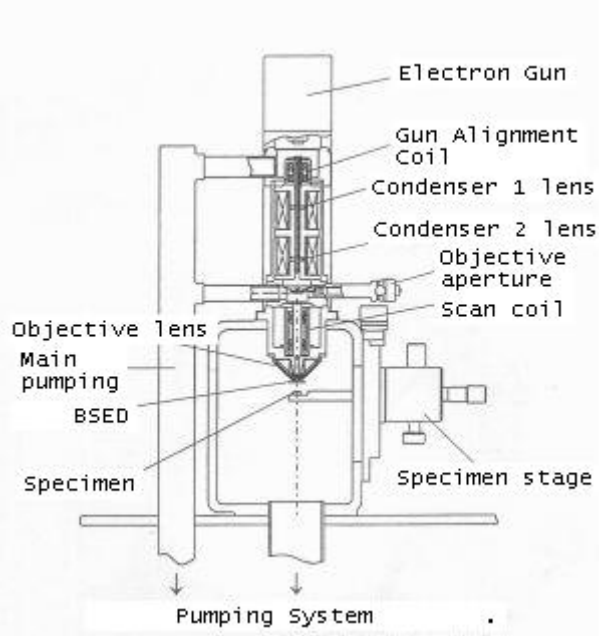
Scanning Electron Microscope (SEM)

(JEOL JSM-6320F, Hitachi S-3000N)

A major limitation of TEM is that, unless the specimen can be made very thin, electrons are strongly scattered reducing resolution or absorbed in the specimen rather than transmitted. Early in the development of electron microscopy it was recognized that a technique that could look at the surfaces of *bulk* specimens at higher resolutions possible in a reflection optical microscope would be useful.

The first scanning electron image published was acquired by Knoll (1935) who used a television tube with an electron source at one end and a target at the other to generate a low magnification image (<x10). The first scanning application with electromagnetic lenses was a Scanning Transmission Electron Microscope (STEM) built by von Ardenne in 1938. With this instrument he showed where detectors could be placed not only for STEM imaging but also SEM imaging (*returning electrons*). During the 1940s Zwyorkin worked on developing an SEM at RCA Labs in New Jersey. Starting from the electron tube approach he tried field emission and electrostatic lenses to give a resolution of 50nm but it was not commercialized. Oatley and colleagues at Cambridge University began a research and construction program after the war culminating in a working SEM in 1951, and a commercial model (built by AEI) in 1958. Sustained commercial production of SEM began in 1965 with Cambridge instruments (Stereoscan).

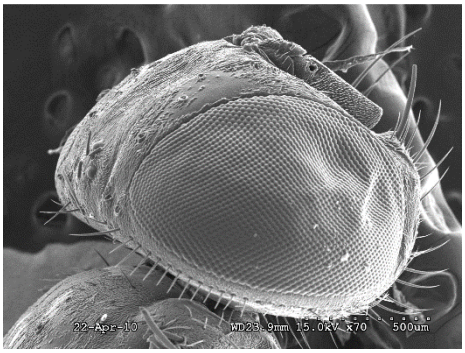
In an SEM, a fine probe of electrons is formed at the surface of a specimen. This electron probe is then scanned over the specimen and an image is built up, point by point, on a monitor. The electrons collected can be either the **secondary electrons** ejected from the atoms by the inelastic scattering of the primary electrons, or primary electrons that are scattered through high angles by one or more near nucleus collisions (**backscattered electrons**) back up the column. Secondary electrons are defined purely on the basis of their kinetic energy (<50eV), a majority of backscattered electrons will have kinetic energies of more than half the accelerating voltage. Unlike specimens for transmission microscopy, the SEM specimen does not have to be thin enough for electrons to travel through, and SEMs are routinely used to look at the surfaces of bulk specimens. The maximum specimen size for a SEM will depend on the make and model of microscope used. For the Hitachi S-3000N the maximum size is 150mm diameter x 20mm high, although it is not be possible to reach all areas of a specimen that large, and for the JEOL JSM-6320F 32mm diameter x 10mm high.



