



1.1 What Is the SEM

The word microscope is derived from Greek *micros* (small) and *skopeo* (look at). Just like any microscope, the primary function of the scanning electron microscope (SEM) is to enlarge small features or objects otherwise invisible to human sight. It does that by way of using electron beam rather than light which is used to form images in optical light microscopes. The images are obtained by scanning an electron beam of high energy on the sample surface, hence the name scanning electron microscope. By virtue of its smaller wavelength, electrons are able to resolve finer features/details of materials to a much greater extent compared with optical light. A modern day SEM can magnify objects up to one million times their original size and can resolve features smaller than 1 nm in dimension. Similarly, electron beam interaction with the specimen emits x-rays with unique energy that can be detected to determine the composition of material under examination. The SEM is, therefore, a tool used for materials characterization that provides information about the surface or near surface structure, composition, and defects in bulk materials. It allows scientists to observe surfaces at submicron and nano-level to elaborate material properties. It has emerged as one of the most powerful and versatile instruments equally valuable to materials and life scientists working in wide-ranging industries.

1.2 Image Resolution in the SEM

A human eye cannot distinguish objects smaller than 200 μm (0.2 mm). In other words, the resolution of a human eye is 200 μm , while a light microscope can typically magnify images up to 1000 \times to resolve details down to 0.2 μm . Resolution limit is defined as the smallest distinguishable distance separating two objects, i.e., minimum resolvable distance. For instance, two objects separated by a distance of

less than $200\ \mu\text{m}$ will appear as one object to the human eye since the latter is not able to resolve details that have dimensions smaller than $200\ \mu\text{m}$. Hence, $200\ \mu\text{m}$ can be considered to be the resolution limit of the human eye. The same objects viewed under a light microscope will appear as two distinct entities since the light microscope can easily differentiate distances less than $200\ \mu\text{m}$. In fact, the objects can be brought closer together further to a distance of $0.2\ \mu\text{m}$ and still maintain their separate identities under a light microscope. However, if the distance between the objects is decreased further to less than $0.2\ \mu\text{m}$, the light microscope will no longer be able to discern them as two separate objects, which will then appear as a single entity. Thus $0.2\ \mu\text{m}$ can be defined as the resolution limit of the light microscope. It follows that the smaller is the value of minimum resolvable distance, the higher is the resolution of a microscope.

Both light microscope and humans use visible light as a means to probe into or interact with an object. The increased ability to observe details in a light microscope compared to the unaided eye is attributed to the lens/aperture system used to magnify the image of an object. It is theoretically possible to keep enlarging the image by increasing the magnification indefinitely. However, it is not possible to keep revealing newer details in an object by simply increasing the magnification. Fine details in an image cannot be resolved beyond a certain magnification. This is due to limitations imposed by the resolving power of the imaging technique as well as that of the human eye. The maximum useful magnification beyond which no further details are revealed is determined by the resolving power of a microscope. The following equation can be used to determine the typical useful magnification of a microscope:

$$\text{Useful Magnification} = \frac{\text{Resolution of the Human Eye}}{\text{Resolution of Microscope}} \quad (1.1)$$

For a light microscope, useful magnification $\left(\frac{200\ \mu\text{m}}{0.2\ \mu\text{m}}\right)$ is around $1000\times$. For a scanning electron microscope, useful magnification $\left(\frac{200\ \mu\text{m}}{1\ \text{nm}}\right)$ is typically $200,000\times$. Increase in the resolution of the instrument results in the increase of its useful magnification.

The ability of visible light to resolve image details is limited by its relatively large wavelength ($\lambda = 380\text{--}760\ \text{nm}$) (see Fig. 1.1). Use of light with a shorter wavelength (such as ultraviolet) and a lens immersed in oil (high refractive index) improves resolution to around $0.1\ \mu\text{m}$. If the image is formed by using a radiation with a smaller wavelength, such as an electron beam, higher resolution limit can be achieved since the smaller the wavelength, the greater the resolving power and the greater the detail revealed in an image. Due to this fact, techniques like the SEM and TEM employ an electron beam to probe the material resulting in an image far superior in resolution compared to that of the light microscope. For example, an electron beam (λ of $0.000004\ \mu\text{m}$) with an accelerating voltage of $100\ \text{kV}$ can achieve a resolution of $0.24\ \text{nm}$. The practical limit to resolution is determined by lens aberrations and defects. Modern-day field emission SEM typically operated at

