#### 1. Scanning electron microscope (SEM):

<u>Objective:</u> uses electrons that are reflected off the near-surface region of a sample to create an image. It also projects and scans a focused stream of electrons over a surface to create an image.

<u>Operating principle:</u> Because the maximum accelerating voltage (typically 30 kv) is lower than for a TEM, the electron gun is smaller, requiring less insulation. Axially symmetric magnetic lenses are used but they too are smaller than the lenses employed in the TEM; for electrons of lower kinetic energy, the polepieces need not generate such a strong magnetic field. There are also fewer lenses; image formation utilizes the scanning principle.

The incident beam in the SEM (also known as an electron probe) needs to be as small as possible: a diameter of 10 nm is typical and 1 nm is possible with a field-emission source. The final lens that forms this very small probe is named the objective and its performance (including aberrations) largely determines the spatial resolution of the SEM.

The electron probe of an SEM is scanned horizontally across the specimen in two perpendicular (x and y) directions.

The x-scan is relatively fast and is generated by a sawtooth-wave generator operating at a line frequency fx. This generator supplies current to two scan coils, connected in series and located on either side of the optic axis, just above the objective lens. These coils generate a magnetic field in the y-direction, creating a force on an electron (traveling in the zdirection) that deflects it in the x-direction.

The y-scan is much slower (Fig. 5.2b) and is generated by a second sawtooth-wave generator running at a frame frequency fy = fx/n where n is an integer (the number of lines per frame). The entire procedure is known as raster scanning and causes the beam to sequentially cover a rectangular area on the specimen.

During its x-deflection signal, the electron probe moves in a straight line, from A to B forming a single line scan.

After reaching B, the beam is deflected back along the x-axis as quickly as possible (the flyback portion of the x-waveform). Because the y-scan generator has increased its output during the line-scan period, it returns not to A but to point C, displaced in the y-direction. A second line scan takes the probe to point D, at which point it flies back to E and the process is repeated until n lines have been scanned and the beam arrives at point Z. This entire sequence constitutes a single frame of the raster scan. From point

Z, the probe quickly returns to A, due to rapid flyback of both the line and frame generators, and the next frame is executed. This process can run continuously for many frames, as in television and video technology.

The outputs of the two scan generators are also applied to the display device on which the SEM image appears, which was originally a cathode-ray tube (CRT). This CRT contained an electron beam that was scanned exactly in synchronism with the beam in the SEM, so for every point on the specimen (within the raster-scanned area) there was an equivalent point on its display screen, thereby satisfying Maxwell's first rule of imaging. In order to create contrast in the image, a voltage signal was applied to the electron gun of the CRT, to vary the brightness of the scanned spot. This voltage was derived from a detector that responded to some change in the specimen induced by the SEM incident probe.

In a modern SEM, the scan signals are generated digitally, by computer controlled circuitry, and the x- and y-scan waveforms are staircase functions with m and n levels, respectively. This procedure divides the image into a total of mn picture elements (pixels) and the SEM probe remains stationary for a certain dwell time before jumping to the next pixel. One advantage of digital scanning is that the SEM computer "knows" the (x, y) address of each pixel and can record the appropriate image-intensity value (as a digitized number) in the appropriate computer-memory location. The digital image, in the form of position and intensity information, is stored in computer memory and more permanently on a magnetic disk or other storage device. The modern SEM uses a flatpanel display screen in which there is no internal electron beam. Instead, computergenerated voltages are used to sequentially define the x- and y-coordinates of a screen pixel and the SEM detector signal is applied electronically to that pixel, to change its brightness. In other respects, the raster-scanning principle is the same as for a CRT display. Image magnification in the SEM is achieved by making the x- and y-scan distances on the specimen a small fraction of the size of the displayed image, since by definition the magnification factor M is given by.

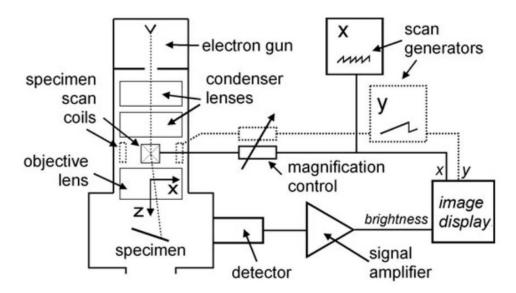
M = (scan distance in the image) / (scan distance on the specimen)

It is convenient to keep the image at a fixed size, just filling the display screen, so increasing the magnification involves reducing the x- and y-scan currents, each in the same proportion (to avoid rectangular distortion). Consequently, the SEM is actually working at its hardest (in terms of current drawn from the scan generator) when operating at low magnification.

