Scanning and Transmission Electron Microscopy

Harish Kumar K S

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1. First steps in Scanning and Transmission Electron Microscopy

In SEM, a fine beam of electrons of upto 40 keV energy is focused on the sample and scanned along a pattern of parallel lines. Various signals are generated as a result of incident electrons which are collected to form an image of the sample surface. These are mainly secondary electrons (few tens of ev) that are back-scattered from the primary beam.

In 1926, ***Busch*** studied the trajectories of charged particles in axially symmetric electric and magnetic fields and showed that such field could act as ***particle lenses.*** Nearly the same time, French scientist ***de-broglie*** showed that a frequency and hence a wavelength associated with charged particles. Following these two discoveries in electron optics, electron microscopy began to take shape.

In 1931, ***Ruska*** and his research group learned that using de-broglie's equation, the wavelength of electrons were almost five orders of magnitude smaller than that of light used in optical microscopy. This means high-resolution images.

Using de-broglie equation: $\lambda = \frac{1.227}{\sqrt{V}}\ (nm)$

	Wavelength λ (nm)	
V (kV)	Uncorrected	Relativistically corrected
20	0·0086	0·0086
40	0·0061	0·0060
60	0·0050	0·0049
80	0·0043	0·0042
100	0·0039	0·0037
200	0·0027	0·0025
300	0·0022	0·0020
400	0·0019	0·0016
500	0·0017	0·0014
1000	0·0012	0·0009

V is the accelerating voltage. For electrons accelerated at 75 KV, the limiting wavelength limit is 0.44 nm.

Knoll built a first scanning microscope in 1935. However, he did not use demagnifying lenses to produce a fine probe. So, the resolution was limited. In 1942, ***Zworykin*** used electron multiplier tube that reached a resolution of 50 nm, which was still lower than that of TEM.

In 1960, ***Everhart and Thornley*** greatly improved the secondary electron detection which provided topographic contrast. A new detector with grid that was positively biased to collect the electrons, a scintillator to convert them to light and a light-pipe to transfer the electrons to photo-multiplier tube was developed.

In 1963, ***Peace and Nixon*** combined all these improvements in one instrument: SEM V, with three magnetic lenses and an Everhart-Thornley Detector (ETD). This was the prototype of the first commercial SEM built in 1965 by Cambridge Scientific Instruments.

SEM and TEM

Principles and Basic Instrumentation

2. Scattering of electrons:

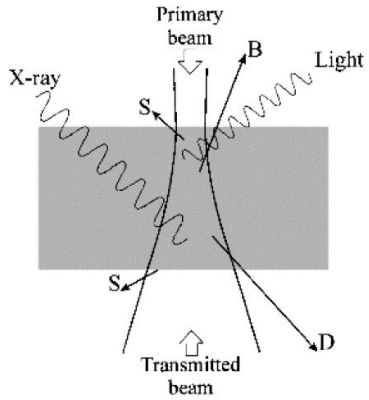
In almost all electron microscopy, the primary electron beam enters the specimen and the same or different electrons leave it to form an image. We need to understand the interactions between the electrons and surface.

2.1 Elastic scattering: It is the process in which, although it might change the direction of primary electrons, the energy does not change appreciably. It results from the columbic interaction between the primary electrons and both the nucleus and all the electrons around it.

2.2 In-elastic scattering: It is a very general term in which the primary electrons lose a detectable amount of energy ΔE. It has to be substantially more than 0.1 ev to be detected. There are many interaction processes that lead to the loss of energy of primary electrons like phonon and plasmon scattering, inner shell excitation etc.

3. Secondary effects: It is the effect caused by the primary electron that can be detected outside the specimen.

3.1 Secondary electrons: These are the electrons that escape from the specimen with energies below 50 ev. They also could be primary electrons at the end of their trajectories that reach the surface with few ev remaining. They usually are abundant and commonly used imaging signal in SEM.



A summary of the effects which may be detected when a primary beam of high energy electrons hits a specimen. S = secondary electrons; B = backscattered electrons; D = diffracted electrons.

3.2 Backscattered electrons: As already seen, some primary electrons loose most of their energy and escape the surface. It turns out most of the electrons do this, while still having a significant amount of incident energy. Backscattered electrons are not usually abundant as secondary electrons.