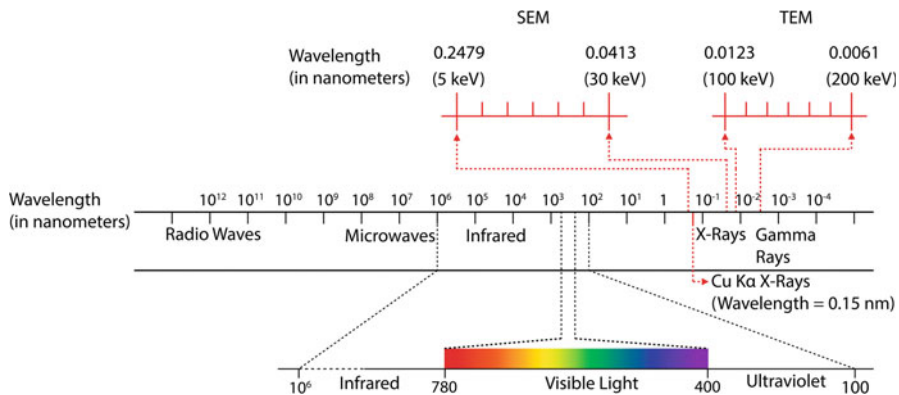


Fig. 1.1 Electromagnetic spectrum showing the size of the wavelength used in the light, scanning (SEM), and transmission electron microscope (TEM)

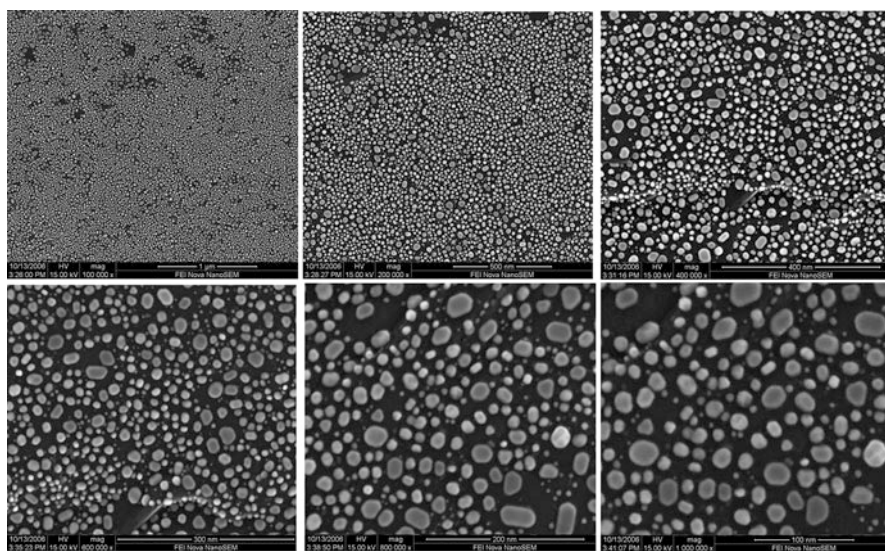


Fig. 1.2 Secondary electron images of tin balls showing good contrast at low to very high magnifications (100,000 \times to 1,000,000 \times)

20–30 kV accelerating voltages can achieve image resolution in the order of 1 nm or better. It is worth noting here that resolving power or resolution (a more commonly used term) of an instrument is demonstrated by manufacturers using a specimen *ideally* suited for that instrument. For instance, tin balls/powder is routinely employed for the SEM since the former is conductive and has strong contrast (see Fig. 1.2). Details in real samples, however, are not usually revealed to that level of resolution.

