Electron Sources

5

CHAPTER PREVIEW

All microscopes need a source of electrons to illuminate the specimen. Fortunately, electron sources are plentiful, but to get the best images and other signals out of our expensive microscope, we need to use the best available source. There are stringent requirements for the beam of electrons and these are best met by only two types of source: thermionic and field-emission sources. Thermionic sources are either tungsten filaments or lanthanum hexaboride (LaB_6) crystals, and field emitters are fine tungsten needles. In this chapter we'll first explain briefly the physics of these two emission processes because then you'll understand why we operate the sources in certain ways. Next we'll tell you the characteristics we need from our electron beam. Then we'll compare the three sources and show you that no one source is best for all aspects of TEM, but all three have their roles. Finally, we'll explain ways to check that a particular source meets your specification.

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Because the source is so critical to the performance of the microscope, the technology is advancing rapidly to the point of complete computer control, which would leave you, the operator, with precious little to do except push the "on" button. This state of affairs is most advanced for the field-emission source, and since these are both delicate and expensive, it is just as well. But the vast majority of TEMs still use thermionic sources, and these need a fair bit of operator control. In these circumstances, you should know how these sources work and why you do certain things to them. So we'll spend most of this chapter talking about thermionic sources, although there's a good chance that field emission will be the source of choice in the future.

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5.1. THE PHYSICS OF DIFFERENT ELECTRON SOURCES

We use two kinds of electron sources in TEMs: the first kind is called a thermionic source, which, as the name suggests, produces electrons when heated, and the second type is a field-emission source, which produces electrons when an intense electric field is applied to it. These sources are part of an assembly which we refer to as the "electron gun." Now, from a physics standpoint, it is really quite interesting to know the details of how electron sources work and there's a great deal of active research into new and improved sources. However, from a practical standpoint, you don't have to know too much of the physics, and we can summarize the essential points very briefly, using a few simple equations. Keep in mind two points as you read about sources:

> Your TEM will use a thermionic source or a field-emission source and the two cannot be interchanged.
> Field-emission sources give "monochromatic" electrons; thermionic sources are less monochromatic and give "whiter" electrons.

The analogy here is to X-rays or visible light. You don't always want to use "monochromatic" electrons, even if the field-emission TEM did cost twice as much as a "conventional" microscope would with a thermionic source.

5.1.A. Thermionic Emission

If we heat any material to a high enough temperature, we can give the electrons sufficient energy to overcome the natural barrier that prevents them from leaking out. This barrier is termed the "work function" (Φ) and has a value of a few electron volts.

The physics of thermionic emission can be summarized in Richardson's Law, which relates the current density from the source, J, to the operating temperature, T in Kelvin

$$J = AT^2 e^{-\frac{\Phi}{kT}} \qquad [5.1]$$

where k is Boltzmann's constant (8.6×10^{-5} eV/K) and A is Richardson's "constant" ($A/m^2 K^2$), which depends on the source material. From this equation then you can see that we need to heat the source to a temperature T such that energy greater than Φ is given to the electrons; then they will escape from the source and be available to form an electron beam. Unfortunately, when we put a few eV of thermal energy into most materials they either melt or vaporize. So the only viable thermionic sources are either refractory (high melting point) materials or those with an exceptionally low work function. In practice we use both types: tungsten has the necessary high melting temperature (3660 K) and lanthanum hexaboride (LaB_6) has a low work function. If you look ahead to Table 5.1, you'll see the relative values of J_c, T, and Φ for tungsten and LaB_6.

We use several different words to describe the sources. We sometimes call tungsten sources "filaments," because tungsten can be drawn into fine "thread" which is about 0.1 mm in diameter and is similar to the filament used in an incandescent light bulb. The wire is bent into a V shape so they're also called "hairpin" filaments, or they may be sharpened to a fine point. For decades these have been the standard source in most electron-beam instruments. LaB_6, or other rare-earth boride crystals (which should not be called filaments) are usually grown with a <110> orientation to enhance emission. Sometimes we call both tungsten and LaB_6 sources "cathodes" because, as we'll see, the complete gun assembly acts as a triode system in which the source is the cathode.

Thermionic sources:	W hairpin Pointed W LaB_6 and other low-Φ materials, e.g., CeB_6

So all you need to know from the physics is that heating up a thermionic source gives you a higher *J*. But there is a limit because higher temperatures shorten the source life through evaporation and/or oxidation. So we seek a compromise operating temperature, and we achieve this by operating under a condition called "saturation," which we'll discuss in Section 5.3.A.