3.3 Relaxation of excited atoms: If an electron is knocked out of an atom, it will be in an excited, higher energy state. At some time later the empty electron state will be filled and the atom will relax, giving of some excess energy. If the vacant electron state is an outer state, then the energy given off will be small in the form of photon in the visible range. This

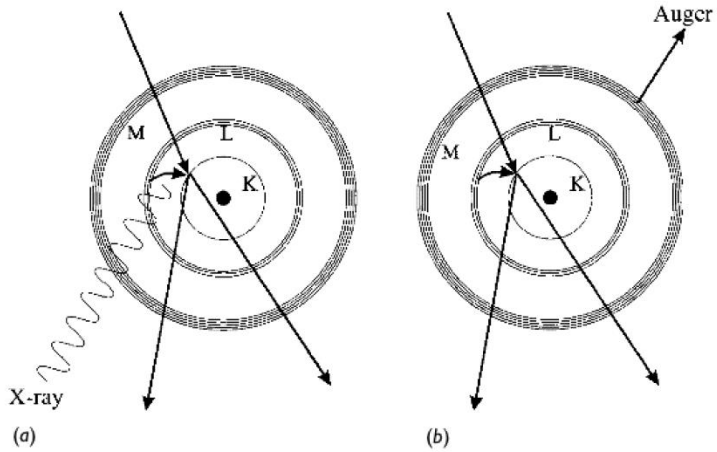


Figure 2.9 The two ways in which an inner-shell-excited atom can relax. In both cases a K shell electron has been knocked out – in (*a*) a characteristic (K_α) X-ray is emitted while in (*b*) an Auger electron (KLM) is ejected.

effect is called ***Cathodoluminescence.*** If on the other hand, the empty electron state is an inner state, then there are two main possibilities: Characteristic X-ray or characteristic electron (Auger) peak will be emitted.

The example shown above if for molybdenum. In both cases an electron in K shell has been knocked out. If an electron from L shell fills out the vacant state, then an energy corresponding to the difference in the energies of K and L shell will be emitted as molybdenum K_α X-rays (17400 ev, 0.071nm). If an electron from M shell fills the vacant state, then the X-ray will be more energetic, emitted as molybdenum K_β X-rays (19600 ev, 0.063 nm). The corresponding wavelength can be calculated as follows:

$$\lambda = \frac{hc}{\Delta E}(nm) = \frac{1240}{\Delta E}$$

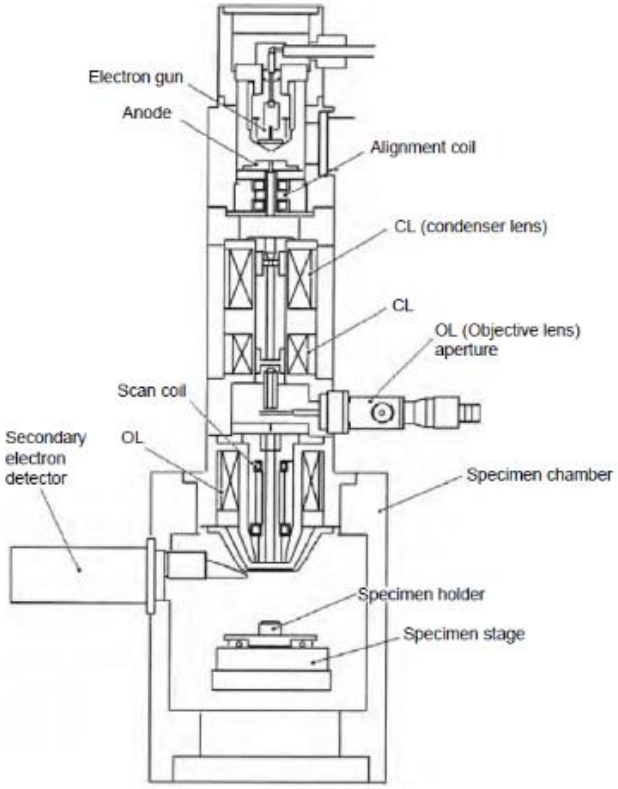
These energies and wavelengths are different from different atoms which can help us identify the chemical nature of the species.

