Chapter 4

Nanostructure Characterization Techniques

4.1: Scanning Electron Microscopy (SEM)

Introduction

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topography and composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, in low vacuum, in wet conditions (in environmental SEM), and at a wide range of cryogenic or elevated temperatures.

Types of signals produced by SEM

The types of signals produced by a SEM include secondary electrons (SE), backscattered Electrons (BSE), characteristic Xrays, and light (cathodoluminescence) (CL), but it is rare that a single machine would have detectors for all possible signals.

Secondary Electrons

The most common SEM mode is detection of secondary electrons emitted by atoms excited by the electron beam The number of secondary electrons depends on the angle at which beam meets surface of specimen, i.e. on specimen topography. By scanning the sample and collecting the secondary electrons with a special detector, an image displaying the topography of the surface is created.

Bacscatter Electrons

Backscattered electrons (BSE) are high energy electrons that are reflected from the sample by elastic scattering. The intensity of the BSE signal is strongly related to the atomic number (Z) of the specimen. Since heavy elements (high atomic number) backscatter electrons more strongly than light elements (low atomic number), and thus appear brighter in the image, BSE are used to detect contrast between areas with different chemical compositions. So BSE images can provide information about the distribution of different elements in the sample.

Characteristics X-rays

Characteristic Xrays are emitted when the electron beam removes an inner shell electron from the sample, causing a high erenergy 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.

Construction & Working Mechanism (Scanning process and image formation)

In a typical SEM, an electron beam is thermionically emitted from an electron gun fitted with a tungsten filament cathode. Tungsten is normally used in thermionic electron guns because it has the highest melting point and lowest vapour pressure of all metals, thereby allowing it to be heated for electron emission, and because of its low cost.

The electron beam, which typically has an energy ranging from 0.2 keV to 40 keV, is focused by one or two condenser lenses to a spot about 0.4 nm to 5 nm in diameter. The beam passes through pairs of scanning coils or pairs of deflector plates in the electron column, typically in the final lens, which deflect the beam in the *x* and *y* axes so that it scans in a raster fashion over a rectangular area of the sample surface.

