Scanning Electron Microscope- Fundamental and Applications

Diffraction for Electron, Neutron and X-ray and Material Science

The condition of Diffraction is, size of obstacle is in order of wavelength of incident radiation. Electron and neutron both behave like wave and operate on the same principle as X-ray diffraction but the three techniques differ from each other in some aspects, particularly in the mechanism of scattering leading to the formation of diffracted beams. These makes them applicable in numerous applications. For example, neutron diffraction is suitable for studying magnetic properties of solids materials that cannot be investigated by electron and X-ray diffraction techniques. However, neutron sources are uncommon and expensive, therefore discussion here is limited to electron diffraction. The wavelength of a beam of electrons is a function of the accelerating voltage in an electron gun and is given by(kinetic energy=1/2mu^2 =eV)

$$\lambda = \sqrt{\frac{150}{V}}$$

Where λ is in Å and V is in volts. For V = 10,000 volts, λ = 0.12 Å and for V = 40,000volts, λ = 0.06 Å. Thus the electron diffraction wavelengths are much smaller compared to X-ray diffraction wavelengths which are in the range 0.7 – 2.2 Å. An electron diffraction pattern is therefore confined to very small θ values. The atomic scattering factors (*f)* for electrons and X-rays are connected as

$$f_{ele} = (Z - f_{x-rays})\frac{\lambda^2}{\sin^2\theta}$$

where Z is the atomic number of target element. It can be seen that $f_{ele} \approx 10^3\ f_{x\text{-}ray}$ and the corresponding line/spot intensities in diffraction patterns of X-rays and electrons are in the proportion of $\approx 1{:}10^6$. Therefore, in order to obtain the same measurable intensity of diffraction, the size of the specimen must be varied according to the radiation used. If the linear dimension of the specimen for X-ray examination is 1mm, it can be about 10^{-5} mm for electron diffraction. And so, is therefore ideally suited for study of nanostructure , ceramics, oxidation products, etc, which is not possible or difficult by X-ray diffraction techniques. The electron diffraction pattern consists of spots or rings similar to X-ray diffraction pattern and can be indexed in a similar way by using the equation: L λ = r*d*; where L λ is a camera constant, r is the distance between centre of the diffraction pattern and the diffraction spot or ring radius.

The geometry is identical for X-ray and electron diffraction which is depicted in Fig. 1 from which the Bragg law can be easily derived.

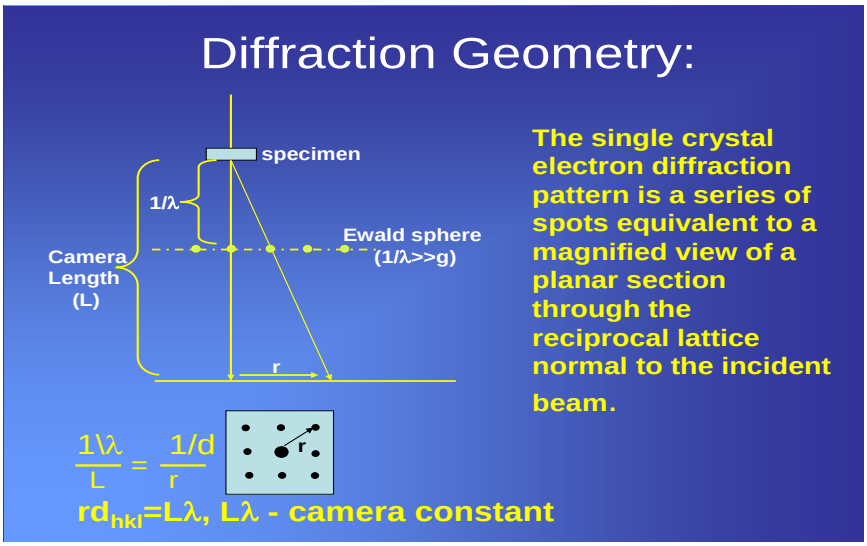

Fig.1: Geometry of X-ray and Electron Diffraction

Scanning Electron Microscopy and Surface Science

The scanning electron microscope (SEM) in which images are obtained by scanning with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's morphology, grain size .