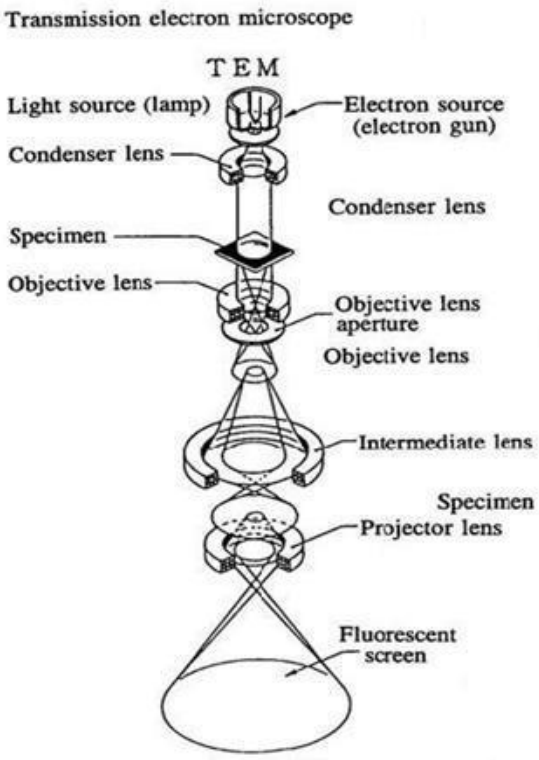
It is entirely possible that the primary electron can excite an X-ray by losing all or part of its total kinetic energy without knocking out an atom. These X-rays are non-characteristic and are called as ***Bremsstrahlung radiation*** (breaking-radiation) and leads to background radiation.

An alternative to X-ray emission is the Auger electron emission, in which an outer electron carries the excess energy of the electronic transition from L to K or M to K shell.

4. Instrumentation of SEM and TEM:

The above figure shows a column structure of conventional SEM and TEM. The electron gun, which is on the top of the column, produces the electrons and accelerates them to an energy level of 0.1–30 keV. The diameter of electron beam produced by hairpin tungsten gun is too large to form a high-resolution image. So, electromagnetic lenses and apertures are used to focus and define the electron beam and to form a small focused electron spot on the specimen. This process demagnifies the size of the electron source (~50 μm for a tungsten filament) down to the final required spot size (1–100 nm). A high-vacuum environment, which allows electron travel without scattering by the air, is needed. The



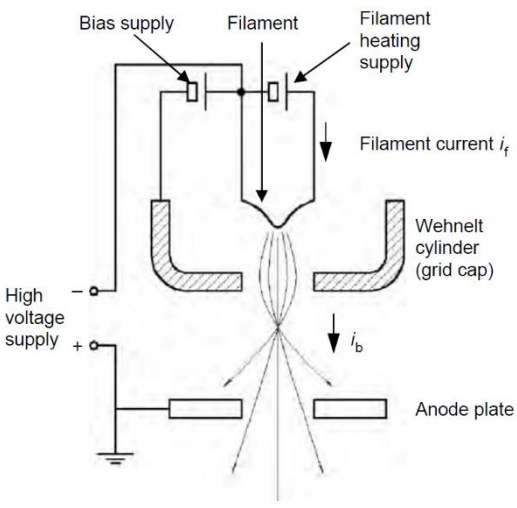


specimen stage, electron beam scanning coils, signal detection, and processing system provide real-time observation and image recording of the specimen surface.

4.1 Electron Gun:

Several types of electron guns are used in SEM system and the qualities of electrons beam they produced vary considerably. The first SEM systems generally used tungsten "hairpin" or lanthanum hexaboride (LaB6) cathodes, but for the modern SEMs, the trend is to use field emission sources, which provide enhanced current and lower energy dispersion. Emitter lifetime is another important consideration for selection of electron sources.

4.1.1 Tungsten Electron guns:



They have been used for many applications especially for low-magnification imaging. It has three parts:

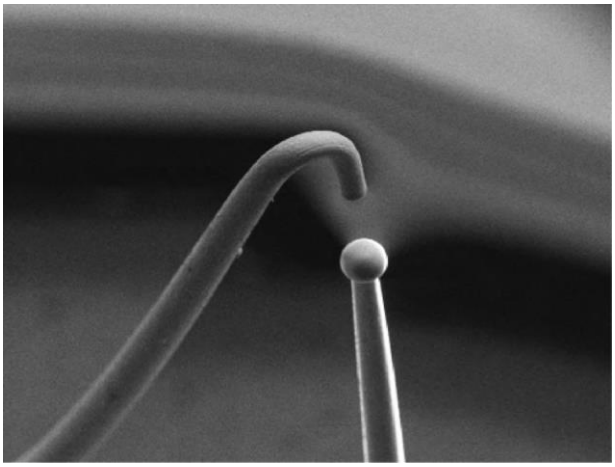
(i) V-shaped hairpin tungsten filament-cathode

(ii) Wehnelt cylinder

(iii) Anode

The tungsten filament is about $100\mu m$ in diameter. It is heated to more than 2800 K by applying current, so that the electrons can escape from the filament.

