Scanning Electron Microscope (SEM)

Principle:

Scanning electron microscopy (SEM) is an electron microscopy technique that analyses a visual image of a sample with high-quality and spatial resolution using an electron beam in nanometre scale. It gives information about topography, morphology, composition, and other properties of a sample.

Two types of electrons are detected: backscattered electrons (BSE) and secondary electrons (SE). Backscattered electrons are reflected back after elastic interactions between the beam and sample. Secondary electrons originate from the atoms of the sample and are a result of inelastic interactions between the electron beam and the sample.

Current model:

Figure 1: Hitachi SU-70 Field Emission Gun Scanning Electron Microscope

Video: https://www.youtube.com/watch?v=ljTEG-B-kGc

Scanning Electron Microscope:

The main SEM components include the electron source, (column down which electrons travel with electromagnetic lenses), electron detector, sample chamber, computer and display to view the images.

The Hitachi SU-70 is a high-resolution field emission scanning electron microscope capable of high-resolution imaging (1.0 nm at 15KV). It features several specialised in-lens detectors in addition to STEM (Scanning TEM) and Oxford Instrument EDX/WDX (Energy Dispersive X-ray analysis and Wavelength Dispersive X-ray analysis) capability. It also allows reduced charge-up imaging and low voltage imaging.

The SEM is critical in all fields that require characterisation of solid materials. Most SEMs are comparatively easy to operate, with user-friendly “intuitive” interfaces.

Typical samples:

Metals, polymers, coated components, powders etc can be analysed using the SEM. Specialised sample preparations are required as the SEM runs under vacuum conditions.

SEM Samples must be solid, and fit into the microscope chamber. Maximum size in horizontal dimensions is usually on the order of 10 cm; Vertical dimensions are generally much more limited and rarely exceed 40 mm. For most instruments’ samples must be stable in a vacuum on the order of 10-5 – 10-6 torr.

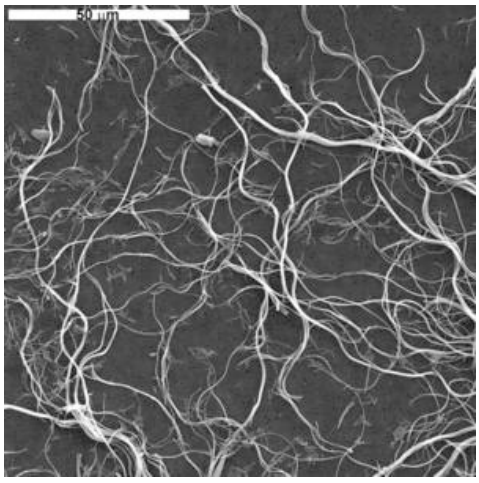

Figure 2: Representative SEM image of Asbestos

Typical Preparation Steps:

- Wet Samples*:* Water must be removed from the samples as water would vaporise in the vacuum.
- Metal Samples*:* Metals are conductive and require no preparation before being used.
- Non-Metal Samples*:* Non-metals need to be made conductive by covering the sample with a thin layer of conductive material. This is done by using a device called a “sputter coater.”

Technical Specifications:

- Schottky (thermal) field emission electron source
- Imaging resolution of 1nm at 15 KV and 1.6nm at 1 kV
- Magnification: Low mag. mode 20x – 2000x; High mag. mode 100x – 800,000x
- Accelerating Voltage: 0.5 kV – 30 kV
- Probe Current: 1 Pa to >200 Na.
- Specimen Stage: x = 110 mm, y = 110 mm, z = 1.5 – 40 mm, tilt =-5 – +70 degrees

- Flexible Detection System utilising: Highly efficient in lens, Lower and Upper
- Secondary Electron Detectors, Low and High Angle Backscattered Electron Detectors and STEM Detector.
- Anti-Contamination Trap “Cold Finger” used to reduce sample contamination.

Standards:

Samples can be assessed in accordance with standards such as: ASTM E986 – 04(2017), ASTM E2809 – 13, ASTM C1723 – 16, ASTM F1372 – 93(2020), ASTM E2142 – 08(2015), ASTM E766 – 14(2019), ISO 21466:2019, ISO 22493:2014

Energy and Wave Dispersive X-Ray Spectroscopy

Principle:

Energy Dispersive X-Ray analysis is an important tool for chemical characterisation and elemental analysis.

