

Metallurgy for Industries

Power | Petrochemical | Fertilizer | Chemical | Refinery | Engineering | Automobile

A Monthly News Letter

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Scanning Electron Microscopy (SEM):

Principle, Advantages and Applications.

Introduction:

Different advanced techniques of material characterization may be grouped as:

- Techniques based on microscopy principle such as Image Analysis, SEM & TEM
- Techniques based on spectroscopy principle such as XRF and XPS

Scanning electron microscopy is a technique for achieving high resolution images of surfaces. It involves scanning a fine beam of electrons over a specimen and detecting the signals which are emitted.

Electron microscopy techniques were developed to overcome some of the limitations of optical microscope. Optical microscope offers poor depth of penetration due to higher wavelength of light beam (4000 -7000 Å). Not only this, the optical or light microscopes have low max. Magnification limit (up to 2000x) and poor resolution (2500 Å or 250 nm).

SEM uses a high energy electron beam having much shorter wavelength (0.5 eV). The electron beam can penetrate into the sample to a much greater depth than light beam. An important advantage of the SEM over the optical microscope is its high resolution. Resolution of the order of 1 nm is now achievable from an SEM with a field emission (FE) electron gun. Since in a SEM the magnification is governed by the scanning system comprising of electromagnetic coils rather than the glass or plastic lenses, it is possible to obtain a wide range of magnifications from 18x up to 3,00,000x.

One of the unique advantages of SEM is its ability to provide higher depth of field than a light microscope. Depth of field is that property of SEM where surfaces at different distances with respect to the lens simultaneously appear in focus, giving a three-dimensional image. The SEM has more than 300 times the depth of field of the light microscope. This makes SEM an ideal tool for topographic studies (i.e. study of surface) of bulk solid samples including fracture surfaces.

What information can be obtained using a SEM?

Scanning electron microscopy can yield following information:

- **Topography:** Information about the surface features of an object or "how it looks", its texture, etc.
- **Morphology:** Details of the shape and size of the microscopic features such as the presence and location of defects.
- **Composition:** Provides information in terms of the elements and compounds and their relative amounts when coupled with EDS or WDS attachment. The SEM is also capable of determining elemental composition of micro-volumes with the addition of an x-ray or electron spectrometer.
- **Crystallographic Information:** i.e. how the atoms are arranged in the object; direct relation between these arrangements and material properties.

How does a SEM work?

TCR News



- TCR advanced received an approval of RITES for mechanical chemical and metallurgical testing of samples.



- Installed high temperature tensile testing furnace with UTM. Laboratory is now capable of testing high temperature testing up to 500 °C temperature.
- TCR Advanced is preferred service provider for prestigious job of condition assessment and Fitness for service of 33 plant equipment for esteemed customer in Dahej.



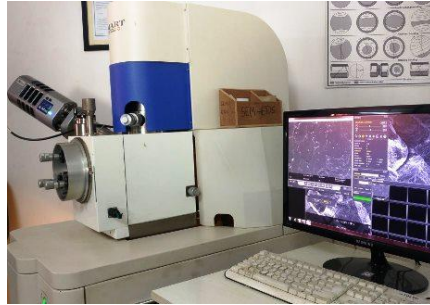
- Conducted course on basics of metallurgy for engineers. The course was attended by the engineers from various metal forming and fabrication industries.



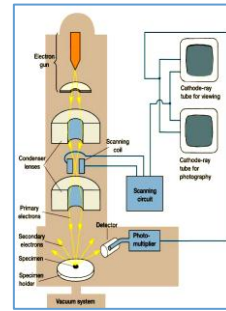
- TCR has started its NDT operations in Bharuch. Mr. R. P. Patel is heading the Office as Vice President- Reliability Engineering. This office will cater for NDT services and Advanced NDT services to customers from nearby industrial zones such as Bharuch, Dahej, Zaghadia, Valia.



As shown in the figure, a typical scanning electron microscope comprises of an electron gun, condenser & objective lenses, scanning coils and imaging system.



Pictorial view of SEM

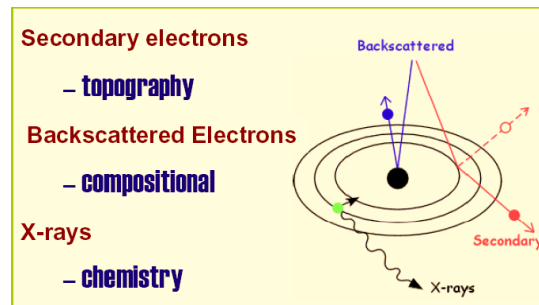


Schematic view of SEM

Electron gun generates an electron beam which is produced from a hairpin type tungsten wire filament, with a diameter of around 100 micrometers. The filament is heated by passing an electrical current through it. Optimum filament temperature for thermionic emission of electrons is around 2700 degrees Kelvin. Electrons emitted by the gun are accelerated towards anode by application of accelerating voltage across anode & cathode, typically of the order of 20-50 kV. Then they are made to pass through a column fitted with condenser and objective lenses, and finally through a set of scan coils and an aperture. Condenser and objective lens focus the beam onto a sample. The diameter of electron beam when it leaves the gun is 25,000 – 50,000° A whereas the average spot size when the beam reaches the specimen surface is 100 - 200° A & for high resolution purpose the spot size is 50° A. An interaction between the beam electrons and the sample atoms emits signals in the form of secondary electrons, backscattered electrons and x-rays. Electrons emitted by the sample are detected by the detectors, amplified and then used to produce an image on the screen. The ability for a SEM to provide a controlled electron beam requires that the electronic column be under vacuum at a pressure of at least 5×10^{-5} Torr. Imaging in the SEM must be carried out under vacuum, as electrons cannot travel through air.