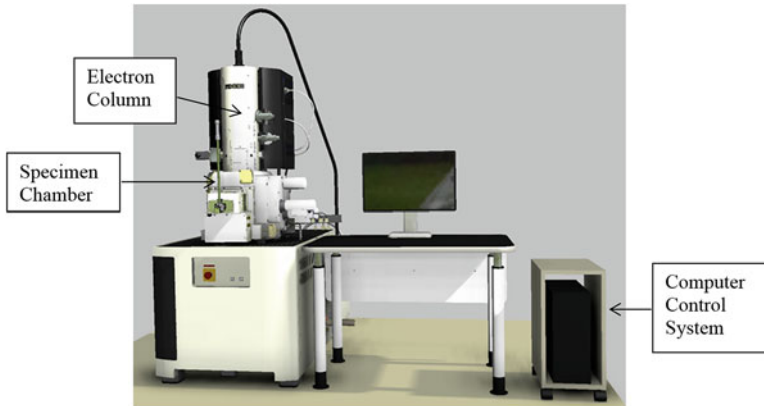


Fig. 1.3 A photograph showing three major sections of the SEM: the electron column, the specimen chamber, and the computer control system. (Courtesy of T. Siong, JEOL Ltd.)

1.3 Image Formation in the SEM

The SEM instrument can be considered to comprise of three major sections: the electron column, the specimen chamber, and the computer/electronic controls, as shown in Fig. 1.3. The topmost section of the electron column consists of an electron gun which generates an electron beam. Electromagnetic lenses located within the column focus the beam into a small diameter (few nanometers) probe. The scan coils in the column raster the probe over the surface of the sample present in the chamber that is located at the end of the column. The gun, the column, and the specimen chamber are kept under vacuum to allow electron beam generation and advancement. The electrons in the beam penetrate a few microns into the surface of a bulk sample, interact with its atoms and generate a variety of signals such as secondary and backscattered electrons and characteristic x-rays that are collected and processed to obtain images and chemistry of the specimen surface. The ultimate lateral resolution of the image obtained in the SEM corresponds to the diameter of the electron probe. Advances in the lens and electron gun design yield very fine probe diameters giving image resolutions of the order of <1 nm. In order to provide a perspective to the way the image is realized in the SEM, a comparison of how light and the transmission electron microscopes work compared to the SEM is shown in Table 1.1 and Fig. 1.4.

1.4 Information Obtained Using the SEM

The scanning electron microscope is used to observe and image the micro- and nanostructural surface details of a wide range of materials such as metals, alloys, ceramics, polymers, rock minerals, corrosion deposits, filters, membranes, foils,

Table 1.1 Comparison of various characteristics of the SEM with light and transmission electron microscope

Characteristics	Light microscope	Scanning electron microscope	Transmission electron microscope
Radiation used to form an image	Visible light	Electrons	Electrons
Wavelength of radiation	380–760 nm depending on the color of light	0.008 nm at 20 kV accelerating voltage ^a	0.0028 nm at 200 kV accelerating voltage ^a
Types of lenses used to focus the radiation	Glass	Electromagnetic	Electromagnetic
Useful magnification	1,000×	200,000×	2,000,000×
Possible magnification	Up to 2,000×	1,000,000×	10,000,000 or more
Magnification mode	With lenses	Without lenses	With lenses
Resolution	200 nm	1 nm	0.1 nm
Source of radiation	Tungsten-halogen lamp	Electron gun/emitter	Electron gun/emitter
Image formation	Light from the source is scattered by the sample surface and redirected by the objective lens to form an image onto the retina of the human eye. The image can also be displayed on an electronic display	Electrons originating from the source travel in vacuum within a column lined with electromagnetic lenses which focus these electrons into a small probe on the surface of the specimen. Electron-specimen interaction results in information emanating from the specimen which is passed through detectors and reconstituted as an image on an electronic display	Electrons originating from the source travel in vacuum within a column. Electron beam passes through a thin foil of sample and then focused and magnified by electromagnetic lenses to form an image on a fluorescent screen or transferred to an electronic display
Type of image	Real image. Color images. Images formed using visible light can be observed directly by the human eye	Processed/reconstituted image. Grayscale images (black and white). Images formed with electrons cannot be observed directly by humans	The real image is projected onto the screen which can be observed by the human eye. Grayscale images
Specimen preparation	Required	Can be omitted (based on specimen type)	Required, tedious
Specimen thickness	Thin, bulk	Bulk	Thin (Electron transparent, ≈100 nm)
Sample area examined	Large areas of a sample can be examined	Small areas of a sample are examined	Extremely small areas of a sample are examined

(continued)

Table 1.1 (continued)

