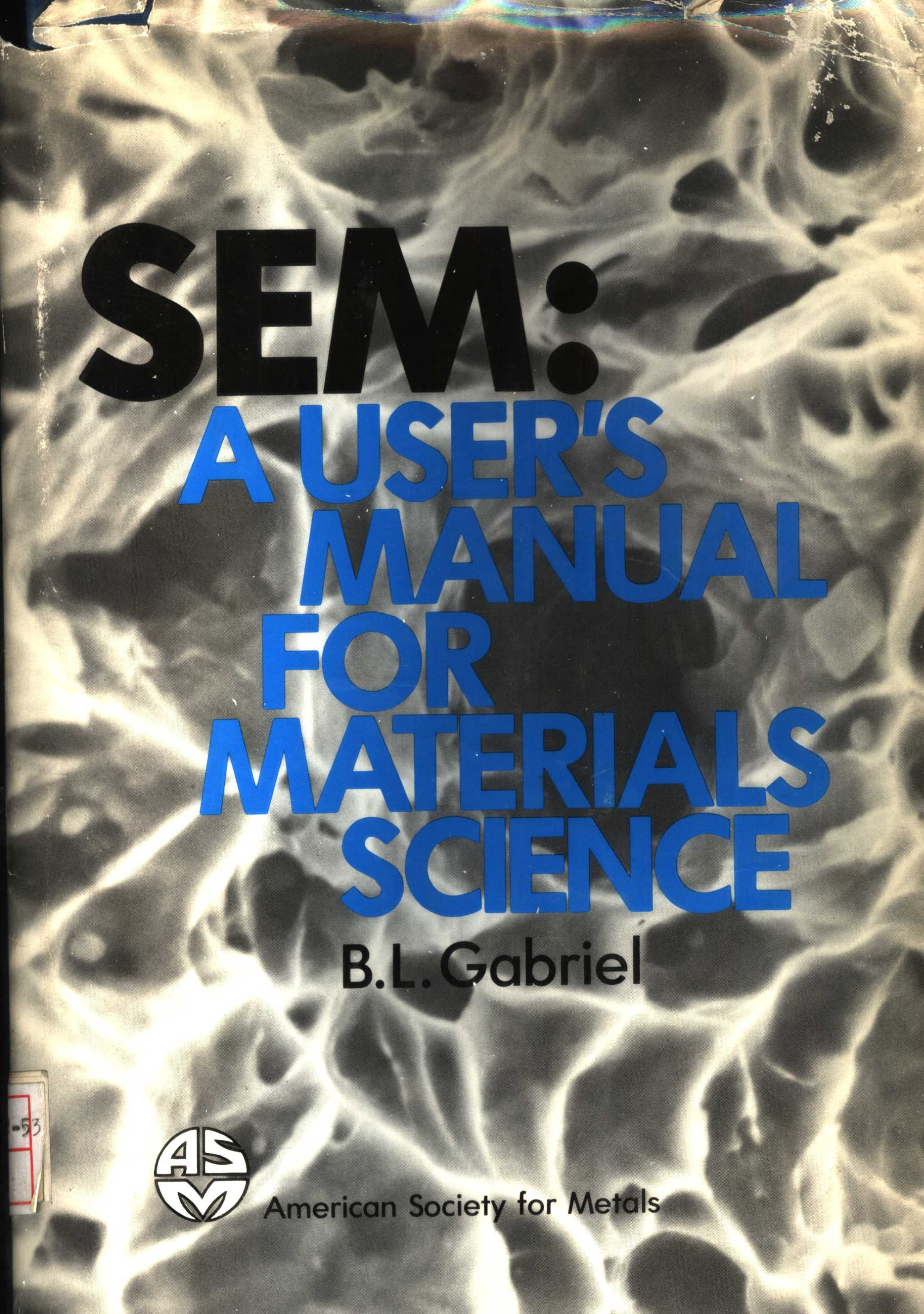

# SEM: A USER'S MANUAL FOR MATERIALS SCIENCE

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American Society for Metals

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## *A User's Manual for Materials Science*

