. The primary beam of electrons thus emitted by thermionic emission interacts with the top atomic layers of surface of the sample. This gives out a variety of signals that can be collected and processed to derive a good quality of information about the morphology of the sample, atomic contrast in the sample and the elemental composition of the top surface of the material. The different possible interactions of the sample with a high energy electron beam are:

- Primary electrons generate very low energy electrons called secondary electrons from the top atomic layers of the sample that are used to analyse its topographic nature.
- Primary electrons that are backscattered during interaction with sample surface produce images with a high degree of atomic number contrast.
- Primary beam of electrons can ionize atoms of the sample that stabilize by shell-to-shell transitions of electrons, which causes either emission of X-rays or Auger electron. The X-rays so emitted are characteristic of the elements that make the top surface layers of the sample.

A wide range of materials whose morphology can be routinely analyzed include :

- Metals, Glass and Ceramics
- Semiconductors
- Plastics
- Fibers (Textile, Glass, Asbestos, Natural)
- Powders and Dust

Range of applications:-

- Classification of materials
- Failure and defect analysis
- Examination of surface morphology
- Analysis and identification of surface and airborne contamination
- Powder morphology, particle size and analysis

The SEM Central Facility is equipped with following equipments:

a. ZEISS EVO Series Scanning Electron Microscope Model EVO 18

Zeiss EVO 18 : (Year of Installation January 2014)

The SEM Central Facility at NIT Raipur has recently acquired a new ZEISS EVO Series Scanning Electron Microscope EVO 18. The ZEISS EVO 18 is a versatile analytical microscope with a large specimen chamber. The EVO 18 series can handle large specimens at the analytical working distance of 8.5mm owing to a combination of the inclined detectors and the sharp conical objective lens. The class leading X-ray geometry allows for the addition of an EDS detector.

Essential Specification: ZEISS EVO 18

| | |
|------------|------------------------------------------------|
| Resolution | 3.0nm@ 30kV (SE with W) 4.0nm@30kV VP mode |
|------------|------------------------------------------------|

| | |
|----------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Acceleration Voltage | 0.2 to 30 kV |
| Magnification | 5x to 1,000,000x |
| Field of View | 6 mm at the Analytical Working Distance (AWD) |
| X-ray Analysis | 8.5 mm AWD and 35° take-off angle |
| Available Detectors | <ul style="list-style-type: none"> • SE in HV - Everhart-Thornley • SE in VPSE • BSD in all modes - quadrant semiconductor diode |
| Chamber | 365 mm (Ø) x 255 mm (h) |
| 5-Axes Motorized Compucentric Specimen Stage | <ul style="list-style-type: none"> • X = 125 mm • Y = 125 mm • Z = 50 mm (35 mm motorized) • T = -10° to 90° • R = 360° (continuous) Stage control by mouse or optional joystick and control panel |
| Image Processing | Resolution: Up to 3072 x 2304 pixel Signal acquisition by integrating and averaging |
| Image Display | Single flicker-free XVGA monitor with SEM image displayed at 1024 x 768 pixel |
| System Control | Smart-SEM** with Windows, operated by mouse, keyboard and optional control panel. |

Sample Requirements for SEM:

General Size: Any dimension (Height or Diameter): Less than 10 mm.
 Conductivity (Electrical): Conducting or atleast semiconducting. If sample is not electrically conducting, it will require carbon or gold coating.

For EDX analysis:

If the sample is a powder, make a normal button size pellet. If the sample is insulator or polymeric or electrically non-conducting it needs to be coated with carbon using flash evaporation technique.

However, other sizes can also be accommodated only after a discussion with the system operator.

b. Oxford- Energy Dispersive X-ray system (INCA 250 EDS with X-MAX 20mm Detector)

Microstructures at SEM can be analyzed for its elemental composition in more detail using EDX system. This is a non-destructive analysis and the elements and their concentration in the sample can be determined reasonably accurately. It's operation is free of liquid nitrogen requirement. Low Z elements like Boron, carbon and oxygen can also be routinely detected.

X-Max 20 - Large Area Analytical Silicon Drift EDS Detector with PentaFET® Precision

- Silicon drift sensor –20mm².
- SATW Light Element Window, for detection of elements from beryllium.
- Resolution guaranteed in accordance with ISO15632:2002 for:
- MnK_α (typically 127eV), FK_α (typically 60eV), CK_α (typically 56eV).

Includes Point ID, Analyzer, mapping, mapping navigator, Cobolt Standard.

Further details can be found on: (<http://www.oxford-instruments.com/products/microanalysis/energy-dispersive-x-ray-systems-ds-edx/eds-for-sem/sdd>)

c. Gold and carbon coater (Quorum-SC7620 Sputter Coater)

Quick Overview

The SC7620 is a compact, SEM sputter coater. When combined with the optional carbon attachment SC7620-CF it makes the ideal SEM gold sputtering and carbon coating system package. A panel-mounted switch allows the system to be easily changed between sputter coating and glow discharge modes.

The SC7620 is primarily designed for coating specimens prior to examination in tungsten filament SEMs.