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Scanning Electron Microscopy: Theory, History and Development of the Field Emission Scanning Electron Microscope

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1.1 THE SCANNING ELECTRON MICROSCOPE

Since its initial development (Everhart and Thornley, 1958) the scanning electron microscope (SEM) has earned a reputation for being the most widely used, high performance, imaging technology that is available for applications ranging from imaging, fabrication, patterning, and chemical analysis, and for materials of all types and applications. It is estimated that 150 000 or so such instruments are now currently in use worldwide, varying in performance and complexity from simple desk-top systems to state-of-the-art field emission gun systems that can now cost in excess of $5 million.

The basic principle of the scanning electron microscope is simple. An incident electron beam is brought to a focus that typically varies in size from a fraction of a centimeter in diameter down to a spot that can be smaller by a factor of many thousands of times, and with an energy varying from 100 eV or less to a maximum of 30 keV or more. This beam spot is typically then scanned (Figure 1.1) in a linear "raster" pattern across the region of interest, although other patterns – such as a radial beam – are sometimes employed for special purposes. Typically the final deposited pattern will contain of the order of 1000 × 1000 or more individual imaging points.

The incident beam electrons can interact with the sample atoms through either elastic or inelastic scattering. Elastic scattering is where the incident electrons are deflected with no loss of energy. Inelastic scattering involves a loss of energy, often by ionizing the sample atoms. The incident electrons will scatter (both elastically and inelastically) many times in

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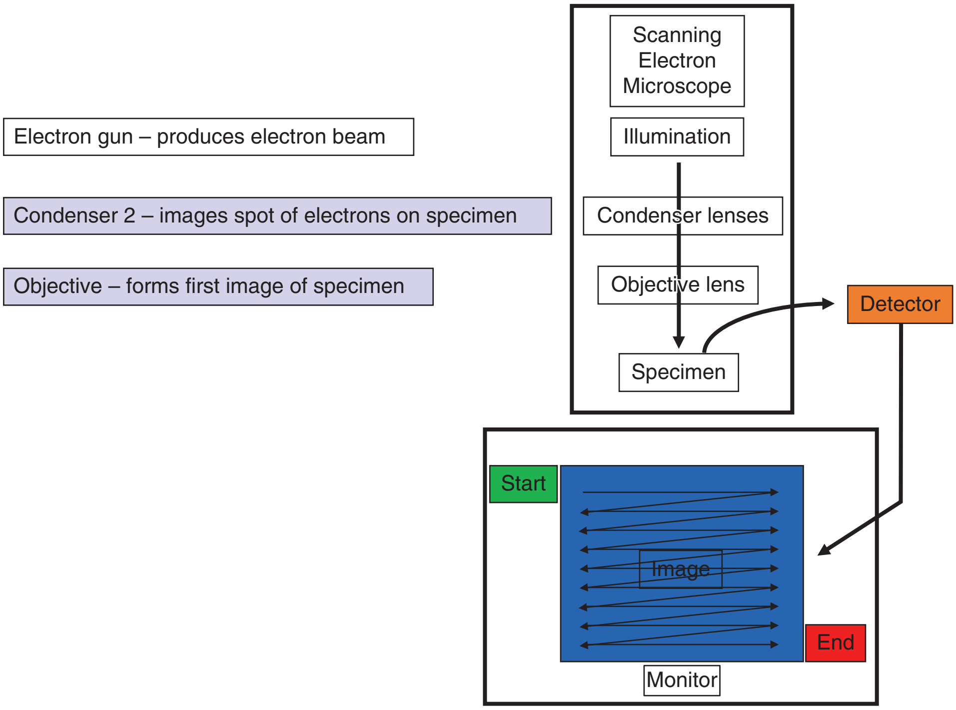

Figure 1.1 The SEM scan raster.