A negative potential, which is varied in the range of 0.1–30 kV, is applied on the tungsten and Wehnelt cylinder by a high voltage supply. As the anode is grounded, the electric field between the filament and the anode plate extracts and accelerates the electrons toward the anode. The electron emission increases with the filament current. There is some "saturation point" of filament current, at which we have most effective electron emission (i.e., the highest electron emission is obtained by least amount of current). At saturation electrons



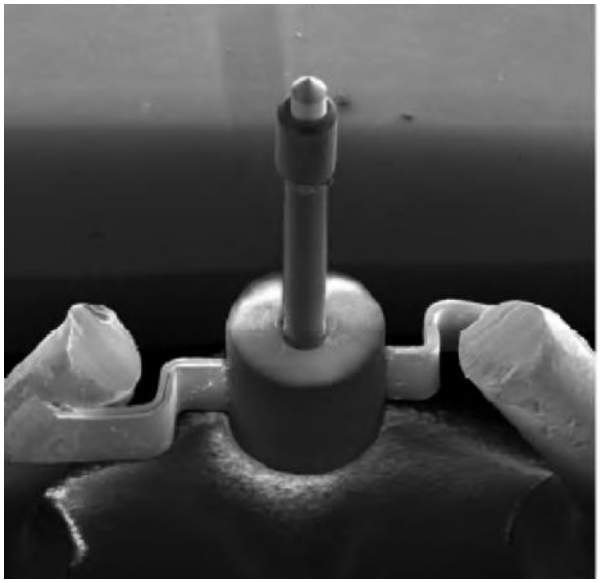
(a) SEM image of a "blown-out" tungsten electron gun

are only emitted from the tip of the filament and focused into a tight bundle by the negative accelerating voltage. If the filament current increases further, the electron emission only increases slightly. Over-heating the filament will cause it to a spherical-melt as shown.

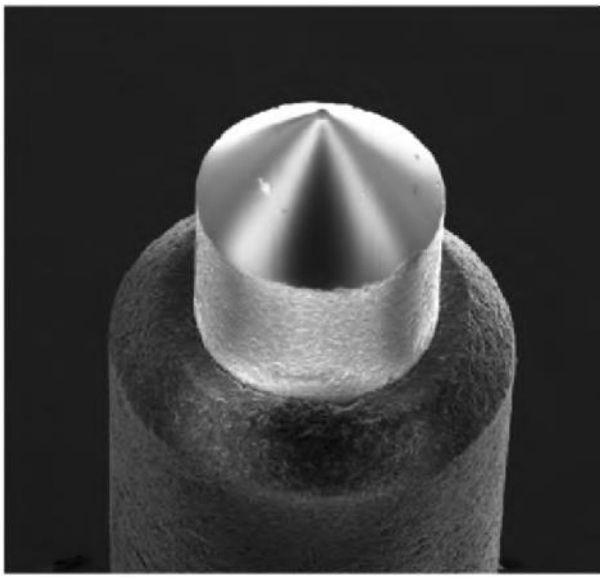
4.1.2 Lanthanum Hexaboride guns (LaB_6)

An alternative to tungsten filament is LaB_6, which has a lower work function of 2.4 ev (4.5 ev for tungsten). This mean that LaB_6 can provide stronger emission of electrons than tungsten at the same heating temperature (lower energy requirement). LaB_6 guns can provide 5-10 times brightness and lasts longer than tungsten filaments. They are 100-200 μm in diameter and 0.3 mm long. It is mounted on graphite or rhenium support, which do not chemically react with LaB_6 and also, they act as heat resistors to elevate the crystal temperature. The effective emission area is much smaller than tungsten-reducing the beam spot size (high resolution).

LaB_6 is readily oxidized at elevated temperatures and the vacuum in electron gun chamber is not high enough to prevent oxidation. The SEM image below shows the electron gun with contamination spots.



