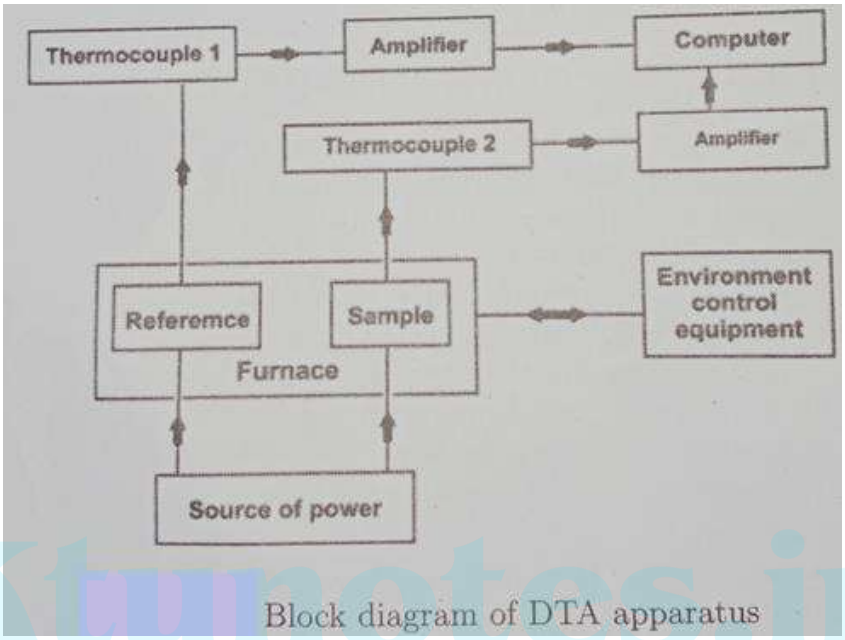
Instrumentation

- The instrument used to measure DTA is DTA apparatus.
- In DTA, the material under study and reference material are heated under identical conditions at a constant rate.
- Two thermocouples T_1 & T_2 record the temperature of the sample and the reference
- The data is amplified and recorded.(processed in a computer)



Block diagram of DTA apparatus

Applications

1) It gives information such as M. P, transition temperature etc. of a crystalline substance
2) Distinguish between endothermic &exothermic process
3) Estimation of enthalpy change (ΔH) by counting peak area
4) Study of decomposition temperature of inorganic solids
5) Characterization of polymers- based on the measurement of properties such as glass transition temperature, M. P, decomposition etc.
6) DTA is widely used in the pharmaceutical, food industries, cement chemistry, mineralogical research, environmental studies and study of archaeological materials

DTA of **Calcium oxalate monohydrate ($CaC_2O_4.H_2O$)**

Magnetic nano-composites are used as ferrofluids (It is a liquid that becomes strongly magnetized in the presence of a magnetic field), high density information storage and magnetic refrigeration.

1) Nanostructured metal-oxide thin films are used as gas sensors (for CO, CO_2 etc)
2) Nano semiconductors are used as window layers in solar cells.
3) Carbon nanotubes based transistors used for miniaturizing elctronic devices.
4) Carbon nanotubes are used for making paper batteries.
5) A mixture of carbon nanotubes and fullerenes is used for making solar cells.
6) Nano medicine is the medical application of nanotechnology where nanoparticles based treatment are used for tumors.
7) Nano-cadmium-telluride exhibit different colour depending upon its size. It can be used for dyeing fabrics such as nano colorants never fades.