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*In memoriam*

ANTHONY R. GABRIEL

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## Part 1: Instrumentation

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# 1

# The Scanning Electron Microscope

The scanning electron microscope (SEM) has unique capabilities for analyzing surfaces. It is analogous to the reflected light microscope, although different radiation sources serve to produce the required illumination. Whereas the reflected light microscope forms an image from light reflected from a sample surface, the SEM uses electrons for image formation. The different wavelengths of these radiation sources result in different resolution levels: electrons have a much shorter wavelength than light photons, and shorter wavelengths are capable of generating higher-resolution information. Enhanced resolution in turn permits higher magnification without loss of detail. The maximum magnification of the light microscope is about 2000×; beyond this level is "*empty magnification*," or the point where increased magnification does not provide additional information. This upper magnification limit is a function of the wavelength of visible light, 2000 Å, which equals the theoretical maximum resolution of conventional light microscopes. In comparison, the wavelength of electrons is less than 0.5 Å, and theoretically the maximum magnification of electron beam instruments is beyond 800,000×. Because of instrumental parameters, practical magnification and resolution limits are ~75,000× and 40 Å in a conventional SEM.

Another difference between light and scanning electron imaging concerns the *depth of field,* defined as the ability to maintain focus across a field of view regardless of surface roughness. Human binocular vision permits observation and interpretation of depth of field in three-dimensional objects. Conventional photographs and photomicrographs are two-dimensional representations; the dimension of depth is suppressed when recording an image with a diffuse light source. In contrast, SEM micrographs maintain the three-dimensional appearance of textured surfaces, a phenomenon due to the high depth of field of scanning instruments. Depth of field is further suppressed in both macrophotography and photomicrography as magnification is increased. At 10×, the relative depth of field of a light microscope is about 250 μm, while that of the SEM is about 1000 μm; at 1200× the depth of field of a light microscope is ~0.08 μm; at 10,000×, the depth of field of the SEM is 10 μm. Thus, photographers are often challenged to record rough surfaces while maintaining depth of field (through determination of the "optimal aperture"); scanning electron microscopists readily record smooth or rough surfaces.

The combination of high resolution, an extensive magnification range, and high depth of field makes the SEM uniquely suited for the study of surfaces. As such, it is an indispensable tool in materials science research and development. Microscope instrumentation and operation are described in this chapter, and subsequent chapters cover energy-dispersive spectroscopy, sample preparation, and applications of SEM in materials science.

## SEM INSTRUMENTATION

The SEM (Fig. 1-1 and 1-2) consists basically of four systems:

1. The *illuminating/imaging system* produces the electron beam and directs it onto the sample.
2. The *information system* includes the data released by the sample during electron bombardment and detectors which discriminate among and analyze these information signals.
3. The *display system* consists of one or two cathode-ray tubes for observing and photographing the surface of interest.
4. The *vacuum system* removes gases from the microscope column which would otherwise interfere with high-resolution imaging.

Each of these systems and their relationships are discussed below. In addition, both the theoretical and practical aspects of microscope operation are analyzed.

## ILLUMINATING/IMAGING SYSTEM

The illuminating/imaging system comprises an electron gun and several magnetic lenses that serve to produce a collimated, coherent beam of electrons which can be focused onto the specimen (Fig. 1-3).

