Transmisson Electron Microscopy Laboratory (TEML)

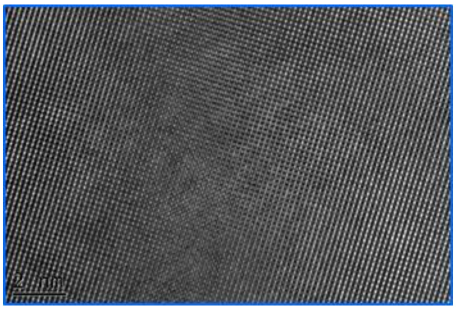
Jeol 2100F High Resolution TEM

Transmission Electron Microscope (TEM) is a unique tool in materials characterization to determine the crystal structures and microstructures of materials by use of simultaneous diffraction and imaging techniques, respectively. In other words, TEM is the only tool to get both crystallographic and morphological information of materials from a region of few nanometers at magnifications up to millions of times.

Central Laboratory has been equipped with Jeol 2100F 200 kV HRTEM (RTEM) to characterize inorganic materials and FEI/Tecnai G^2 Spirit Biotwin 120 kV TEM (CTEM) to examine biological, polymeric and other organic materials.

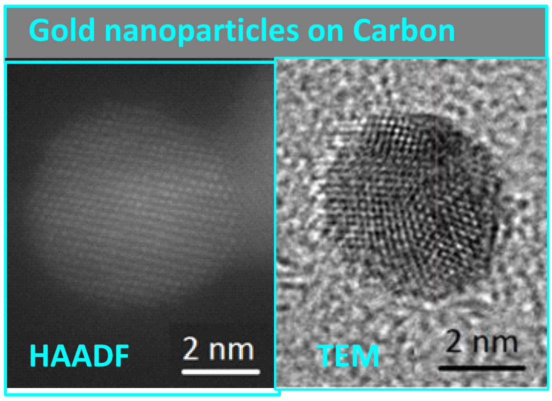
BASIC PRINCIPLES

TEM is based on the principle of sending a coherent electron beam to the thin region at the centre of the specimen and gathering it under the specimen either as the transmitted beam, which passes directly through the specimen, or as the diffracted beams that are scattered from certain crystal planes of the material at Bragg's angles. Bright field and dark field images are so formed and then mostly used to identify the submicron features in the sample and to distinguish second phases. Crystal defects and orientation relationship between different phases can also be determined by TEM.



Lattice fringes in cross grated Gold nanoparticle sample

Lattice imaging is a more advanced technique to observe projection of lattice planes of materials. Scanning Transmission Electron Microscopy (STEM) mode equipped with a High Angle Annular Dark Field (HAADF) detector is used to obtain High Angle Annular Bright Field and High Angle Annular Dark Field images. Since the STEM signal generated at any point on the specimen is detected, amplified and a proportional signal is displayed at an equivalent point on the CRT, it takes several seconds or even minutes to build up the STEM image but the defects in the imaging lenses do not affect the image resolution, which is controlled by the scanning beam only. Selected Area Electron Diffraction is a technique to determine crystal structures of materials. However, Convergent Beam Electron Diffraction is a more sophisticated technique to determine complete crystallographic analysis of materials, i.e. determination of space and point groups.



Electron Energy Loss Spectroscopy (EELS) and EDX are indispensable tools of modern quantitative microscopy for elemental analysis of nanometric regions of the material using the energy distribution of electrons inelastically scattered from the specimen and the characteristic X-ray released when the electron beam interacts with the specimen, respectively. Energy Filtering TEM (EFTEM) employs the energy distribution of inelastically scattered electrons to enhance image contrast.

APPLICATIONS

Polymeric samples:

- Nanofiber
- Nanocomposite
- Nanoparticle

Biologic samples:

- Bacteria
- Tissue
- Cell

Pharmaceutical applications:

- Plant extracts

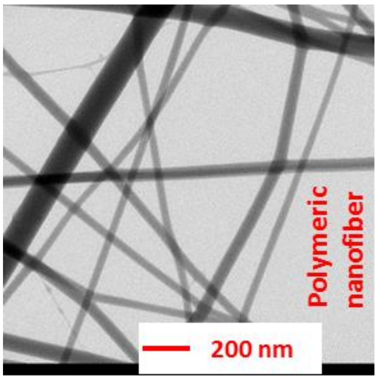
Inorganic samples:

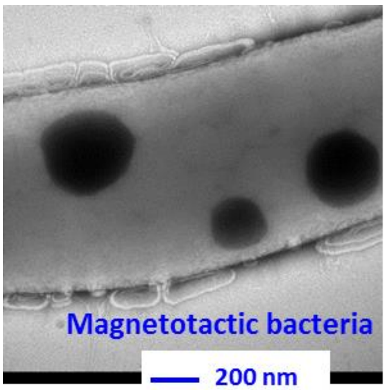
- Catalyst materials
- Mesoporous materials
- Metallic materials
- Ceramic materials
- Jeological materials i.e., minerals, rocks
- Nanotubes, Nanomaterials
- Semiconductive materials
- Thin films

RTEM has resolution of 0.19 nm in TEM mode and 0.14 nm in STEM mode. In addition to conventional Bright Field, Dark Field, Selected Area Electron Diffraction, Lattice imaging techniques, the microscope is accessorized with High Angle Annular Dark Field detector in Scanning TEM (STEM), Convergent Beam Electron Diffraction (CBED), Nano Beam Diffraction (NBD) imaging modes for advanced applications as well as an image filtering system for Electron Energy Loss Spectroscopy (EELS), Energy Filtering TEM imaging (EFTEM) and an Electron Dispersive Spectrometer (EDS) for analytical microscopy.

FEI/Tecnai G^2 Spirit Biotwin High Contrast TEM

CTEM is designed to obtain maximum contrast from low contrast samples by means of special lens configuration and by using absorption contrast mechanism. Resolution of CTEM is 0.34 nm. Operation of CTEM with a fast transition of accelerating voltages between 20 kV to 120 kV is an ideal feature to reduce electron beam damage in biological and polymeric samples. Bright field, dark field and selected area diffraction imaging modes can be used. In addition, the tomography holder makes it possible to take images from the sample with different tilt angles and then images are combined by Xplore 3D programme and it is possible to obtain three dimensional images of samples.





TEM sample preparation methods differ according to the form of the sample i.e., powder, bulk, thin film. Powder samples are first suspended in a volatile medium such as ethanol, acetone, distilled water, etc. and kept in an ultrasonic bath until homogenous dispersion is obtained. Then by use of a micropipet, a 5-10 µL drop is placed on a proper TEM grid and dried overnight. It is not possible to obtain electron transparency criteria unless initial powder size is less than 100nm. On the other hand, metallic bulk samples are sliced into 100 µm thickness by the means of a precise diamond cutter, then 3 mm diameter discs are obtained from those slices by use of a disc punch. Each disc is further decreased to 70-80 µm thickness by using a grinding jig. They are then dimple grinded for ion milling. Final Perforation for Electron Transparency has been carried out either by electropolishing technique or ion beam thinning technique. Bulk polymeric samples which have appropriate dimensions (minimum 5 mm thickness), as specified in the sample acceptance criteria, are examined after obtaining 100 nm thick slices, at room or cryo temperatures, with diamond knife by ultramicrotomy. Biological sample preparation including special steps like sectioning, fixation, dehydration, embedding, and contrasting, which is not carried out at central laboratory. However, if ready, biological samples are always welcomed to examine in CTEM.

Ultramicrotome

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