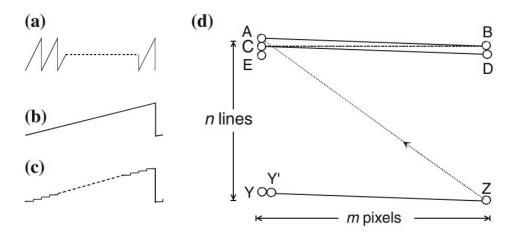
The scanning is sometimes done at video rate (around 50 or 60 frames/second) to generate a rapidly refreshed image that is useful for focusing the specimen or for viewing it at low magnification. At higher magnification, or when making a permanent

record of an image, slow scanning (several seconds per frame) is possible; the additional recording time results in a higher-quality image containing less electronic noise.

The signal that modulates (alters) the image brightness can be derived from any property of the specimen that changes in response to electron bombardment. Most commonly, the emission of secondary electrons (atomic electrons ejected from the specimen as a result of inelastic scattering) is utilized. Alternatively, a signal derived from backscattered electrons (incident electrons elastically scattered through more than 90°) is used. In order to understand these (and other) possibilities, we need to consider what happens when an electron beam enters a thick (often called bulk) specimen.



**Fig. 5.1** Schematic diagram of a scanning electron microscope. The same x- and y-scan waveforms are applied to the SEM column and to the display device. Signal from a detector modulates the brightness of the display



**Fig. 5.2** a Line-scan waveform (scan current versus time). **b** Analog frame-scan waveform and **c** its digital equivalent. **d** Elements of a single-frame raster scan: AB and YZ are the first and last line scans in the frame, Y and Y' represent adjacent pixels

#### 2. Transmission electron microscope (TEM):

<u>Objective:</u> TEM is a microscopy technique in which a beam of electrons is transmitted through a specimen to form an image. TEM is a technique used to observe the features of very small specimens like nanomaterials or aerosol particles. Tems are capable of imaging at a significantly higher resolution than light microscopes.

It is used to view thin specimens (tissue sections, molecules, etc) through which electrons can pass generating a projection image. Also, it uses a particle beam of electrons to visualize specimens and generate a highly-magnified image. The TEM has been equally helpful in the life sciences like it can be used to examine plant and animal tissue, bacteria, and viruses

# Operating principle:

Above the specimen, there are typically two or three lenses, acting rather like the condenser lenses of a TEM. But whereas the TEM (if operating in its conventional imaging mode) produces a beam of diameter ≈1 µm or more at the specimen

The conventional TEM uses a stationary incident beam.

Some early TEMs used a gas discharge as the source of electrons but this was soon replaced by a V-shaped filament made from tungsten wire, which emits electrons when heated in vacuum. The vacuum was generated by a mechanical pump together with a

diffusion pump, originally made of glass and containing boiling mercury. The electrons were accelerated by applying a high voltage generated by an electronic oscillator circuit and a step-up transformer.

A modern TEM (resolution below 0.2 nm), individual atomic planes or columns of atoms can be distinguished. Modern TEMs use electron-accelerating voltages between 60 and 300 kV, high-voltage instruments (HVEMs) have been constructed with voltages up to several MV.

**Fig. 1.8** Early photograph of a horizontal two-stage electron microscope (Knoll and Ruska 1932). This material is used by permission of Wiley-VCH, Berlin