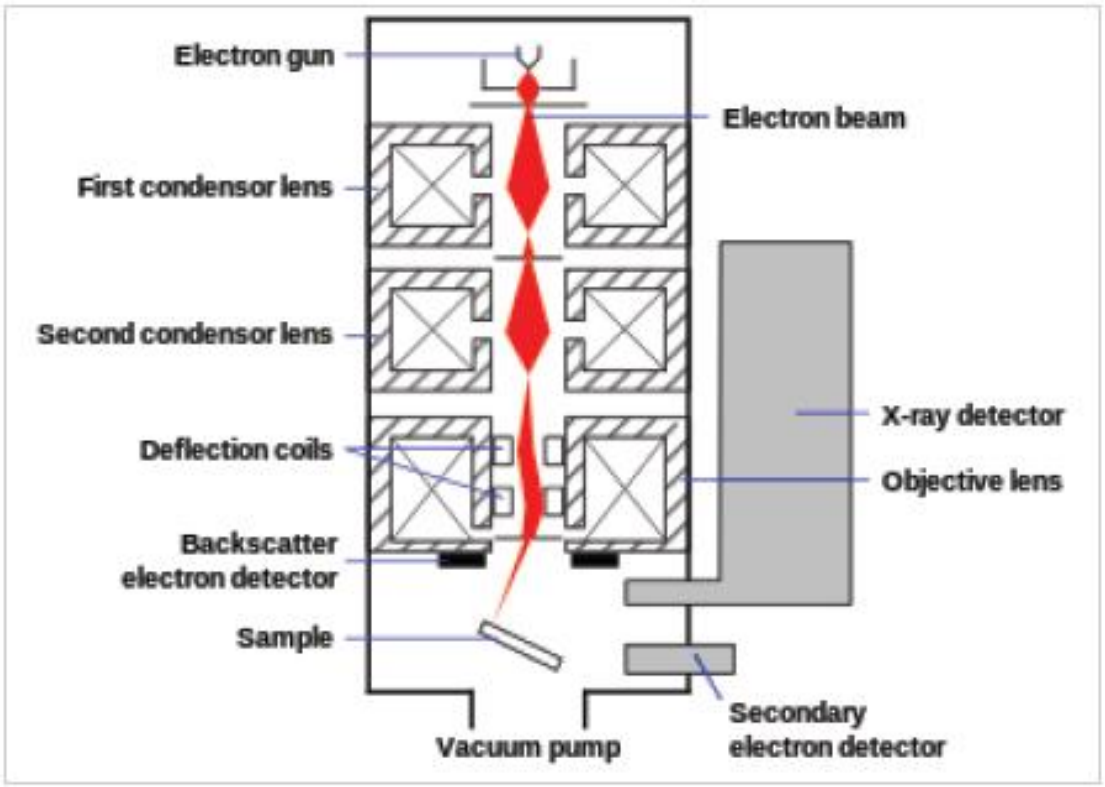

Fig. 4.1 (a) Schematics of Scanning electron microscope setup

When the primary electron beam interacts with the sample, the electrons lose energy by repeated random scattering and absorption within a tear drop shaped volume of the specimen known as the interaction volume, which extends from less than 100 nm to approximately 5 μm into the surface. The size of the interaction volume depends on the electron's landing energy, the atomic number of the specimen and the specimen's density.

The energy exchange between the electron beam and the sample results in the reflection of high energy electrons by **elastic scattering**, emission of secondary electrons by **inelastic scattering** and the emission of electromagnetic radiation, each of which can be detected by specialized detectors.

The most common imaging mode collects low energy (<50 eV) secondary electrons that are ejected from the k shell of the specimen atoms by inelastic scattering interactions with beam electrons. Due to their low energy, these electrons originate within a few nanometers from the sample surface. The electrons are detected by an Everhart Thornley detector, which is a type of scintillator photomultiplier system. The secondary electrons are first collected by attracting them towards an electrically biased grid at about +400 V, and then further accelerated towards a phosphor or scintillator positively biased to about +2,000 V. The accelerated secondary electrons are now sufficiently energetic to cause the scintillator to emit flashes of light (cathode luminescence), which are conducted to a photomultiplier outside the SEM column via a light pipe and a window in the wall of the specimen chamber. Electronic amplifiers of various types are used to amplify the signals, which are displayed as variations in brightness on a computer monitor (or, on a cathode ray tube). Each pixel of computer video memory is synchronized with the position of the beam on the specimen in the microscope, and the resulting image is therefore a distribution map of the intensity of the signal being emitted from the scanned area of the specimen.

The brightness of the signal depends on the number of secondary electrons reaching the detector. If the beam enters the sample perpendicular to the surface, then the activated region is uniform about the axis of the beam and a certain number of electrons "escape" from within the sample. As the angle of incidence increases, the "escape" distance of one side of the beam will decrease, and more secondary electrons will be emitted. Thus steep surfaces and edges tend to be brighter than flat surfaces, which results in images with a well defined, threedimensional appearance. Using the signal of secondary electrons image resolution less than 0.5 nm is possible.

In older microscopes image may be captured by photography from a high resolution cathode ray tube, but in modern machines image is saved to a computer data storage.

Magnification

Magnification in a SEM can be controlled over a range of up to 6 orders of magnitude from about 10 to 500,000 times. Unlike optical and transmission electron microscopes, image magnification in the SEM is not a function of the power of the objective lens. SEM's may have condenser and objective lenses, but their function is to focus the beam to a spot, and not to image the specimen. Provided the electron gun can generate a beam with sufficiently small diameter, a SEM could in principle work entirely without condenser or objective lenses, although it might not be very versatile or achieve very high resolution.

In a SEM, as in scanning probe microscopy, magnification results from the ratio of the raster scan size on the display device to the the raster scan size on the specimen. Assuming that the display screen has a fixed size, higher magnification results from reducing the size of the raster on the specimen, and vice versa. Magnification is therefore controlled by the current supplied to the x, y scanning coils, or the voltage supplied to the x, y deflector plates, and not by objective lens power.