Characteristics	Light microscope	Scanning electron microscope	Transmission electron microscope
Applications	Materials/life sciences	Materials/life sciences	Materials/life sciences
Examination of live specimens	Living organisms can be examined	Vacuum usage and high energy electron radiation precludes examination of live samples	Vacuum usage and high energy electron radiation precludes examination of live samples
Depth of field	Small ^b 15 μm (at 4 \times) 0.2 μm (1000 \times)	Large (3-D like images) ^b 4 mm (at 10 \times) 0.5 μm (500,000 \times)	Small
Lab size requirements	2 \times 2 m	5 \times 5 m	6 \times 6 m
Capital and maintenance cost	Low	Medium	High
Features studied	Surface	Surface or subsurface	Microstructure
Interpretation of images	Easy	Moderate	Difficult

^aElectron wavelength is calculated from De Broglie equation: $\frac{1.23 \text{ nm}}{\sqrt{\text{accelerating voltage in thousands}}}$

^bThe exact depth of field depends on the working distance (i.e., the distance between the lens and the imaged surface).

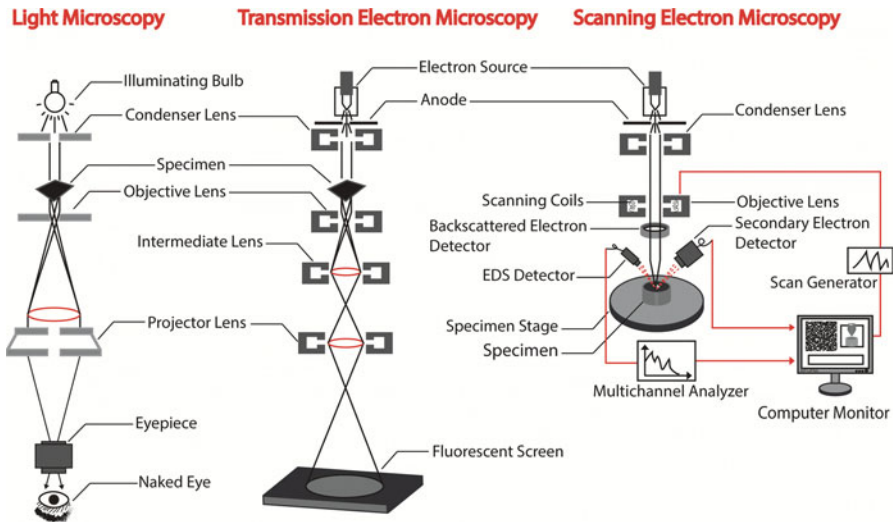


Fig. 1.4 Schematic comparing the modes of image formation in the light, transmission, and scanning electron microscopes

fractured/rough surfaces, biological samples, etc. The materials can be conductive or non-conductive either in solid or powder form and can be examined in an as-received or prepared (sectioned, ground, polished, etched, coated, etc.) condition. The SEM has the ability to examine materials in the dry or wet state as well as obtain microchemical information from fine structural details. The SEM equipped with a field emission gun can distinguish surface features that are only 1 nm apart (i.e., lateral spatial resolution = 1 nm). Extraordinary ability to depict large depths of field (10–100% of horizontal field width) allows large areas of a sample to remain in focus at one time and thus yield 3-D characteristics in SEM images (see Fig. 1.5). Imaging can be performed using both secondary electrons (for topographic contrast) and backscattered electrons (for topographic and/or compositional contrast). Microchemical information is generally obtained using energy dispersive x-ray spectrometer (EDS) detector attached to the SEM. Both qualitative and quantitative elemental information from microstructural features can be obtained from beryllium to uranium with limits of detection of approximately 0.2–0.5 wt%. The electron beam in the SEM can penetrate as much as a few microns into the sample depending



Fig. 1.5 Secondary electron SEM image of an ant showing detailed three-dimensional details. (Image courtesy of TESCAN)

on sample density, beam accelerating voltage, etc. Typical applications include observation of metallographically prepared samples (such as steel) to study surface morphology, grain size/shape, inclusions, precipitates, dendrites, grain boundaries, etc. It is also employed to observe materials in an unprepared (as-received) condition, e.g., fracture surfaces for metallurgical failure analysis, electronic devices for electronic failure analysis, corrosion deposits, catalyst shape, size and surface structure, polymer additives, rock mineral samples, etc. Apart from bulk samples, it is also used to examine coatings and thin films deposited on substrates. Table 1.2 highlights various applications and the range of information obtained using SEM and related techniques.