5.1.B. Field Emission

Field-emission sources operate on a fundamentally different principle than thermionic sources. The principle behind field emission is that the strength of an electric field *E* is considerably increased at sharp points, because if we have a voltage *V* applied to a (spherical) point of radius *r* then

$$E = \frac{V}{r} \qquad [5.2]$$

The technique of field-ion microscopy is another well established experimental tool. It requires specimens with a very fine needle shape, and so there's a lot of expertise available to help produce field-emission electron sources. One of the easiest materials to produce with a fine tip is tungsten wire, which can readily be given a tip radius of <0.1 μm. If we apply a 1-kV potential to this tip, then *E* is 10^{10} V/m and this lowers the work function barrier sufficiently for electrons to tunnel out of the tungsten. This process imposes quite severe stress on the tip and the material has to be strong. Field emission, like thermionic emission from LaB_6, depends on the crystallography of the tungsten tip; the <310> orientation is found to be best.

To allow field emission, the surface has to be pristine, that is, free of contaminants and oxide. We can achieve this by operating in UHV conditions (<10^{-11} Torr), and in this case the tungsten is operated at ambient temperatures and the process is called "cold field emission." Alternatively, we can keep the surface in a pristine condition at a poorer vacuum by heating the tip. The thermal energy assists in electron emission so much that, in fact, the electrons don't tunnel through the barrier. For such "thermal field emission," surface treatments with ZrO_2 improve the emission characteristics, particularly the stability of the source, and such "Schottky" emitters are becoming popular. New sources such as semiconductor p-n field emitters are also provoking some interest.

5.2. THE CHARACTERISTICS OF THE ELECTRON BEAM

The electron beam in a TEM requires certain characteristics which are controlled by the source itself and how we integrate the source into a gun assembly. We describe the performance of an electron source by such terms as "brightness," "coherency," and "stability." While these words mean something to you already, they have very precise meanings in TEM terminology, so we'll go through the various characteristics, tell you what they mean, and why they are important in the TEM. We'll then compare the properties of the various sources that you may have in your microscope and you'll see that there's no "best" source for all applications, but for specific applications one source or another is clearly the best.

Before we define the electron beam characteristics needed in a TEM, it is worth summarizing here a few of the properties of electron beams in general and how these vary with accelerating voltage.

5.2.A. Brightness

The word "brightness" is often confused with "intensity" and indeed the two terms are related. For instance, when we look at the viewing screen of a TEM, we may say how "bright" it is, when we are really referring to the intensity of light coming from the screen. When we think of the intensity of any radiation source, it is in terms of the flux emanating from it. For a light bulb, it would be the number of photons per unit area per unit time. For electron sources we talk about the current density, which is the number of electrons (or charge) per unit area per unit time.

While current density can be a useful term, it is more important to define the *brightness*. Brightness is the *current density per unit solid angle* of the source.

Electron sources differ considerably in their size and, as a result, the electrons leave the source with a range of angles, so we can't ignore the angular distribution of the electrons. Brightness is particularly important when we are using very fine electron beams, as we do in analytical and scanning microscopy. The concept of brightness is less important in conventional TEM, where we use a relatively large, defocused beam, but it is still relevant to the intensity we see on the screen, and so it affects how easy it is to operate the microscope and see our images and diffraction patterns.

So we can consider an electron source as having the following characteristics:

- a diameter d_0,
- giving off a certain cathode emission current i_e,
- the electrons diverging from the source with a semiangle α_0.

The actual way in which this is achieved we'll talk about in Section 5.3, where we discuss the complete gun assembly, but if you look ahead at Figure 5.1 you'll see that i_e, d_0, and α_0 are actually defined at the gun crossover, that is, the point at which the electrons are focused after leaving the source. The current density (current per unit area) is $i_e / \pi(d_0/2)^2$ and the solid angle of the source is $\pi\alpha_0^2$, so we define the brightness, β, as

$$\beta = \frac{i_e}{\pi\left(\frac{d_0}{2}\right)^2 \pi(\alpha_0)^2} = \frac{4\, i_e}{(\pi\, d_0\, \alpha_0)^2} \qquad [5.3]$$

This equation is an important one which you should remember. The units of β are usually A/(cm^2 sr) or A cm^{-2} sr^{-1}. Again we see that microscopists are not comfortable using SI units, which would be A m^{-2} sr^{-1}, increasing the traditional brightness number by a factor of 10^4. What is not shown in this equation is the important fact embodied in equation 5.1 that β increases linearly with increasing accelerating voltage for thermionic sources. This is one reason for the development of intermediate voltage (300–400 kV) instruments.