During FESEM analysis, the emission of characteristic X-Rays is stimulated by the high-energy electron beam. This X-Ray energy is then converted to a voltage signal by the Silicon Drift Detector. Signal processing generates a spectrum, allowing the identification of elemental constituents in the sample. Quantitative analysis can be performed on clean, polished, flat samples in which peak heights or areas are compared in the unknown with a standard material.

Wave Dispersive X-Ray Spectroscopy differs from EDX in that it uses the diffraction patterns created by light-matter interaction as its raw data. WDX has a much finer spectral resolution than EDX and as such allows spectrum lines to be resolved which are obscured by peak overlaps in EDS. WDX is generally used to detect trace elements (typically <10%).

Technical Specifications:

- Unique single sensor large area SDD sensors
- Up to 50mm² active area (100mm2 sensor size)
- Count rates > 500,000 cps
- Throughput > 200,000 cps
- MnKα guaranteed @ 124eV, CKa guaranteed @ 48eV
- Vacuum enclosed sensor to reduce oxygen absorption
- Only one pulse processing channel required
  Pile up correction software for accurate analysis at high count rates
- WDX:
- Fully focusing spectrometer using 210mm Rowland circle and 2theta range of 33° to 135°.
- Five diffracting crystals on a six position, computer controlled turret, changeable at any position. For analysis of all elements down to Be (Z = 4). Crystals include: LiF. PET, TAP, LSM60N and LSM200.Spectral resolution as low as 2eV making the separation of closely spaced X-ray lines simple
- Excellent detection limits, which are less than 100ppm for many elements
- Accurate quantitative analysis using the XPP matrix correction algorithm
- Proven results on many types of samples, including light elements, and high spatial resolution using low beam currents (<5nA).

SEM Ancillary Equipment

Cressington 208C Carbon Evaporation Unit

Principle:

The Cressington Turbo 208C Carbon Evaporation Coating Unit is a high vacuum coating system designed to evaporate carbon layers onto samples prior to FE-SEM analysis.

Coatings as low as 0.2nm can be applied to samples and the thickness monitor ensures precision when measuring the amount of carbon deposited.

The thickness monitor works on the principle of the quartz crystal microbalance. When evaporated carbon is deposited on the oscillating quartz crystal its frequency is decreased. This change in frequency is then used to calculate the film thickness using the value for carbon density.

Current Model

Technical Specifications:

- Voltage controlled rod source gives multiple evaporation capability.
- Quartz crystal thickness monitor gives reproducible results.
- Thickness Range – 0 – 999.9nm
- 80 l/sec turbo pump on a 150mm chamber gives very rapid pumpdown.
- 150mm Ø (5.9″) chamber size
- Variable height – 165mm – 250mm
- Sample Stage – Static table holds 12 SEM ½” stubs, optional rotary-planetary-tilt stage
- Digital voltage setting, 0.1 – 5.5V
- Pumpdown time – 1.5 min. to 1×10-4mb

Cressington 208HR Sputter Coater

Principle:

The Cressington 208HR Sputter Coating Unit is a high vacuum coating system designed to sputter a range of metals onto samples prior to FESEM imaging.

As coatings can interact with different sample surfaces in different ways, the apparent grain size of the coating can vary between samples. In order to minimise this issue, a range of metals and alloys can be used depending on sample type. Imaging of non-conducting materials requires coating the sample in Au/Pd or Pt/ Pd, whilst semi-conducting samples can be sputtered with a very thin Cr layer.

Thin layers require a long working distance and thick layers a long working distance, therefore the 208HR coating unit comes with varying glass heights to facilitate working distance requirements. Also, the precise control of both conformity and uniformity of the coating can be regulated due to the wide range of operating pressures available as well as correct utilisation of the thickness controller.

Current model:

Technical Specifications:

- Wide choice of coating materials – Au/Pd, Pt/Pd and Cr (quick target change)
- Precision thickness control – thickness optimised to FESEM operating voltage
- Multiple sample stage movements – separate rotary, planetary and tilting movements allow optimised coating thickness and coverage, variable speed rotation.
- Variable Chamber Geometry:-Chamber geometry is used to adjust deposition rates from 1.0 nm/sec to 0.002nm/sec to optimize structure.
- Chamber Size – 150mm
- Variable Chamber height – 165mm – 250mm
- Availability of four sample holders
- Thickness Range – 0nm – 999.9nm

- Pumping System – Turbo-drag/rotary pump combination – Optional diaphragm pump instead of rotary pump
- Pumpdown time – 1 min. to 1×10-3mb

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