A combination of factors such as good resolution, large depth of field, compositional information, time-efficient analysis, as well as relative ease of use and image interpretation in both materials and life sciences has made the SEM one of the most heavily used instruments in academia, research, and industry. Although the SEM can generate rich morphological and compositional data for a wide variety of materials, it is often necessary to employ various other analytical tools to undertake complete materials characterization. Selection of these tools will depend on the type of material under study and the nature of information required. For instance, if the objective is to reveal true microstructure (not surface topography) of a material, transmission electron microscopy is employed. This technique can also extract chemical information from features at the nanometer scale. Likewise, if the bulk composition is desired, x-ray fluorescence is a better choice as it analyzes large volumes of material. Phase identification at a macro level is often carried out using x-ray powder diffraction analysis. Analysis of structure and composition of a thin surface constituting a few atomic monolayers is undertaken with Auger and x-ray photoelectron spectroscopy. All these techniques are tools available to a scientist to carry out essential materials characterization and information obtained from one complements the others. A brief comparison of the imaging and analytical abilities of these techniques is shown in Table 1.3.

1.5 Strengths and Limitations of the SEM

Strengths of the SEM include:

1. A wide variety of specimens can be examined.
2. Relatively easy and quick sample preparation.
3. Ease of use due to user-friendly and automated equipment.
4. Rapid imaging, quick results, time-efficient analysis, and fast turnaround time.
5. Relatively straightforward image interpretation.
6. Large depth of field (ability to focus large depths of samples at one time and produce 3-D like images)
7. Microchemical analysis capability from Be to U.
8. Samples can be dry or wet.
9. Nondestructive (some beam damage may result).

Table 1.2 Summary of capabilities of the SEM and related techniques

Information obtained using SEM and related techniques	<p>Morphology (grain shape, precipitate size, dimension, texture and volume fraction, and phase distribution of physical features) [using secondary electron (SE) imaging]</p> <p>Surface topography (distribution/arrangement of features at the surface of a sample, defects, cracks, voids, structure) [using secondary electron imaging]</p> <p>Atomic number or compositional contrast, grain boundaries, domains in magnetic materials [using backscattered electron (BSE) imaging]</p> <p>Microanalysis, elemental identification and quantification, line scans, x-ray maps [using energy dispersive x-ray spectroscopy (EDS)]</p> <p>Kikuchi maps of bulk samples—crystallographic orientation of grains, phase identification [using electron backscattered diffraction (EBSD)]</p> <p>Pore size/shape/distribution in rock minerals, organic samples [using cryogenic techniques]</p> <p>Image non-conductive charging samples such as polymers, rocks, etc., without coating [using low vacuum (LV) mode]</p> <p>Imaging fine details of surface structures [using low accelerating voltage]</p> <p>Preparation of precise plan and cross-sectional view TEM samples, fabrication of nano devices [using SEM combined with focused ion beam]</p> <p>In situ experiments under hydration, thermal cycling, or gaseous environment [using environmental SEM (ESEM)]</p> <p>Printing of circuit boards in the electronics industry [using E-beam lithography]</p>
Applications	Materials identification, materials science, forensic science, metallurgical and electronic materials failure analysis, corrosion science, rock mineralogy, geo sciences, nano devices, polymer science, catalysis, semiconductor design, desalination, life sciences, oil and gas, mining
Types of samples examined	Metals, alloys, semiconductors, polymers, coatings, ceramics, rocks, sand, corrosion products, catalysts, membranes, carbon nanotubes, nanopowders, tissues, cells, insects, leaves
Industry	Academic and research, oil and gas, power generation, metals and alloys, industrial manufacturing, automobile, aero, aerospace, petrochemical, geosciences, nanotechnology, semiconductor, computer, chemical process industry, mining
Sample form/state/size	<p>Form: Solid, bulk, thin, nanostructured, powder, pellet, organic, inorganic</p> <p>State: Dry, wet</p> <p>Sample size: 1 mm to 150 mm (typically 10–20 mm)</p>
Spatial resolution	1 nm
Sampling depth	1–5 μm (depends on sample density and electron beam accelerating voltage)
Sample area analyzed	Submicron to >100 μm (depends on electron probe size, magnification)
Detection limit of elements	\approx 0.2 wt% (depends on relative elemental content and atomic weight)

