

Imaging Laboratory Exercise

using a

Scanning Electron Microscope

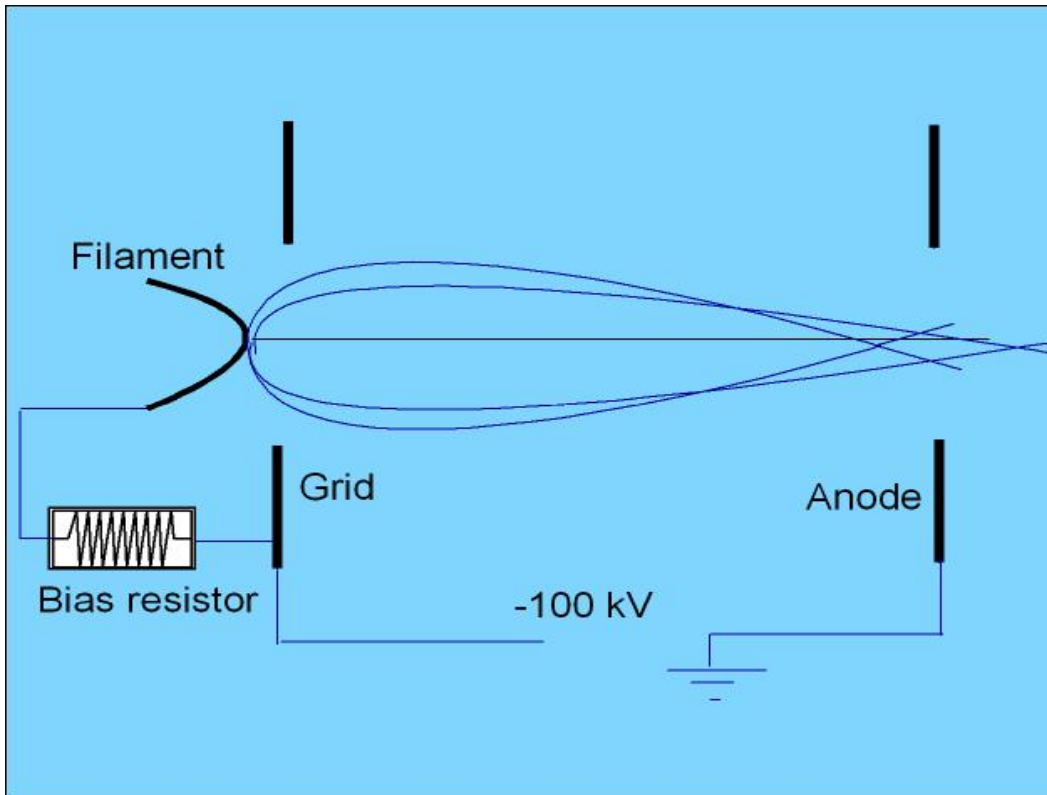
Anton van Leeuwenhoek of Holland (1632-1723) is known as the father of the microscope. He developed methods of polishing to create small lenses with high curvatures and large magnification. He used these lenses in microscopes and was the first who reported observing bacteria and other forms of life previously unseen. Today better understanding of optics, the science of light, and development of new methods and techniques in making better optical instruments has brought us to a point where our ability to see small objects under a microscope is only limited by the physics of optics. This limit, called the diffraction limit, is due to the bending of light (diffraction) around objects whose dimension is comparable to the wavelength of the illuminating light. As a result of this limitation, the optical microscope's resolution is roughly $0.1 \mu\text{m}$ - too large to let us see even viruses, let alone atoms. To read more about this limitation, visit the pages on [Limiting Vision](#). This limit is roughly the size of the wavelength of the probe (i.e. light for visible microscopes). deBroglie's wave description of matter objects allows a way out of this limitation. The electron's mass is 9.11×10^{-31} kg. So, even a fast moving electron's momentum is small enough to produce a rather small deBroglie wavelength (see pages on [Quantum Mechanics](#) for further explanation on deBroglie waves). In this way we could probe nm scale objects using electrons. This was utilized back in the 1930s when the first electron microscope was invented. This invention was most likely responsible for Richard Feynman's vision of the bottom-up approach. But it took another 50 years and the invention of Scanning Tunneling Microscope (probe microscopy) to open up the new investigations in Nanotechnology. Today a number of imaging devices allow researchers and technologists to create pictorial representations of objects as small as a single atom. In our laboratory experiment we will create images of some test objects using a Scanning Electron Microscope (SEM).

Electron Microscope

This device operates similarly to a conventional optical microscope. Instead of imaging photons, as in the visible microscope, electron microscopes use electrons. In these instruments electron beams are accelerated to tens or hundreds of kilo-electron volts (keV). Using electric and magnetic fields the moving charged electrons are manipulated same as photons are in conventional optics. These instruments operate in two forms: transmission and scanning. In the Transmission Electron Microscope (TEM) the electron beam passes through the object being examined and then is imaged on a sensitive detector. In the Scanning Electron Microscope (SEM) the beam is scanned over the object and the reflected (scattered) beam is then imaged by the detector. In the case of TEM the sample needs to be very thin so as to allow for the passage of the electrons. The SEM requires highly conductive samples so as not to create blurring due to the electric field of deposited charges. (In both of these instruments high resolution imaging requires a conductive sample.) In most applications, the sample is coated with a thin layer of a metal, such as gold, before it is imaged in the microscope. Because of this, these devices have a limited application, especially for examining dynamic samples, such as in life forms.