a region of the sample known as the interaction volume. The size of the interaction volume will depend on the incident energy and the nature of the sample, but can be of the order of a micrometer in diameter. A number of different types of signal generated by the beam–sample interaction can be detected. The intensity of the signal detected can be plotted as a function of probe position to form an image. Two important signals are secondary electrons (SEs) and back-scattered electrons (BSEs). Secondary electrons are electrons from the sample atoms that are released through ionization. They are relatively low in energy <~25 eV and tend to only escape from the top few tens of nanometers of the surface. They provide strong topographical imaging of surfaces. Back-scattered electrons are incident electrons that have been multiply scattered and emerge again from the surface. The strength of the scattering that can return the electrons to the surface depends strongly on atomic number, Z, and so BSE imaging gives compositional contrast. Another common signal detected is X-rays from the decay of the ionized atoms. The energy of the X-ray photon emitted is characteristic of the element ionized, and so energy-dispersive X-ray (EDX) spectroscopy allows mapping of element species.

Most modern SEMs will likely have, and make use of, several types of detector so as to optimally detect, capture, collect, and display other analytical and imaging modes as desired.

In operation the electron source must be carefully set up and optimized so as to generate the smallest spot size for the electrons while still ensuring that the beam current reaching the specimen is adequately stable for periods of many hours without the need for any further operator interactions. The overall measure of imaging performance for the electron source is determined by its brightness β, which is defined as

$$\beta = 4I/(d^2.\pi^2.\alpha^2)\ \text{amps/cm}^2/\text{steradian}$$

where d is the diameter of the spot size of the beam at the target, I is the incident beam current, and α is the solid angle subtended by the illumination at the specimen.

For an electron beam source of some specified energy the beam brightness is said to be "conserved", which means that varying the beam current – as, for example, by varying the beam spot size or the convergence angle of the beam – will always result in compensating changes in the other parameters in the system so that the magnitude of β in the equation remains constant. As a result, the intensity of the incident beam current I varies as $d^2\alpha^2$ and if either the beam spot size d or the beam convergence angle α are reduced, then the beam current will decrease, which may ultimately result in the beam becoming lost in the background noise of the instrument. The imaging performance of an SEM is very important and therefore is always very dependent on optimum alignment.

1.2 THE THERMIONIC GUN

For the first 25 years or so of the SEM era the only available sources of the energetic electrons required for microscopy were the so-called thermionic ("hot beam") emitters mentioned above. Even today so-called "table-top" SEM instruments remain in widespread use because of their low cost, good resolution, and operating convenience.

In operation the required electron beam current is generated by heating a tungsten wire filament. This so-called "thermionic emitter" is usually fabricated from high quality tungsten wire that has been bent into a "V" shape and is maintained at a temperature of about 2700 K by means of a separate power supply that heats the tip region. The "V" shape noted above is maintained at some negative voltage typically from about ~1 keV – 30 keV with reference to ground potential. The corresponding incident beam currents typically can vary from 10^{-6} down to 10^{-12} amps or so.

To optimize the yield of the emitted beam current that is generated a "grid cap", or a "Wehnelt" cylinder – with a circular aperture centered on the tip of the emitted beam current– is employed. The cap is maintained in position by a potential source that is set to about 50 volts or greater, so that the emitted beam from the source can be brought to a focused crossover at a point chosen some distance beyond the column grid cap. The generated electron beam can then be accelerated down the column and on to the specimen. For a given beam energy the intensity of the imaging incident beam will be restricted by, and will be highly dependent upon, the emission performance of the gun, so advanced electron microscopes in particular always require carefully optimized beam sources and hardware.