(a) SEM image of LaB_6 electron gun

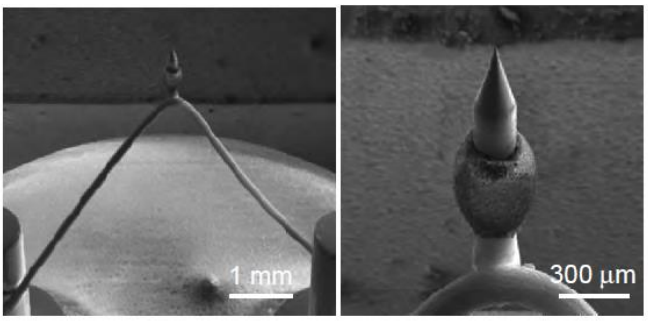


(a)Magnified SEM image of LaB_6 with contamination spots

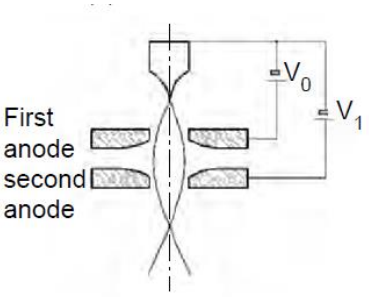
4.1.3 Field Emission Guns:

Thermionic sources as discussed above depend on high temperature to overcome the workfunction of the metal so that electrons can escape from cathode. Though they are inexpensive, they have disadvantages of less brightness, large energy spread and short lifetime. In modern electron microscopes, field emission guns are used.

In FEGs, a single crystal tungsten wire with very sharp tip, prepared by electrolytic etching is used. A strong electric field forms on the finely oriented tip, and the electrons are drawn toward the anode. Two anodes are used in FEGs: the first anode with voltage V_1 is used to extract electrons and V_o is the accelerating voltage.



(a) and (b) SEM images of FEG



(c) Configuration of FEG

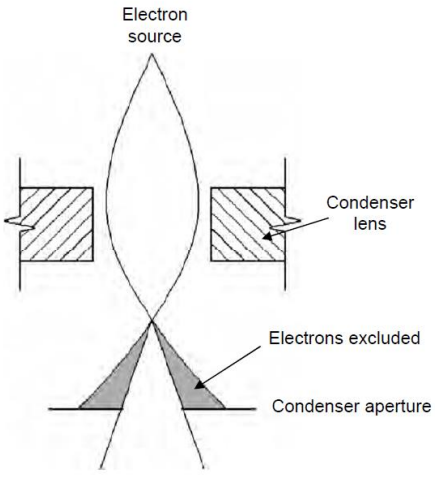
FEG produces 100 times the brightness produced by thermionic sources. Very low energy of electron spread of 0.3 ev can be achieved. But a high vacuum of 10^{-9} torr is required to stabilize electron emission and prevent contamination.

4.2 Electron Lenses

Electron beams can be focused by electrostatic or magnetic field. But electron beam controlled by magnetic field has smaller aberration, so only magnetic field is employed in SEM system. Coils of wire, known as "electromagnets," are used to produce magnetic field, and the trajectories of the electrons can be adjusted by the current applied on these coils. Even using the magnetic field to focus the electron beam, electromagnetic lenses still work poorly compared with the glass lenses in terms of aberrations. The electron lenses can be used to magnify or demagnify the electron beam diameter, because their strength is variable, which results in a variable focal length. SEM always uses the electron lenses to demagnify the "image" of the emission source so that a narrow probe can be formed on the surface of the specimen.

4.2.1 Condenser Lens:

The electron beam will diverge after passing through the anode plate from the emission source. By using the condenser lens, the electron beam is converged and collimated into a relatively parallel stream. A magnetic lens generally consists of two rotationally symmetric iron pole pieces in which there is a copper winding providing magnetic field. There is a hole in the center of pole pieces that allows the electron beam to pass through. The position of the focal point can be controlled by adjusting the condenser lens current. A condenser aperture, generally, is associated with the condenser lens, and the focal point of the electron beam is above the aperture to exclude inhomogeneous and scattered electrons.