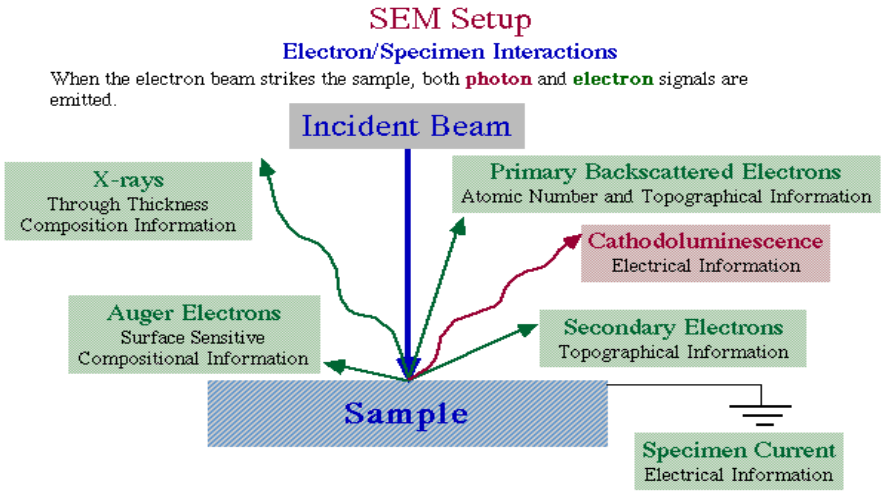

Fig.2 (a): SEM set up (above) (b) Equipment (below)

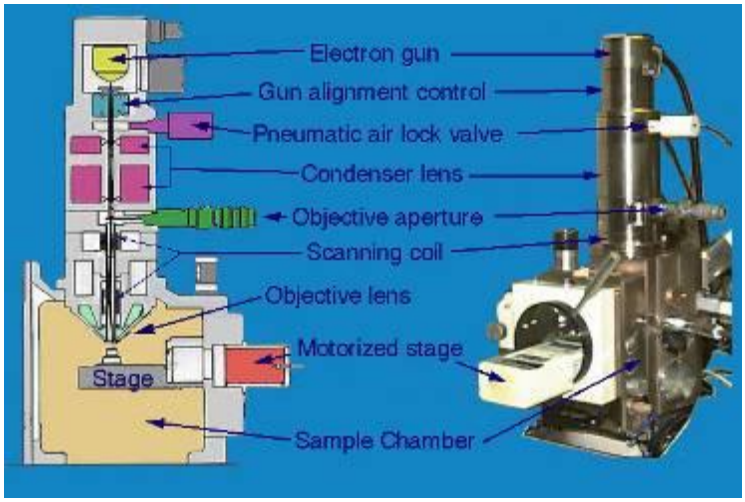

Fig-2(b)

The electron beam coming from electron gun source hits the materials, producing secondary electrons, which are electrons are collected by a secondary detector or a backscatter detector, converted to a signal voltage, and amplified. The amplified voltage is applied to the grid of the CRT and causes the intensity of the spot of light to change. The image consists of thousands of spots of varying intensity on the screen of that correspond to the topography of the materials. The types of signals produced by SEM include secondary electrons, back-scattered electrons (BSE), characteristic X-rays, light (Cathodo luminiscence source). The signals result from interactions of the electron beam with atoms at or near the surface of the sample. In the most common or standard detection mode, secondary electron imaging, SEM can produce very high-resolution images of a sample surface, revealing details about less than 1 to 5 nm in size. Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample..

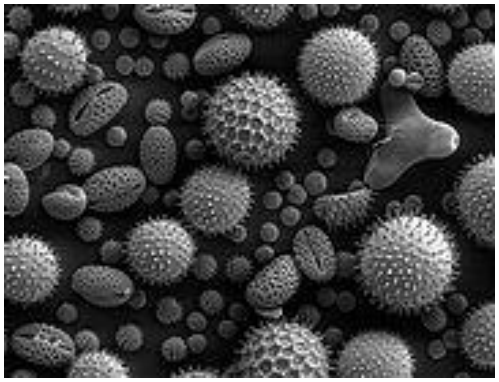

Fig.3: SEM picture of pollen grains show the characteristic depth of field of SEM micrographs

This is exemplified by the micrograph of pollen shown in the Fig.3. A wide range of magnifications is possible, from about 10 times to more than 500,000 times, about 250 times the magnification limit of the best light microscopes

Back Scattered Electron

Back-scattered electrons (BSE) are beam electrons that are reflected from the Materials by elastic scattering and are often used in analytical SEM along with the spectra made from the characteristic X-rays. Because the intensity of the BSE signal is strongly related to the atomic number (*Z*) of the specimen . Characteristic X-rays are emitted when the electron beam removes an inner shell electron from the sample(K-L-M shell) causing a higher energy electron to fill the shell and release energy. These characteristic X-rays are used to identify the composition and measure the abundance of elements in the sample, as already described under EDS(elemental detection through spectroscopy). The EDS associated with SEM can determine elemental composition.

