6.4.4 PARTICLE SIZE DETERMINATION

There are various methods for determining particle sizes in different structures. ...anoparticles dispersed in a matrix, the distance between them are greater than ...ir average diameters or dimensions. The size of a micrometer-sized grain can be ...ermined by looking at it in a microscope. The size of a nanosized particle is ...ermined with the help of a transmission electron microscope (TEM).

The manner in which particles scatter light provides a means of determining ...ir sizes. The scattering amplitude is a function of relationship between particle ...e d and wavelength λ of light and also of the polarization of the incident light ...ve. The light reflected from the sky during the day appears blue whereas that ...ansmitted by the atmosphere at sunrise and sunset appears red. This is because ...e scattering cross-section σ is proportional to $\frac{1}{\lambda^4}$. So blue colour of light is ...attered most whereas the scattering for red colour is least.

Particle size is determined by using a monochromatic laser beam scattered at a ...ticular angle (usually 90°) for parallel and perpendicular polarizations. The ...attered intensities can give information regarding particle size, particle ...centration and the refractive index. For particles with size $d < 0.1\lambda$, size of ...noparticle is measured with optical wavelengths. The method of laser beam ...noparticle determination is applicable for nanoparticle with diameters greater than 2 nm.

The method of **mass spectrometry** is used for particle size determination for ...rticles with diameters less than 2 nm. The schematic diagram of gas mass ...ectrometer is shown in fig. 6.3.

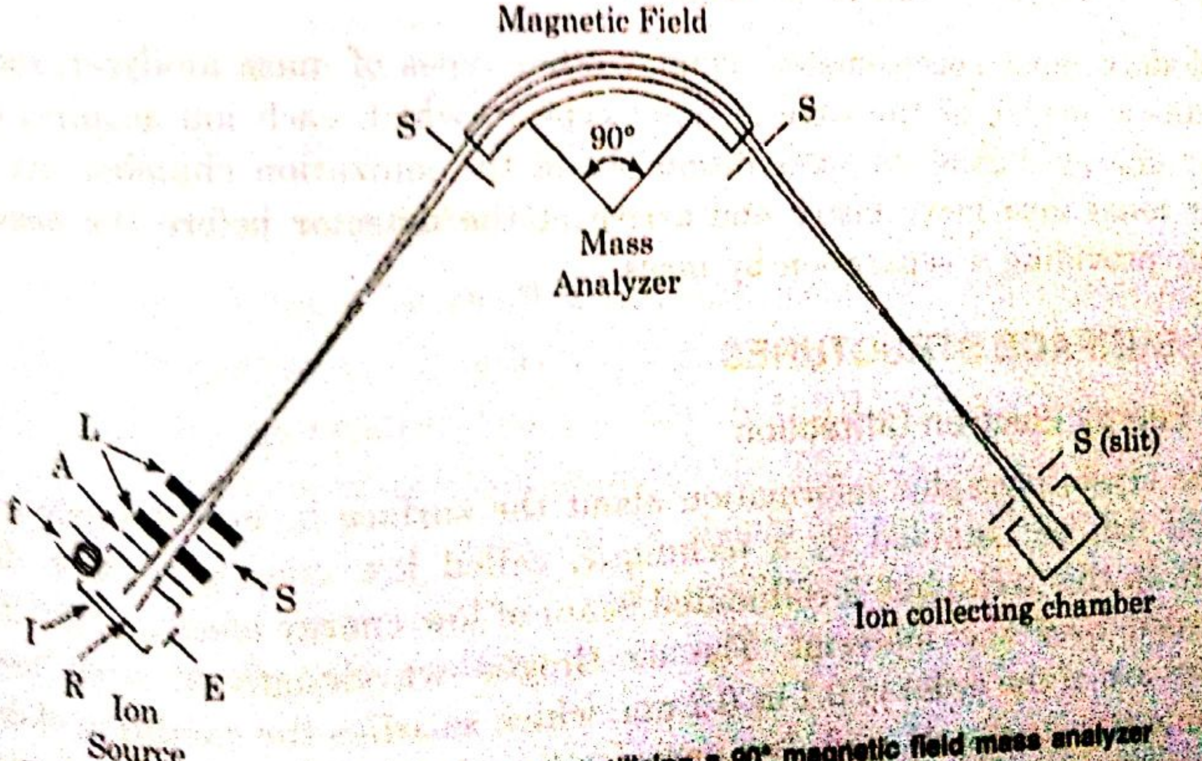

Fig. 6.3. Sketch of a mass spectrometer utilizing a 90° magnetic field mass analyzer