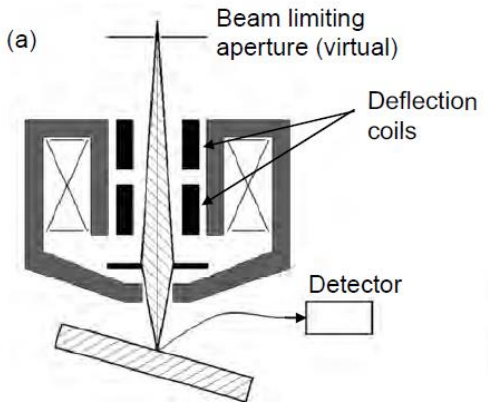


4.2.2 Objective Lens:

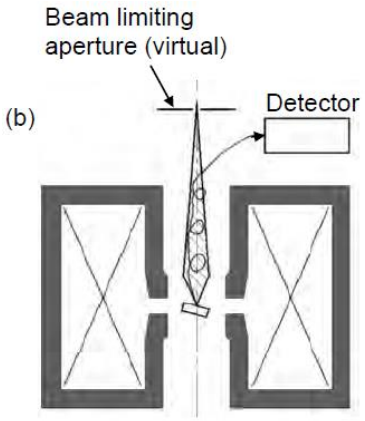
The electron beam will diverge below the condenser aperture. Objective lenses are used to focus the electron beam into a probe point at the specimen surface and to supply further

demagnification. An appropriate choice of lens demagnification and aperture size results in a reduction of the diameter of electron beam on the specimen surface (spot size), and enhances the image resolution.

The asymmetric pinhole lens is the most common objective lens. There is only a small bore on the pole piece, and this keeps the magnetic field within the lens and provides a field-free region above the specimen for detecting the secondary electrons. However, this configuration has a large lens aberration.

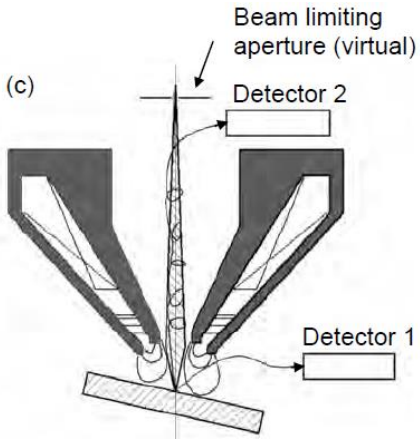


(a) Pinhole Lens



(b) Symmetric immersion Lens

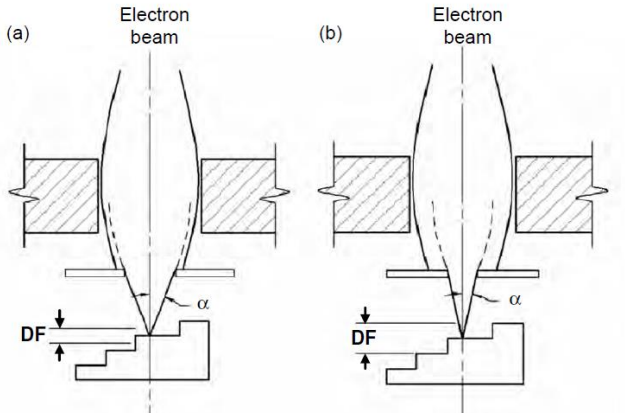
For the symmetric immersion lens, the specimen is placed inside the lens, which can reduce the focal length significantly. This configuration provides a lowest lens aberration because lens aberration directly scales with the focal length. But the specimen size cannot exceed 5 mm. The Snorkel lens produces a strong magnetic field that extends to the specimen. This



(c) Snorkel Lens

kind of lens possesses the advantages of the pinhole lens and the immersion lens, combining low lens aberration with permission of large specimen.

Various other factor like aperture size, Working Distance (WD), electron energy and lenses current affect the size of spot area.



Aperture: The smaller the aperture, the higher is the resolution and depth of field. The resolution is directly proportional to the angle of convergence α. Higher depth of field produces more detailed images. The image below shows how decreasing the aperture size improves detail of ZnO nano-rods. Decreasing the aperture size also increasing the Working Distance (WD), meaning that more information can be obtained from the background.