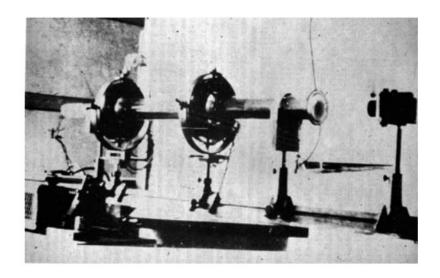
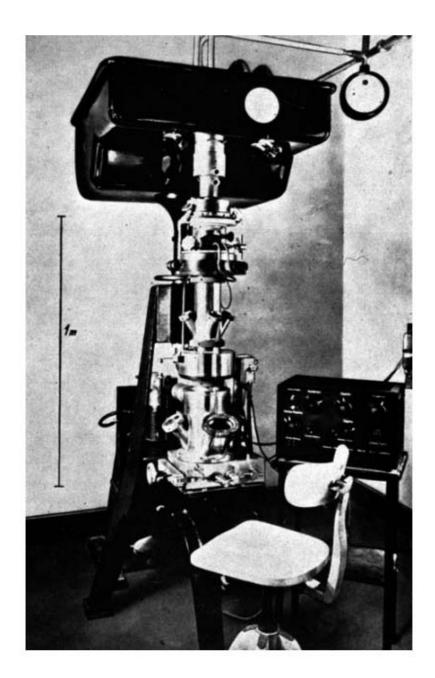
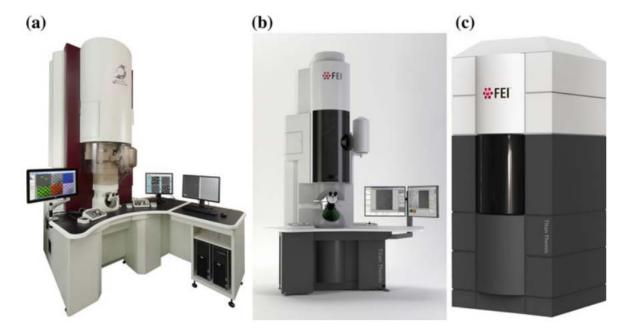


Fig. 1.9 First commercial TEM from the Siemens Company, employing three magnetic lenses that were water-cooled and energized by batteries. The objective lens had a focal length down to 2.8 mm at 80 kV, giving an estimated resolution of 10 nm





**Fig. 1.10** Two recent transmission electron microscopes: **a** the JEOL Grand ARM (300 kV, 63 pm resolution with aberration correction), **b** the FEI TITAN Themis, which can reach 60 pm resolution at 300 kV if fitted with aberration correction, and whose 5.4 mm objective-lens polepiece gap allows in-situ experiments

#### 3. Scanning Transmission Electron Microscopy (STEM):

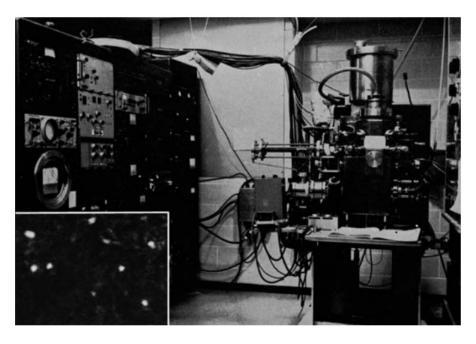
<u>Objective:</u> Scanning transmission electron microscopes (STEM) are used to characterize the nanoscale and atomic-scale structure of specimens, providing important insights into the properties and behaviour of materials and biological cells.

## **Operating principle:**

The fine-probe/scanning technique can also be used with a thin (transmission) specimen, the result being a scanning-transmission electron microscope (STEM). Rather than secondary electrons, it is usual to record the primary electrons that are scattered in a particular direction and emerge from the beam-exit surface.



**Fig. 1.15** Hitachi-SU5000 scanning electron microscope. This instrument uses a Schottky-emission source and provides a resolution of 1.2 nm at an accelerating voltage of 30 kV or 3 nm at 1 kV (2 nm in deceleration mode). Variable pressure mode (10–300 Pa) is an option



**Fig. 1.16** Photograph of the Chicago STEM and (*bottom-left inset*) an image of mercury atoms on a thin-carbon support film. Courtesy of Dr. Albert Crewe (personal communication)

### 4. Analytical Electron Microscope (AEM):

**Objective:** provide high-resolution information about the structure of a specimen.

<u>Operating principle:</u> As atomic number Z increases, the nuclear charge increases, drawing electrons closer to the nucleus and changing their energy. Outer (valence) electrons are affected by the chemical bonding with nearby atoms but the inner-shell electrons are not, so their energies can provide an indication of the nuclear charge and therefore atomic number. When an inner-shell electron makes a transition from a higher to a lower energy level, the atom can emit an x-ray photon whose energy (hf = hc/λ) is equal to the difference in the two quantum levels. This property is utilized in an x-ray tube, where primary electrons bombard a solid target (the anode) and excite inner-shell electrons to a higher energy, followed by the de-excitation process in which characteristic x-rays are generated.

In a similar way, the primary electrons entering a TEM, SEM, or STEM specimen cause x-ray emission, and by identifying the wavelengths or photon energies of these x-rays, we can perform elemental analysis.

Other forms of AEM make use of Auger electrons emitted from the specimen with characteristic energies, or the primary electrons themselves after they have traversed a thin specimen and have lost characteristic amounts of energy.