### Electron Gun

The electron gun can be subdivided into (1) a *filament* (cathode) or electron source, which generates electrons and is thus held at a negative potential with respect to ground; (2) an apertured *shield* at slightly

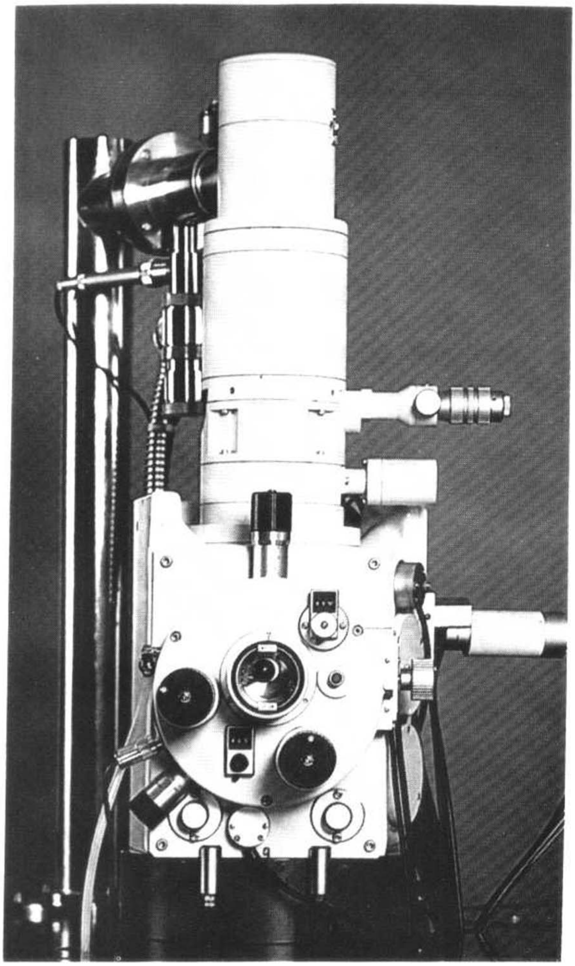

**Fig. 1-1. The JSM-840 scanning electron microscope. (Courtesy of JEOL)**

positive potential relative to the filament; and (3) an *anode* held at very high positive potential with respect to the filament. Together these components function as an electrostatic lens: electrons are produced by passing a current through the filament and heating it to a point where the voltage gradient between the filament and anode produces electrons, which are then accelerated by the potential difference between the anode and filament.

The *tungsten hairpin filament* is the most common type of electron source in use today. Although analogous to incandescent light bulb filaments, SEM filaments are designed to carry a much higher voltage (10,000-20,000 volts) than light bulbs. A 0.125-mm tungsten wire is shaped into a hairpin and soldered to two electrodes which are connected to the high-voltage system (Fig. 1-3). A ceramic insulator prevents arcing between the filament and high-voltage source. Tungsten