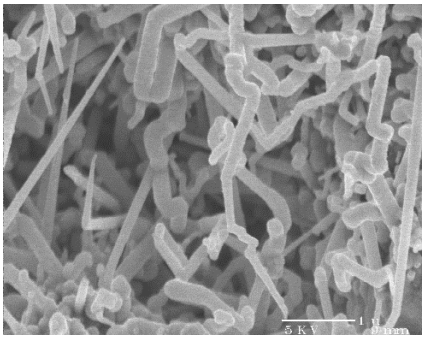
The SEM usually comprises of the following components, in vacuum:

- An electron source to provide the illumination. This can be either a heated filament (tungsten hairpin or LaB_6) or a sharp tip, from which electrons are "pulled out" by the action of a strong electric field (field emission). Most conventional SEMs have a heated filament, field emission sources are used for high spatial resolution imaging (JSM-6320F). The source will have a single stage accelerator to increase the electron beam energy to a maximum of 30kV.
- An illumination optical system consisting of two electromagnetic condenser lenses. These lenses are controlled to give, with the objective lens, a range of focused cross overs at the specimen from large probe size, high current to small probe size, low current.

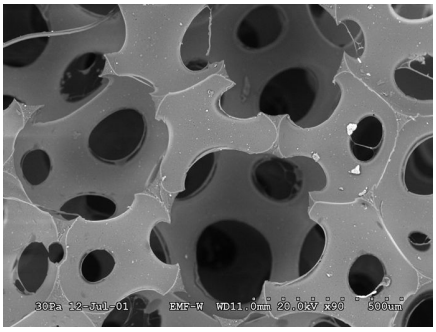
- A set of coils which allow the beam to be scanned over the specimen. Reducing the area scanned on the specimen increases the magnification of the image.
- A specimen stage and objective lens. The specimen is mounted below the field of the objective lens. The design and properties of this lens and the distance from the lens to the specimen control the resolution obtainable by the microscope. The stage can have five axes of movement (X, Y, Z shifts, tilt and rotation).
- A detection system. Usually there are secondary (SED) and backscattered (BSD) electron detectors. These are typically below the objective lens, and limit the ability to use short working distances. On the JSM-6320F there is a secondary detector within the objective lens bore and higher resolutions are possible at low accelerating voltages with very short working distances (Semi-in-lens). The secondary detector gives very good topographical information, while a backscattered detector gives a compositional image. Usually also fitted with a X-ray Energy Dispersive Spectrometer (XEDS) to collect X-rays generated by the electron beam hitting the sample.



Secondary electron image of a Pt/Pd coated bug head



Secondary electron image of Germanium nanowires



Backscattered electron image of uncoated Viscoelastic Sponge in VP mode

In a conventional SEM (JSM-6320F) the specimen must either conduct, or the charge build up must be balanced (by operating at low accelerating voltage). In many cases this is not possible and the specimen must be coated with a conducting film (carbon, gold, chromium). The coating may, however, hide the information you are looking for. In a variable pressure SEM (S-3000N) the specimen can be observed in high vacuum or in a poorer vacuum between 1-270Pa (Variable Pressure (VP) mode). In this higher pressure regime the incident electron beam ionizes gas atoms near the specimen which are attracted to, and neutralize any charge build up. Nearly all dry specimens can be looked at without any preparation or coating. Because of the poor vacuum the conventional SED cannot be used due to the high voltages associated with the detector. The VPSEM primarily uses a segmented BSD which allows compositional and topographic imaging, however the microscope does also have a VPSED.

The interaction of the beam with the specimen also gives rise to X-rays, which can be collected using an XEDS detector. However, depending on the accelerating voltage, the spatial resolution for microanalysis can be as much as 1um, even if the imaging resolution is sub-10nm. In low vacuum mode the incident beam can be significantly scattered by the gas which can degrade the spatial resolution for X-ray analysis to as much as 1mm.