Electron beam sample interaction:

The sample is irradiated by the electron beam and interactions occur inside the irradiated sample, affecting the incident electron beam. These interactions and the resultant effects are detected and transformed into an image.



Effects of electron Beam & Sample Interaction

In SEM visual inspection of the surface of a material utilizes signals of two types, namely secondary and backscattered electrons. Secondary and backscattered electrons are constantly being produced from the surface of the sample while the sample is exposed to the electron beam.

Secondary electrons are a result of the inelastic collision and scattering of incident electrons with specimen electrons. They are generally characterized by possessing energies of less than 50 eV (typically 2-5 eV) and are known as low-energy electrons and are influenced more by surface properties than by atomic number. The depth from which SEs escape the specimen is generally between 5 and 50 nm due to their low energy. They are used to reveal the surface structure of a material with a resolution of ~10 nm or better.

Backscattered electrons are produced as a result of the elastic collision and scattering events between incident electrons and sample nuclei or electrons. Backscattered electrons can be generated further from the surface of the sample (i.e. from the sub-surface) and help to resolve topographical contrast and atomic number contrast with a resolution of >1 micron. The depth from which BSEs are emitted from the specimen is dependent upon the beam energy and the specimen composition. The intensity of the BSE signal is a function of the average atomic number (Z) of the specimen, with heavier elements producing more BSEs.

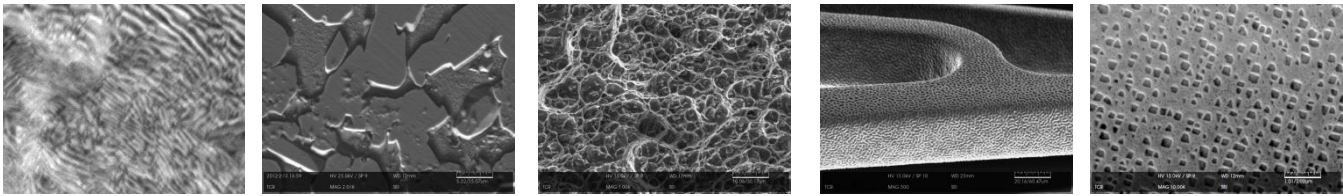
Likewise, the x-ray signal is a result of recombination interactions between free electrons and positive electron holes that are generated within the material.

Thus, these interactions can yield information about the topography (surface features of an object), morphology (shape and size of the particles making up the object), composition (the elements and compounds that the object is composed of and the relative amounts of them) and crystallographic information (how the atoms are arranged in the object).

Applications of SEM:

Electrons in scanning electron microscopy penetrate into the sample within a small depth, so that it is suitable for surface topography, for every kind of samples (metals, ceramics, glass, dust, hair, teeth, bones, minerals, wood, paper, plastics, polymers, etc). It can also be used for analyzing the chemical composition of the sample since the brightness of the image formed by backscattered electrons increases with the atomic number of the elements.

Typical applications of SEM include:



SEM of pearlite phase | Copper microstructure | Ductile fracture surface | Porous Organic coating | Gamma prime in incoloy

- Interpretation of extremely fine grain microstructures. For example, resolution of pearlite in case of alloy steels or carbides in ball bearing steels.
- Single or multi-layer plating / coating such as zinc coating in galvanized steel for evaluation of composition, thickness and continuity.
- Identification of phases and inclusions.
- Fractographic examination for initiation of crack/cracks; cleavage steps in brittle fracture; dimples in ductile fracture; striations in fatigue fracture; mode of crack propagation during fatigue or SCC and study of various types of wear.
- Corrosion related assessment like pitting corrosion, stress corrosion cracking, hydrogen induced cracking, erosion corrosion, etc.
- Powder metallurgical applications such as the determination of size, shape and distribution of metal powder in confined area; change in grain size and porosity at various stages of sintering, etc.
- Analysis of geological samples for identification of minerals and their surface characteristics, morphology of minerals to know origin and type of minerals

The main limitation of SEM is that the sample must be conductive. Hence, non-conductive materials are Gold-coated. Alternatively, they are subjected to sputter coating with C, Cr, or Au-Pd alloy. The carbon tape, carbon paint, Indium foil, or silver tape or paste may also be used to overcome this problem. Secondly, the materials with atomic number smaller than the Boron are not detected with SEM.

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