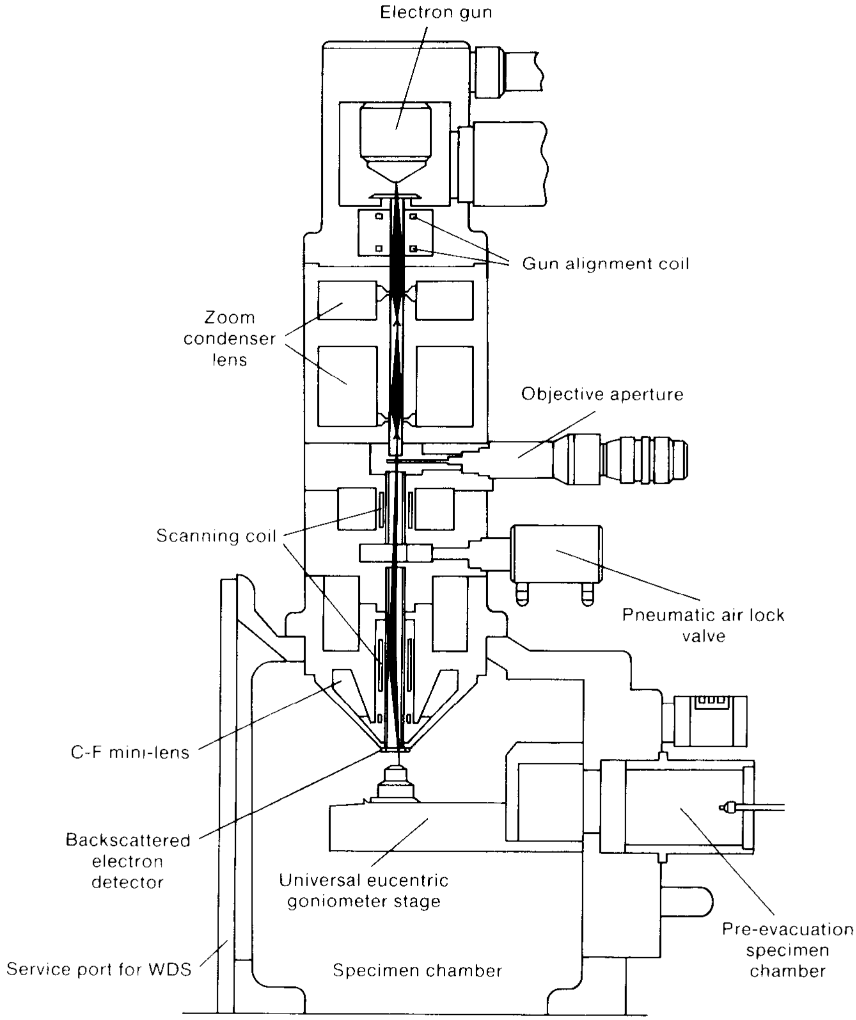


**Fig. 1-2. Schematic cross section of the JSM-840 SEM shown in Fig. 1-1. (Courtesy of JEOL)**

filaments are extremely popular because they operate under high (as opposed to ultrahigh) vacuum ($\geq 10^{-4}$ torr), they are inexpensive, and they are easy to exchange. In a well-operated microscope, tungsten filaments will function for ~40 hr before failing.

Nonconventional electron sources include *lanthanum hexaboride* ($LaB_6$) and *field emission* (FE) guns, both of which are much brighter than tungsten cathodes. The typical operating brightness for each source is as follows: tungsten hairpin, $5 \times 10^4$ to $10^5$ A/cm$^2$ steradian; $LaB_6$, $6 \times 10^6$ A/cm$^2$ steradian; FE, $10^7$ to $2 \times 10^8$ A/cm$^2$ (Table 1-1). Both types of sources, field emission in particular, offer advantages in high-resolution SEM, but they have not replaced tungsten filaments because modification of the SEM vacuum system is required for ultrahigh vacuum, special care must be exercised in operation and maintenance of

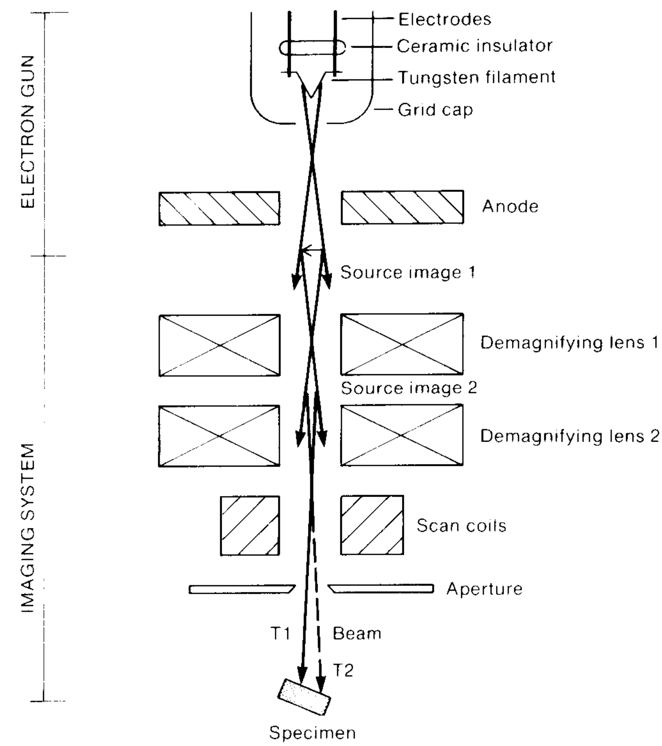


**Fig. 1-3. Cross section of the illuminating/imaging system.**

these guns, and both are expensive options. Table 1-1 summarizes the characteristics of these cathodes; further information on FE and $LaB_6$ guns may be found in Broers (1975).