SEM (Scanning Electron Microscope)

It is a powerful tool for the surface characterization of materials. It is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. This examination can provide information about the surface topography (surface features), morphology(shape &size of the particles) composition and crystallography

Principle

The electrons interact with atoms in the sample, producing various signals that can be used to provide information about the surface topography and composition of the sample. The electrons which are produced at the top of the column are accelerated down and passed through a combination of lenses and apertures to produce a focused beam of electrons which hits the surface of the sample. As a result of electron- sample interaction, a no. of signals are produced. These signals are the detected by appropriate detectors.SEM produces a black and white 3D images

Instrumentation

SEM consists of the following components

1) **Electron Source ("Electron Gun**"):-It is the source of electron (eg:Tungsten wire). It generates a beam of energetic electrons down the column and onto a series of electromagnetic lenses. These lenses are tubes wrapped in coil and referred to as solenoids
2) **Condenser Lens**:-compresses the electrons to a narrow beam
3) **Aperture:**- It controls the diameter of the electron beam
4) **Objective lens**:- It focuses the electron beam to the sample
5) **Sample chamber & Stage**:-This chamber keeps the sample.Most samples require some preparation before being placed in the vacuum chamber. Two most commonly ued preparations are
    (i) Sputter coating for nonconductive samples
    (ii) Dehydration of biological specimens
6) **Detectors**:-To detect the signals
7) **Amplifier**:-To amplify the signals

8) **Display / Data output devices**:-To show the SEM image obtained

Procedure

- When the incident electrons interact with the sample, energetic electrons are released from the surface.
- The scatter pattern made interaction gives information about size, shape and composition of the sample.
-A variety of detectors are used to attract different types of scattered electrons including secondary (SE), back scattered electrons (BSE) and X-rays.
- BSE are incidental electrons reflected backwards. This comes from the deeper regions of the sample.
- SE originates from the surface of the sample.
- X-rays emitted from beneath the sample surface can provide element and mineral information.
- SEM produces a black and white 3D images.

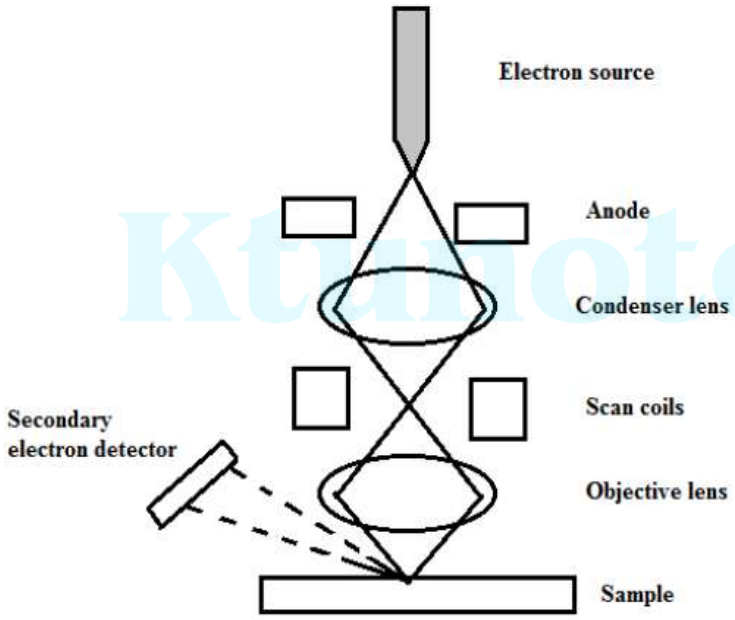


Figure 3.13: Block diagram of SEM

Advantages

- It gives 3D topographical images.
- Instrument is very fast & easy to operate
- Data is available in digital form

Disadvantages

- Instrument is expensive & large
- Special training is required to operate the machine
- Risk of radiation associated with electrons

Applications

SEM is used

1) As very essential research tool in fields such as life science, nano science, gemology, medical &forensic science &metallurgy
2) SEMs have practical industrial &technological applications such as semiconductor inspection, production line of miniscale products &assembly of microchips for computers
3) To characterize nanowires & their gas sensing behavior
4) In material science for research, quality control etc.
5) It helps in the characterization of solid materials
6) It can detect &analyze surface fractures surface contamination &provide information of micro structure