Sample preparation for SEM

Materials must also be of an appropriate size to fit in the specimen chamber and are generally mounted rigidly on a specimen holder called a specimen stub. For conventional imaging in the SEM, specimens must be electrically conductive, at least at the surface, and electrically grounded to prevent the accumulation of electrostatic charge at the surface. Metal objects require little special preparation for SEM except for cleaning and mounting on a specimen stub. Nonconductive specimens tend to charge when scanned by the electron beam, and especially in secondary electron imaging mode, this causes scanning faults and other image artifacts. They are therefore usually coated with an ultrathin coating of electrically-conducting material, commonly gold, deposited on the sample either by low vacuum sputter coating or by high vacuum evaporation. Conductive materials in current use for specimen coating include gold, gold/palladium alloy, platinum, osmium, iridium, tungsten, chromium and graphite [5-7]. Coating prevents the accumulation of static electric charge on the specimen during electron irradiation. Non conducting specimens may be imaged uncoated using specialized SEM instrumentation such as the "Environmental SEM" (ESEM) or field emission gun (FEG) SEMs operated at low voltage. Environmental SEM instruments place the specimen in a relatively high pressure chamber where the working distance is short and the electron optical column is differentially pumped to keep the vacuum adequately low at the electron gun..

The high pressure region around the sample in the ESEM neutralizes charge and provides an amplification of the secondary electron signal. Low voltage SEM of non-conducting specimens can be operationally difficult to accomplish in a conventional SEM and is typically a research application for specimens that are sensitive to the process of applying conductive coatings. Operating conditions must be adjusted such that the local space charge is at or near neutral with adequate low voltage secondary electrons being available to neutralize any positively charged surface sites. Embedding the sample in a resin with further polishing to a mirror-like finish can be used for both biological and materials specimens when imaging in backscattered electrons or when doing quantitative X-ray microanalysis.

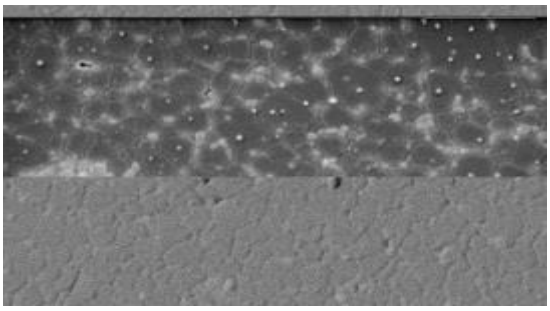

Fig 4.: Comparison of SEM techniques: **Top**: backscattered electron analysis composition; **Bottom**: secondary electron analysis – topography

Backscattered electrons can also be used to form an Electron Back Scatter Diffraction (EBSD) image that can be used to determine the crystallographic structure of the specimen.

Case studies

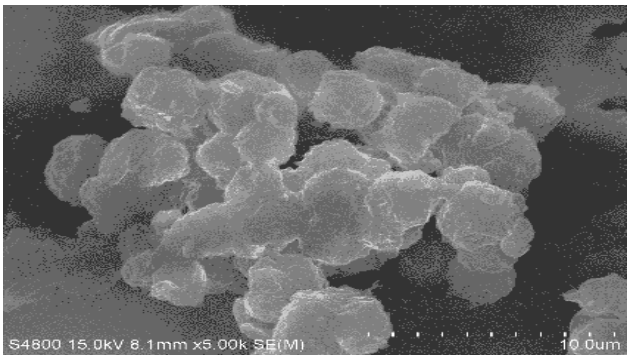

Fig-5 SEM micrographs of *Lauh Bhasma* at different magnifications (a) 5000

Ref- Rakesh Kumar Singh et.al., Crystal Structure and Magnetic Property Studies on Nanocrystalline Lauh (Iron) Bhasma-An AyurvedicMedicine, Int. J. Ayu. Alt. Med., 2016; 4(1):17-23