Resolution of the SEM

The spatial resolution of the SEM depends on the size (diameter) of the electron spot, which in turn depends on both the wavelength of the electrons and the electron-optical system that

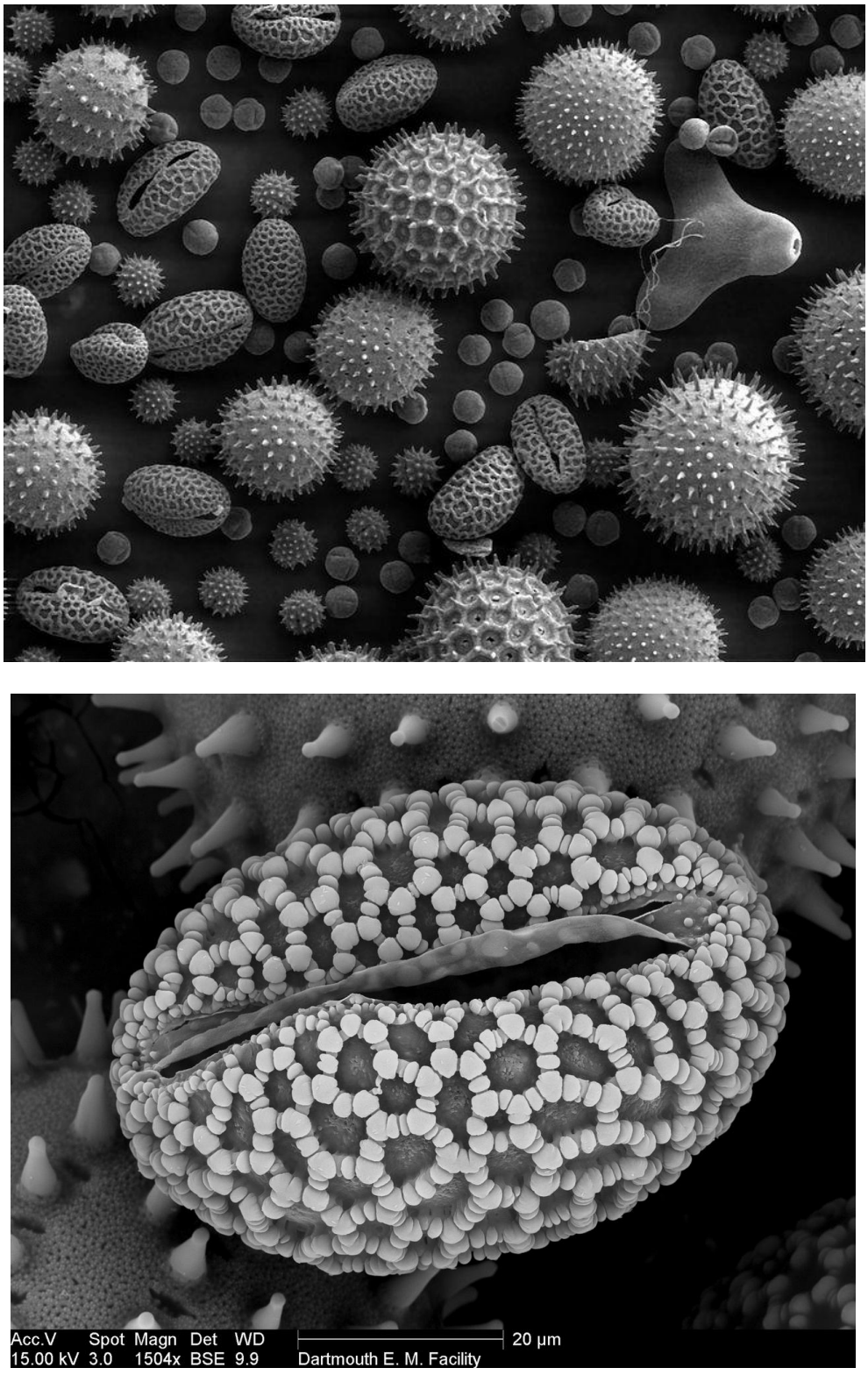

Fig. 4.1 (b): Scanning electron microscope images of pollens

produces the scanning beam. The resolution is also limited by the size of the interaction volume, the volume of specimen material that interacts with the electron beam. The spot size and the interaction volume are both large compared to the distances between atoms, so the

resolution of the SEM is not high enough to image individual atoms, as is possible with transmission electron microscope (TEM).

The SEM has some compensating advantages, including the ability to image a comparatively large area of the specimen; the ability to image bulk materials (not just thin films or foils); and the variety of analytical modes available for measuring the composition and other properties of the specimen. Depending on the SEM, the resolution can fall somewhere between less than 1 nm and 20 nm.