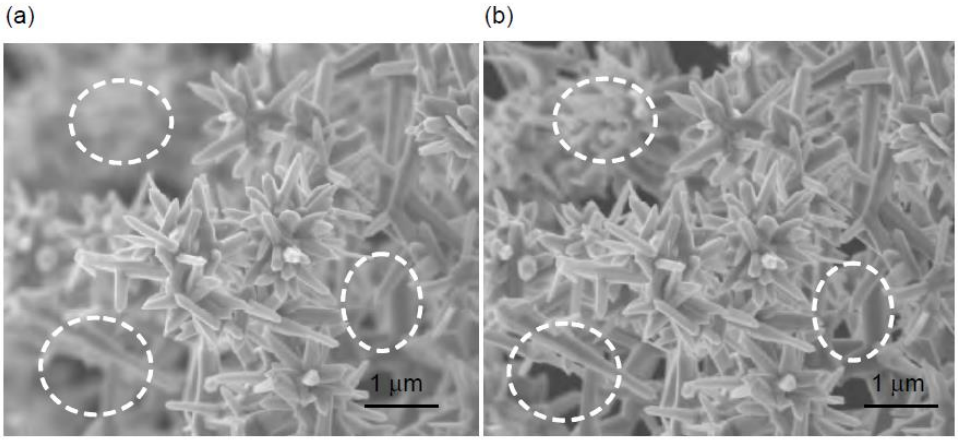
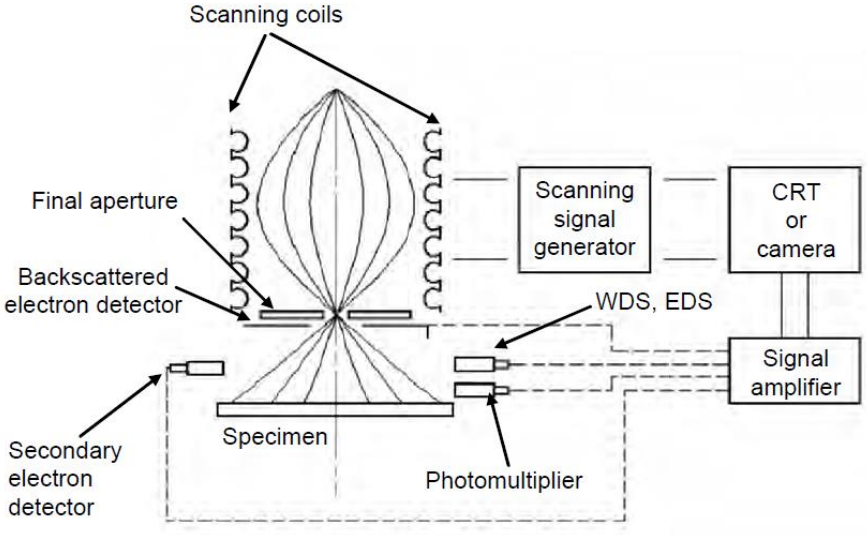


FIGURE 1.16. Electron micrograph of branch grown ZnO nanorods taken with different aperture sizes: (a) 30 µm and (b) 7.5 µm. The enhancement of depth of field is emphasized by circles.

4.3 Image formation:

Complex interactions occur when the electron beam in an SEM impinges on the specimen surface and excites various signals for SEM observation. The secondary electrons, BSEs,



transmitted electrons, or the specimen current might all be collected and displayed. For gathering the information about the composition of the specimen, the excited x-ray or Auger electrons are analyzed.

4.3.1 Signal generation:

Two kinds of scattering process, the elastic and the inelastic process, are considered. The electrons retain all of their energy after an elastic interaction, and elastic scattering results in the production of BSEs when they travel back to the specimen surface and escape into the vacuum. On the other hand, electrons lose energy in the inelastic scattering process and they excite electrons in the specimen lattice. When these low energy electrons, generally with energy less than 50 eV, escape to the vacuum, they are termed “secondary electrons.” In addition to secondary electrons and BSEs, x-rays are excited during the interaction of the electron beam with the specimen. There are also several signals that can be used to form the images or analyze the properties of specimen, e.g., Auger electrons, cathodoluminescence, transmitted electrons, and specimen current.

4.3.2 Scanning coils

the electron beam is focused into a probe spot on the specimen surface and excites different signals for SEM observation. By recording the magnitude of these signals with suitable detectors, we can obtain information about the specimen properties, e.g., topography and composition. However, this information just comes from one single spot that the electron beam excites. In order to form an image, the probe spot must be moved from place to place by a scanning system. Scanning coils are used to deflect the electron beam so that it can scan on the specimen surface along x- or y-axis. Several detectors are used to detect different signals: solid state BSE detectors for BSEs; the ET detector for secondary and BSEs; energy-dispersive x-ray spectrometer and wavelength-dispersive x-ray spectrometer for the characteristic x-rays; and photomultipliers for cathodoluminescence. The detected signal is also processed and projected on the CRT screen or camera. The scanning process of CRT or camera is synchronized with the electron beam by the scanning signal generator and hence a point-to-point image for the scanning area is produced.