Table 1.3 Comparison of imaging and analytical techniques generally used to study materials structure, chemical composition, and defects

Technique	Information/ applications	Spatial resolution	Sampling depth	Typical sample area analyzed	Elemental detection limits
Light microscopy	Imaging/materials and life sciences	200 nm	Surface	Millimeters	–
Scanning electron microscopy	Imaging, microchemical analysis/materials, and life sciences	1 nm	Submicron to several microns	Micron to millimeters	0.2–0.5 wt%
Transmission electron microscopy	Imaging, microchemical analysis, crystal structure/materials and life sciences	0.1 nm	<100 nm (thin foils)	<1 to few microns	0.01–0.1 wt%
Atomic force microscopy	Imaging, topography/ materials, and life sciences	Sub- nanometer	Sub- nanometer	Tens to hundreds of microns	–
Electron probe microanalysis with wavelength dispersive x-ray spectroscopy	Imaging, microchemical analysis/materials science	1 nm	Submicron to several microns	Micron to millimeters	0.01 wt%
X-ray diffraction	Structural analysis, lattice parameters, phase constitution, crystal structure, crystallite size, pore size, stress, texture, lattice distortion, thin film analysis/materials science	–	Tens to hundreds of microns	Sq. cm	–
X-ray fluorescence	Bulk chemical analysis/materials science	–	Tens to hundreds of microns	Sq. cm	ppm
X-ray photoelectron spectroscopy	Surface chemistry (binding energy, chemical state)/ materials science	–	0.5–5 nm	Tens of sq. μm	0.001 wt% to ppm
Auger electron spectroscopy	Surface chemistry, imaging (electronic structure, depth profiling)/materials science	8 nm	<5 nm	Nanometers to microns	0.01–0.1 at%

10. High spatial resolution (1 nm) achieved by modern equipment.
11. A versatile platform that lends support to other sophisticated tools, devices, and techniques.
12. Capable of several modes of imaging, spectroscopy, and diffraction analysis.
13. Affordability and availability compared to more expensive equipment.

Limitations can be summarized as follows:

1. The sample size is limited.
2. Samples are solid.
3. EDS detector cannot detect H, He, or Li.
4. Poor detectability limit of elements (with EDS) compared to wet analytical methods.
5. Samples need to be examined under vacuum.
6. The instrument typically requires an installation space of 5×5 m.
7. Non-conductive samples need to be coated.

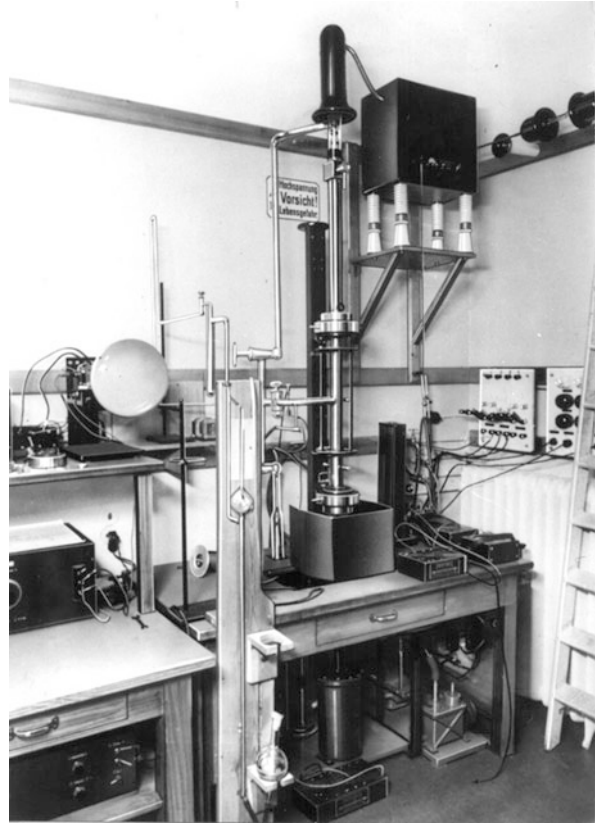
1.6 Brief History of the SEM Development