In typical current SEMs equipped with such a thermionic gun the smallest usable beam spot size will be of the order of a few nanometers, and can provide a beam current of between 10 and 1000 picoamps. To achieve an acceptable signal-to-noise ratio in the chosen area of the image range typically requires exposure times of between 30 and 100 seconds depending on the performance of the gun. Higher performance gun sources, discussed later, can reduce the exposure time required by several orders of magnitude, but the ultimate resolution of an SEM with such a thermal emitter is limited both by the need to maintain an adequate incident beam current and by the inherent energy spread of the emitting source. Despite these limitations, thermionic emitters are still in widespread use as they are well suited for imaging at magnifications below 50 k×, although they can only offer relatively poor imaging resolution because the low brightness of the source sets a minimum limit to the useful spot size and the high temperature of the emitting tip tends to broaden the energy spread of the electron beam.

Some further enhancement in imaging performance can by achieved by employing "pointed filaments". As their name implies, in these devices the emitter tip region of the "V" shaped filament is sharpened so as to further increase the field present at the top of the tip. This then results both in an improvement of the electron yield and in a reduction in the apparent source size of the emitter. However, the improvement in performance so achieved is not much better than modest, and the lifetime of the emitter is reduced by the modifications that must be made to the tip. Other sources of even higher performance are therefore still required.

1.3 THE LANTHANUM HEXABORIDE ("LaB_6") SOURCE

This was first described by Lafferty (Lafferty et al. 1951) and later on was further developed and optimized by Broers (Broers et al. 1960) at IBM in the late 1960s. A LaB_6 source can provide significantly better performance than the conventional tungsten emitter described earlier because the LaB_6 has a much lower work function (temperature). The resultant performance enhancement is of importance because for the typical "hairpin" beam sources discussed above each 10% reduction in the work function of the source will increase the emission current density J by a factor of about 1.5 times. As a result of this a LaB_6 emitter operating at 1500 K can generate a significantly higher brightness image than that generated by a conventional tungsten thermionic source operating at 2700 K. In addition, the sharply pointed tip geometry of the LaB_6 emitter results in an effective source size, which is lower than that of a conventional tungsten thermionic emitter and so further enhances the image resolution.

The reduction in the operating temperature that can be employed also serves to enhance the lifetime of the LaB_6 source itself. However, it must be noted that LaB_6 is itself extremely reactive and rapidly forms compounds with all materials other than carbon and rhenium. As a result, once an LaB_6 emitter achieves, and is able to maintain, good stability and brightness it must never be allowed to completely cool down again, and nor should it be exposed to the atmosphere. In summary, LaB_6 emitters are competitive, high brightness, sources with a good signal production, but they must be kept running and under vacuum continuously to maintain and provide the desired stability and performance.

1.4 OTHER ENHANCED "HIGHER BRIGHTNESS" SOURCES

The thermionic electron sources discussed above are simple to build, adequately reliable in use, low in cost, and can offer more than adequate performance for low and medium resolution imaging. However, they cannot provide the significantly higher beam currents, nor the smaller beam source sizes, that are required when imaging specimens in the nanometer scale range. An additional problem is that the thermionic emitter itself must operate at a high temperature, and this in turn generates chromatic aberrations that give rise to a loss of imaging resolution, particularly at low beam energies. Because such uses are increasingly important, there is an increasing need for electron sources that can offer not only higher brightness performance but also smaller source sizes and a reduction in the energy width of the beam.

The development of efficient, high brightness, electron sources began with Thompson's discovery of the electron (Thompson et al, 1895), which demonstrated that charged particles could be emitted from the interior of a conducting material provided that sufficiently large electric fields were applied. Some twenty years later the phenomena of field emission was re-visited by Fowler and Nordheim in 1928 (Fowler and Nordheim, 1928) who proposed the existence of the process now known as "quantum tunneling". While strictly speaking their work only applies to field emission generation from bulk crystalline solids, it does provide a convenient way of understanding what processes are involved.