Returning to our discussion of the conventional electron gun, surrounding the tungsten filament is the *shield* (synonyms: grid cap, Wehnelt cylinder). The shield is a slightly biased cylindrical cup which serves to collimate the electrons from the filament and direct them toward the anode. The round opening of the shield is centered over the filament tip. The distance separating the tip and shield (1-2 mm) is critical in that it controls beam current. A *bias* voltage is applied to the shield which allows only those electrons emitted from the tip of the filament to form the beam. If the gap between the filament tip and the grid cap is too small, the filament will quickly burn out; if the two

**Table 1-1. Characteristics of Electron Guns (Adapted from Broers, 1975)**

| Cathode | Vacuum requirement, torr | Brightness, $A/cm^2$ steradian | Minimum beam diameter, Å | Lifetime, hr |
|---|---|---|---|---|
| Tungsten hairpin | $>10^{-4}$ | $5\times10^4$ to $10^5$ | $\cong$50 | 40-60 |
| Lanthanum hexaboride | $>10^{-5}$ | $6\times10^6$ | $\cong$25 | $\cong$3000 |
| Field emission | $>10^{-9}$ | $10^7$ to $2\times10^8$ | $\leq$10 | Indefinite |

are widely spaced, the beam current is reduced and poor imaging results. Therefore, manufacturer specifications for the size of this gap must be followed for optimal operation.

Electrons passing through the aperture of the shield are attracted toward the *anode*, the third component of the electron gun. The anode is at a large potential difference relative to the filament, causing acceleration of the electrons. The difference in potential between the filament and the anode is the *accelerating voltage*. A range of voltages between 1 and 30 keV is available on most SEMs. As will be discussed later, the choice of accelerating voltage depends upon specimen type (conductive vs nonconductive samples) and the type of information desired in an analysis.

As mentioned above, the electron gun acts like an electrostatic lens to produce the imaging beam. The effective operating conditions for the electron gun are based upon incandescent heating of the tungsten filament. In this context, filament temperature and current are essentially identical, in that increasing temperature increases current. At very low filament currents, selective crystalline areas of the filament emit electrons, thereby forming a multiple electron source; this condition is referred to as *undersaturation*. As higher currents pass through the filament, the emitting areas correspondingly increase until the tip of the filament symmetrically generates electrons; this condition, which is referred to as *saturation*, is the effective electron source for imaging. Additional current input has no effect on beam intensity but drastically reduces filament lifetime (Fig. 1-4). Proper use of the SEM entails operating at saturation. Attempting operation at undersaturation reduces image clarity because electron-optical alignment cannot be achieved (having too many electron sources results in weak, multiple beam formation), whereas oversaturation drastically reduces filament lifetime. Under acceptable operating conditions, a tungsten filament will emit for ~40 hr; an abused filament will fail after only a few hours of use.

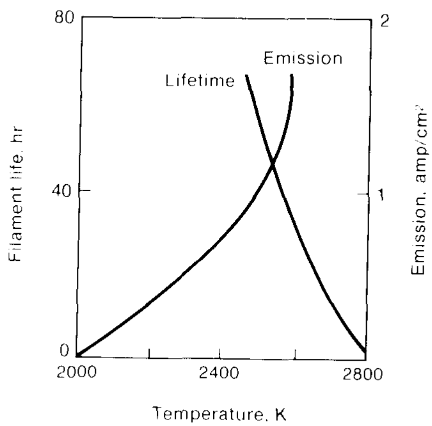


**Fig. 1-4.** Effect of temperature on a tungsten filament.