Limitation of light microscope in resolving fine details of organics cells provided the impetus to develop an electron microscope in the early twentieth century. The first electron microscope was a transmission electron microscope developed by German scientist Max Knoll and electrical engineer Ernst Ruska in Germany in 1931. It employed a working model similar to light microscope except that a beam of electrons, instead of visible light, was made to pass through the body of a sample to form an image on a fluorescent screen. Use of electrons as an imaging medium afforded a resolution of 10 nm compared to a resolution of 200 nm achievable by a light microscope. The resolution achieved at the time might seem modest today, but the real breakthrough was the fact that for the first time in history, electrons had been successfully employed to create images of matter. Subsequent improvements in the accelerating voltage, lens technology, vacuum systems, electron guns, power supplies, and overall design of the microscopes in the next few decades led to the imaging of atoms (i.e., atomic resolution was achieved). Due to his “fundamental work in electron optics and for the design of the first electron microscope,” Ernst Ruska received a Nobel Prize in physics in 1986.

German physicist Max Knoll introduced the concept of a scanning electron microscope in 1935 [1]. He proposed that an image can be produced by scanning the surface of a sample with a finely focused electron beam. Another German physicist Manfred von Ardenne explained the principles of the technique and elaborated upon beam-specimen interactions. He went on to produce the earliest scanning electron microscope in 1937 [2–4] as shown in Fig. 1.6.

Later, the SEM with a resolution of 50 nm was built by American scientists Zworykin, Hillier, and Snijder in 1942 [5] and later by Professor Sir Charles W. Oatley and his postgraduate student D. McMullan at the University of Cambridge

Fig. 1.6 First scanning electron microscope built by Manfred von Ardenne in 1937



in 1952 [6]. Scintillator-based secondary electron detector was developed by Everhart and Thornley in 1960 [7]. Further improvements in the technology led to the development of first commercial SEM known as “Stereoscan” by Cambridge Scientific Instruments in 1965 [8, 9]. The SEMs built in the 1960s had a resolution of about 15–20 nm. In the 1970s and 1980s, the resolution was improved to 7 nm and 5 nm (at 1 kV), respectively. Next couple of decades saw resolution improvements down to 3 nm and then to 1 nm. Currently, manufacturers claim resolutions of 0.5 nm in the SEM. Although the scanning electron microscope was developed subsequent to the transmission electron microscope, the former quickly became popular due to its ease of use, simple sample preparation, and ability to generate 3-D like images of the sample topography. A detailed history of the SEM development has been documented by some authors [10–13]. Main events in the development of SEM techniques and instrumentation are listed in a chronological order in Table 1.4 [10–14]:

Table 1.4 Development of SEM instrumentation and techniques in a chronological order [10–14]

Year	Key developments	Contributors
1935	The concept of the SEM	M. Knoll (1935)
1938	Concept and development of the scanning transmission electron microscope (STEM)	M. von Ardenne
1942	Development of the SEM with 50 nm resolution	Zworykin et al.
1952	Development of the SEM with 50 nm resolution	D. McMullan and Prof. C. Oatley
1956	Signal processing, double deflection scanning, stigmator (improved image quality)	K. C. A. Smith [15]
1960	Scintillating secondary electron detector, photomultiplier tube (improved signal-to-noise ratio)	Everhart and Thornley [7]
1957	Observation of voltage contrast	Oatley and Everhart [16]
1960	Stereographic 3-D SEM images	O. C. Wells [17]
1963	Development of “SEM V” microscope with three magnetic lenses and Everhart-Thornley (ET) detector	R. F. W. Pease [8]
1963–1965	Development of first commercial SEM “Stereoscan”	R. F. W. Pease and Nixon [8, 9]
1970s	Microchemical analysis using energy dispersive x-ray spectrometer (EDS) coupled with the SEM	Contributions from various individuals and commercial manufacturers
1970s	Lanthanum hexaboride (LaB ₆) cathode gun	
1970s	Field emission gun	
1970s	Electron backscattered diffraction (crystal structure and grain orientation)	
1970s	Cathodoluminescence in the SEM	
1973	Hot stage to examine samples at elevated temperatures	
1970s, 1980s	Large specimen stages to hold sample sizes up to 23 cm	
1980s	Auto focus and auto stigmator functions	
1980s	Yttrium doped silicate scintillator as backscattered electron detector	
1980s	Low temperature (Cryo) stage	
1990s	Variable pressure SEM (for charging samples)	
Since 1990s	Improved automation and analysis due to computers	
2000 to date	High resolution microscopes to study nanomaterials	

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