In 1937 Erwin Muller (Muller, 1937) developed the first practical applications of this technology with his "field ion microscope". This device consisted of a sealed chamber containing a low pressure of hydrogen gas. At one end of the chamber was a stiff tungsten wire, terminating in a sharp point and maintained at a low temperature by the cooled hydrogen gas. At the other end of the chamber was a fluorescent screen. When a sufficiently large potential was provided to the tip, this resulted in the generation of very high electric fields ($\sim 10^7$ volts/cm) in the region around the tip, which was held at a negative potential with respect to ground and so attracted positive ions towards itself. The negative ions that were being generated at the same time were accelerated away from the tip region by the drift field and allowed to travel towards a viewing screen. The drift region itself was field free and so the ions traveled in diverging straight lines to produce bright spots on the screen, each of which could be traced back to the particular atom on the tip from which it had originally come. Subsequent work after the Second World War with this simple device ultimately led to the production of the first ever images of individual atoms (Muller and Bahadur, 1950).

All of the requirements noted above can now be satisfied by employing a field emission gun ("FEG") source. The first attempts to use such an emission source for imaging in a scanning microscope were in fact made by Cosslet and Haines (Cosslet and Haines, 1954) but their efforts were not really successful because they were unable to achieve a sufficiently high vacuum to guarantee stable emissions. Fortunately the rapid improvements in ultra-high vacuum technology that occurred in the 1950s helped to eliminate these problems and led to the publication of a book by Gomer (1961), which discussed the practical advantages of a field emission source for electron microscopy. It was recognized that the most important step in achieving the goal of imaging at near-atomic levels required developing an electron source that offered both the highest possible brightness and the highest resolution at typical SEM energies, that is, 10 to 30 keV. From Gomers' work it was already evident that an optimized field emission source would be the best choice provided that a variety of practical problems could be solved.

The important breakthrough was made in 1968 by Professor Albert Crew and his group at the University of Chicago (Crew, Isaacson, and Johnson, 1968). Their instrument used a field emission cathode in the form of a tungsten rod with a very sharp tip (<100 nm diameter) at one end. When the cathode is held at a negative potential relative to the anode then the electric field at the tip is so strong (typically of the order of 10^{10} volts/cm) that the potential barrier effectively becomes very narrow as well as being reduced in height. As a result electrons can then tunnel directly through the energy barrier and leave the cathode without requiring any additional thermal energy. The anodes act as a pair of electrostatic lenses and form a real image of the emitter tip at a distance of a few centimeters below the second anode. This arrangement, although simple, can produce a beam probe just a few nm in diameter or smaller, and with a beam current of 10^{-12} A or higher.

1.5 THE TWENTY-FIRST CENTURY SEM

The current versions of "FEG SEM" instruments are usually housed in an ultra-high vacuum environment and can operate at energies from 1 keV or less and up to 30 keV or more. Immersion optics that are able to identify materials and to optimize the resolution and high resolution performance, can be obtained for both iSE and BSE modes of operation when the operating conditions are properly optimized. For example, the Crewe type "electron beam" source and its descendants can now routinely provide nanometer scale imaging resolution when used under optimized conditions. Although achieving still higher imaging resolution is possible, it becomes increasingly more challenging because no electron–optical system is ever going to be perfect. In reality, every sample that is made inherently contains a finite number of errors and deviations from perfection, and these serve to limit the effective resolution level that can be achieved. For example, electron beams can be focused into a sub-angstrom diameter spot, but the resultant depth of field of the image so formed may then become too restricted to yield anything of practical value.

1.6 THE FUTURE FOR ION BEAM IMAGING – ABOVE AND BEYOND

The twenty-first century solution to improving the resolution and imaging capability of the SEM is to make the change from using an electron beam and to use ion beams instead. This is advantageous because the wavelength of a hydrogen beam at a given energy is a factor of about 750× shorter than that of an electron beam of the same energy and so the physical size of the beam will no longer be the limiting factor for imaging resolution. It will ultimately then be possible for the operator to image and process a much wider range of materials and in real time. Current ion beam instruments usually make use of beam materials such as hydrogen, helium, neon, or gallium beams, but other chemistries for special applications are also possible and could be the basis of some very interesting studies.

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