4.3.3 Secondary Electron Detectors:

The original goal in building an SEM was to collect secondary electron images. Because secondary electrons are of low energy, they could come only from the surface of the sample under the electron beam and so were expected to provide a rich variety of information about the topography and chemistry of the specimen. However, it did not prove to be an easy task to develop a collection system which could detect a small current (~10^{-12} A) of low energy electrons at high speed enough to allow the incident beam to be scanned, and which worked without adding significant noise of its own. The only practical device was the electron multiplier. In this, the secondary electrons from the sample were accelerated onto a cathode where they produced additional secondary electrons that were then in turn accelerated to a second cathode where further signal multiplication occurred. By repeating this process 10 or 20 times, the incident signal was amplified to a large enough level to be used to form the image for display. Although the electron multiplier was in principle sensitive enough, it suffered from the fact that the cathode assemblies were exposed to the pump oil, water vapor, and other contaminants that were present in the specimen chambers of these early instruments with the result that the sensitivity rapidly degraded unless the multiplier was cleaned after every new sample was inserted.

The solution to this problem was provided by ***Everhart and Thornley***, as discussed in the introduction. They devised a detector that had three components: a scintillator that converts electron signal into light, a light-pipe to transport the photons and a photo-multiplier tube to convert the light back into electrical signal.

4.4 Vacuum System:

An ultra-high vacuum system is indispensable for SEMs in order to avoid the scattering on the electron beam and the contamination of the electron guns and other components. More than one type of vacuum pump is employed to attain the required vacuum for SEM. Generally, a mechanical pump and a diffusion pump are utilized to pump down the chamber from atmospheric pressure.

4.4.1 Mechanical pumps:

Mechanical pumps consist of a motor-driven rotor. The rotating rotor compresses large volume of gas into small volume and thereby increases the gas pressure. If pressure of the

compressed gas is large enough, it can be expelled to the atmosphere by a unidirectional valve.

4.4.2 Diffusion pumps:

The vaporized oil circulates from top of the pump to bottom. The gas at the top of the pump is transported along the vaporized oil to the bottom and discharged by the mechanical pump. It can achieve pressures upto $5x10^{-5-}$ torr. It cannot work at pressure above 10^{-2} torr.

4.4.3 Ion pumps:

Ion pumps are used to attain the vacuum level at which the electron guns can work, especially for the LaB_6 guns (10^{-6} –10^{-7} Torr) and field emission guns (10^{-9}–10^{-10} Torr). A fresh surface of very reactive metal is generated by sputtering in the electron gun chamber. The air molecules are absorbed by the metal surface and react with the metal to form a stable solid. A vacuum better than 10^{-11} Torr is attainable with an ion pump.

4.4.4 Turbo pumps:

The basic mechanism of turbo pump is that push gas molecules in a particular direction by the action of rotating vanes. As the vanes rotate, they push the molecules from the chamber side to the backing pump side and finally go to front pump system. On the one hand, if the molecules were incident from the backing pump side to the chamber side, it will be pushed to the backing pump side. In this way, a preferred gas flow direction is created and a pressure difference is maintained across vane disk.

5. Developments in Electron Microscopy:

5.1 Scanning Transmission Electron Microscopy (STEM):

This technique combines both SEM and TEM by collecting a transmission image by scanning method. Since the scanning method is limited by the diameter of the probing beam, a very fine beam is required for STEM for high resolution. It can be achieved by FEG as already seen. STEM is typically used with UHV environment. So, they are not prone to contamination. Useful information has been obtained from regions as small as 0.5 nm in diameter. The beam or specimen should not drift more than 0.1 nm during the collection period.

5.2 Atomic Probe Field Ion Microscope (APFIM):

It is one of the most sensitive techniques developed in the field ion microscope. If a high potential is applied to FIM, a layer of atoms can be stripped from the surface. These will be ionized and accelerated towards an imaging screen. In order to analyse a selected atom, it is necessary to orient the specimen in such a way that the image of the atom falls over a hole in the fluorescent screen.

5.3 Auger microscopy and spectroscopy:

The emission of characteristic electrons (Auger) has already been discussed. As these electrons are of low energy, they can be analysed by electron spectrometer. If the spectrometer is mounted on SEM, it becomes Scanning Auger Microscopy (SAM). Most of the Auger electrons are re-adsorbed on the solid specimen and will not escape. Therefore, high electron beam currents are necessary to produce useful Auger peaks. This means large aperture size and the resolution is limited to 50 nm.