In both SEM and TEM electrons are generated in the portion of the microscope that is referred to as the electron gun. There are two processes used to generate an electron beam. The most common of these is thermionic emission. In this method a metal with a high melting point (tungsten 3643 or molybdenum 2890 K) wire is heated by passing an electric current through it. At temperatures of around 2200 K, in addition to light, electrons are ejected from the metal surface similar to when water molecules leave the surface of a pan of water as it gets hotter and hotter. To create a beam of electrons the filament (heated wire) is held at a voltage below (negative biased) another metal plate that has a hole to allow the electrons to exit. In electron microscopes the gun is in fact a triode made of the filament (cathode) a

negatively biased grid (often called the Wehnelt grid) and a positively biased anode plate (see diagram below). Thus, the effect of the grid is to concentrate the ejected electrons through a small opening that serves as the effective illuminating spot size of the electron beam.



The second method for creating an electron beam is called field emission. The field emitting gun is similar to the one described above, but the filament is made to have a very sharp tip. Also, the grid is held at a much higher potential so that the electrons at the tip experience a very large electric field that draws them away from the tip. The cathode is then used to accelerate the ejected electrons. The effect of the high field at the sharp tip is to lower the barrier height and to allow for tunneling of electrons across the vacuum barrier. This process results in the creation of far larger electron currents than is possible by thermionic emission. Although an aside, it is worth noting that thermionic emission is a self limiting process. This is not the case for field emission.

It is important to note that the large accelerating voltages used in electron microscopes create electrons moving at velocities for which classical mechanics no longer applies. So, to calculate electron's dynamical variables we then need to rely on Einstein's Relativity Theory. As is illustrated in the table below, even at an acceleration potential of 60 kV relativity makes more than a 2% correction.

Accelerating potential (kV)	λ (nm) classical	λ (nm) relativistic
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

To calculate the electron wavelength with the relativistic correction we can use the relativistic expressions for the mass and energy to obtain the relativistic relation:

$$\lambda = [1.5 / (V + 10^{-6} V^2)]^{1/2} \text{ nm}$$

Once electrons are emitted from the gun they are bent by electrodes and electromagnets and brought to focus in the same way that light is refracted in optical lenses. In the case of an electron beam, it is the Lorentz force that bends the electron path:

$$\mathbf{F} = q \mathbf{E} + q \mathbf{v} \times \mathbf{B}$$

In electron microscopes a variety of electromagnets (condenser lens, zoom condenser lens, objective lens, etc.) are used for their optics. In almost all of these, the role of these magnets is the same as the optical elements in a standard optical microscope.

For the transmission electron microscopes (TEM) the principle of image formation is very similar to that of an optical microscope. In the TEM the electron beam passes through a thin ("transparent") sample and then forms an image of the sample on a detecting screen. This screen could be a phosphorescent target that glows (i.e. emits light) once electrons strike it, very similar to conventional CRT (cathode ray tube) television screens. In the TEM the magnification of the image is a function of the electron "optics". However, the image created on the fluorescent screen could receive further magnification using a standard optical design. It is worth noting that in TEM it is the diffraction effect that plays the role of refraction in the optical imaging. Also worthy of notice is that the energetic electron beam could lead to other processes and thus produce secondary effects, such as X-ray emission, that could be used for elemental analysis of the sample.

There are a wide variety of TEMs, each suited for a particular application. In general, the accelerating potential ranges from 60 to 200 keV for most common machines. Medium range machines accelerate electrons in the 300 to 400 keV, and the high end machines go as high as 600 to few mega-electron volts. (The Toulouse electron microscope goes as high as 3.5 MV.) The primary advantage of using higher energy electron beams is to penetrate thicker samples. The 100 to 125 kV machines require sample thicknesses no more than a few nm for providing high resolution images. With the MV machines samples as thick as 0.1 mm could be imaged with relatively good resolution. Still, a primary drawback is sample damage at these high energies. Another obvious drawback of these ultra high energy machines is their construction cost and their large operating expenses.

All of these microscopes require good vacuum environments for protecting their electron beam from interact with background matter (air). (See the table to the right for more technical information about vacuum requirements.) Because of this all of the beam generation, steering, and detection apparatus must be enclosed in vacuum maintaining enclosures. Typically, these enclosures are thick stainless steel vessels that are continuously pumped using a collection of vacuum pumps. As the accelerating voltages increases high and ultrahigh vacuum becomes necessary and cause the size of the microscope to increase substantially. The MV microscopes can get as tall as 10 meters or taller.

Image formation in the scanning electron microscope (SEM) is very different. In the SEM a well focused electron beam is scanned in parallel lines, rasters, over the sample. Reflected (and secondary) electrons emitted from the sample are then collected by a fixed detector. Although different SEMs may use different types of electron detectors (electron counters), it is ultimately the number of electrons collected from the

target that represent a measure of the image. The collected electron signal acts as a signature of the target at the point where the incident electron beam strikes. Typically, the collected electrons strike a scintillator (phosphorous) that emits light. This light is collected with the aid of a highly sensitive detector, such as a photomultiplier. The photomultiplier signal is further amplified, using conventional electronics, and is used to recreate an image. The ultimate resolution of the SEM is thus very much a function of how tightly the impinging electron beam is focused on the sample and how it is rastered over it. Because of this and that reflected electrons form a less compact region of emission, the secondary electrons are the more preferred signal for imaging.

The primary advantage of SEM over TEM is that it can image totally non-transparent (opaque) samples. It is therefore the electron microscope of choice for materials characterization, while TEM is most often used for imaging biological samples. However, SEM samples also need to be conductive otherwise electrons can collect on the sample and interact with the electron beam itself resulting in blurring. As stated earlier, nonconductive samples have to first receive a thin metal coating. Gold or aluminum are the most commonly used coatings to cover nonconductive samples. The process of metal coating, of course, does not allow imaging of living/evolving samples. But similar to TEM the scanning electron microscope can also produce X-rays indicative of sample composition, as well as sample characterization using X-ray diffraction.

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