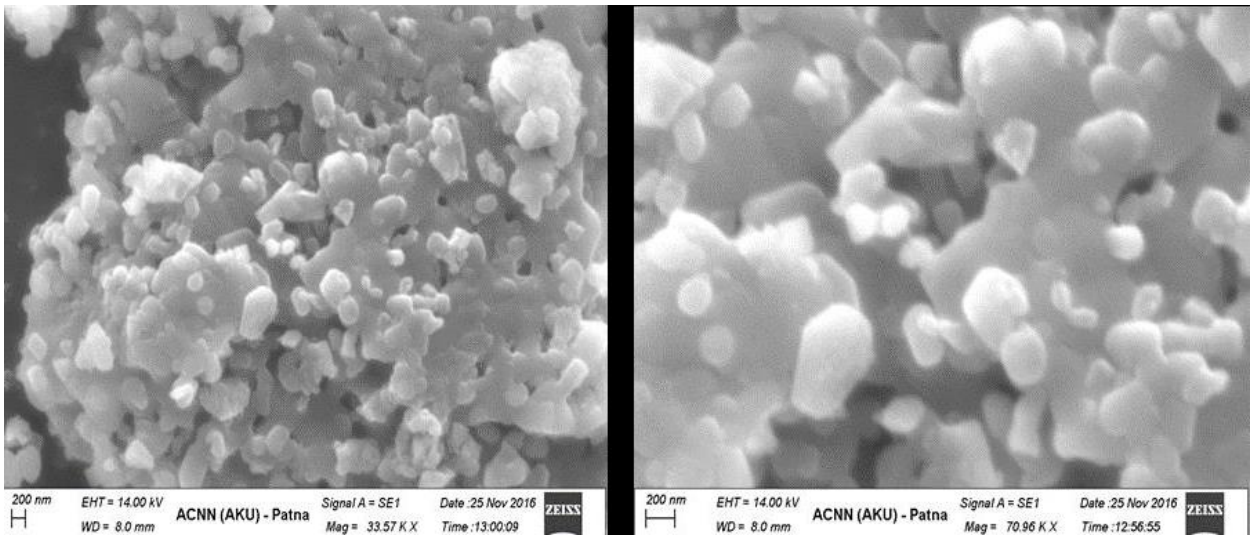

Fig6- SEM micrographs of Abhrakh bhasma as nanomedicine in different magnifications

SEM measurement shows that the microstructure is uniform which must in contest is as concern for the disease treatment i.e. the action of medicine should be uniform for better action. The present study reveals that the present method of bhasma preparation can be used as a standard method to produce uniform particle size of Abhrakh bhasma for better use in disease treatment

Ref- R.K. Singh et al. / GSC Biological and Pharmaceutical Sciences 2018, 05(02), 041–047

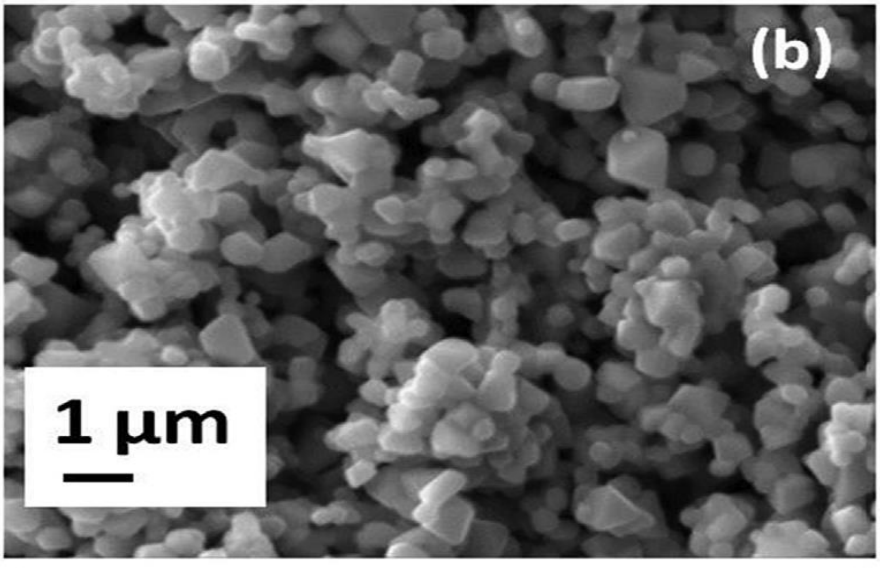

Fig-7- Scanning electron micrograph of ferrite pellet showing porous microstructure in Hydroelectric cell

Ref- R.K.Kotnala et al. Int. j. hydrogen energy.42(2017) .

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More detail, we discuss in counselling classes

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