In it, the nanoparticles are ionized to form positive ions by the impact of ...ectron emitted from the heated filament in the ionization chamber of the ion source. These ions are accelerated through a voltage V between the repeller (R) and accelerator (A) plates. After it, they are focused by lenses L and then collimated by slits S during their transit to the mass analyzer. The magnetic field B, oriented ... the page exerts a magnetic force $F = qvB$ and bends the ion beam through an angle of 90° at the radius r, after with they are detected at the ion collector.

The necessary centripetal force $\left(\frac{mv^2}{r}\right)$ is provided by the magnetic force (qvB).

On balancing these forces, we get

$$\frac{mv^2}{r} = qvB$$

or

$$2m \times \frac{1}{2} mv^2 = q^2 B^2 r^2$$

or

$$2m \times qV = q^2 B^2 r^2$$

or

$$\frac{m}{q} = \frac{B^2 r^2}{2V}$$

which gives the mass to charge ratio of the particles (ions).

In a particular instrument, r is fixed. So either magnetic field B or accelerating voltage V can be scanned to focus the ions of different masses at the detector. Since ... is generally known for nanosized ion, therefore m can be determined. Further, the material forming the nanoparticle is known, thus its density $\rho = m/V$ is known. Here V represents the volume of the material. Therefore its size or linear dimension $d = (V)^{1/3} = (m/\rho)^{1/3}$ can be determined.

Modern mass spectrometers employ other types of mass analyzer, such as the quadrupole model, or the time of flight type in which each ion acquires the same kinetic energy during its acceleration out of the ionization chamber. As a result, lighter mass ions move faster and arrive at the detector before the heavier ions, thereby providing a separation by mass.

➡ 6.5. SURFACE STRUCTURES

I. Low Energy Electron Diffraction

The crystallographic information about the surface layers of single-crystalline material can be obtained by a technique, called low energy electron diffraction (LEED). In this technique a collimated beam of low energy electrons (20-200 eV) is bombarded over the material. The de Broglie wavelength of such low energy electrons are of the order of 0.1 or 0.2 nm, which satisfies the condition of diffraction on atomic structures. At such low energies the electrons penetrate only very short distances into the surface, so their diffraction pattern reflects the atomic positions in the surface layer. Diffracted electrons are seen as spots on fluorescent screen. The intensities of diffracted beams are recorded as a function of incident electron beam energy to generate I-V curves. These curves are then compared with theoretical curves to provide information on atomic positions on the surface in question.

If the diffraction pattern arises from more than one surface layer, the contribution of lower lying crystallographic planes will be weaker in intensity. The electron beam behaves like a wave and is therefore reflected from crystallographic planes in the same manner as an X-ray beam. The electron de-Broglie wavelength is given by

$$\lambda = \frac{1.226 \times 10^{-9}}{\sqrt{E}} \text{m} = \frac{1.226}{\sqrt{E}} \text{nm}$$

where E is the energy of electrons in units of electron volt. For example an electron energy of 25.2 eV corresponds to a de Broglie wavelength of 0.2442 nm. This wavelength is comparable with the GaAs bond distance in gallium arsenide $\left(\frac{a\sqrt{3}}{4} = 0.2442 \text{ nm}\right)$, with lattice constant $a = 0.565$ nm. Thus low energy electron diffraction experiments are capable of providing accurate information about surface structure of materials. In this method the sample itself must be a single crystal with a well ordered surface structure in order to generate a back scattered electron diffraction pattern

II Refl... Electron Diffraction