Obviously, the higher the value of β, the more electrons we can put into an electron beam of a given size, and so the more information we can extract from the specimen and the more we can damage sensitive specimens. The beam current is an important part of the brightness equation. Having some way of measuring the beam current *in situ* can be a very good diagnostic tool. We'll talk about this later in the chapter when we discuss measuring the source brightness, but for the time being you can again look ahead to Table 5.1 to see how the three sources compare in brightness, which we have given in non-traditional SI units.

Now we can consider some real numbers. With a cold FEG at 100 keV, we can put 1 nA into an area of diameter 1 nm at a maximum. If you convert this current density to units of power (1 watt = 1 J/s), you'll find that the energy the electron beam puts into this small area of the specimen is nearly 150 MW/mm^2. The output of a typical electric power generating turbine is only about 600 MW. Clearly, we can change our specimen when we look at it in the TEM as we discussed in relation to beam damage. The energy density we just calculated means that an electron source is the brightest continuous radiation source known; it is considerably brighter than a supernova.

The brightness is particularly important in AEM, which is the technique of quantitative analysis of the many signals that come from a specimen irradiated by an electron beam, shown back in Figure 1.3. Similarly, as we go to higher magnifications in HRTEM, the screen intensity becomes less because we are viewing only a fraction of the illuminated area of the specimen. The electron density can be increased by using the brightest available source. Then images can be recorded with reasonably short exposure times.

5.2.B. Temporal Coherency and Energy Spread

The coherency of a beam of electrons is a way of defining how well the electron waves are "in step" with one another. You know that white light is incoherent, because it consists of photons with a range of wavelengths (colors), and so to get a coherent beam of electrons we must create one in which all the electrons have the same wavelength, just like monochromatic light. We refer to this aspect of coherency as "temporal coherency," which is a measure of how similar the "wave packets" are. If they are all identical they have the same coherence length. A definition of the coherence length λ_c is

$$\lambda_c = \frac{v\, h}{\Delta E} \qquad [5.4]$$

where v is the electron velocity, ΔE is the energy spread of the beam, and h is Planck's constant. This means we must have stable power supplies to the source and a stable high-voltage supply (or high tension, as it's sometimes called for historical reasons) so that all the electrons have a small ΔE, thus giving a well-defined wavelength. Now in practice it's impossible to create a truly monochromatic beam and we have to live with a certain range of electron energies/wavelengths, although the stability of electronic components has improved substantially over the years. Again if you look at Table 5.1 you'll see typical ΔE values for the three sources and they're in the range 0.1 to 3 eV (which is remarkably small compared with a total energy of 100 to 400 keV). So it isn't really correct to imply that thermionic sources give "white" electrons since ΔE is still small. From these values of ΔE, if you take care to get the units consistent, you can calculate typical coherence lengths, which turn out to be a few hundred nanometers.

Temporal coherency is important when the energy spread of the electrons that are *incident* on the specimen affects the microscopy. Because we can make such good high-tension power supplies, this rarely limits any aspect of TEM except perhaps high energy-resolution electron spectrometry (see Chapters 37–40). In other words, for most practical purposes our electron sources are stable enough. However, we'll see that it's a very different matter when we have to consider the electrons that have come *through* the specimen because they may have lost substantial amounts of energy.

5.2.C. Spatial Coherency and Source Size

Spatial coherency is related to the size of the source. Perfect spatial coherence would imply that the electrons were all emanating from the same point at the source. So source size governs spatial coherence and smaller source sizes give better coherency (just as they give higher brightness). The spatial coherence is strictly defined by looking at electron interference fringes in the equivalent of a Fresnel biprism experiment in light optics, with which you may be familiar. We can define the distance d_c, the effective source size, for coherent illumination to be

$$d_c << \frac{\lambda}{2\alpha} \qquad [5.5]$$

where λ is the electron wavelength and α is the angle subtended by the source at the specimen. We can control α by inserting an aperture in the illumination system, as we'll see when we describe the construction of a TEM in Chapter 9. But if this aperture is not limiting then it is the smallest source which subtends the smallest angle, and thus has the highest spatial coherence. Putting reasonable values for 100-keV electrons into equation 5.5 we find that the spatial coherence is at best only about a nanometer. To maximize the coherency, you can choose several approaches:

- Make the source size d_c smaller, e.g., by using a field-emission source.
- Use a smaller illumination aperture, thus reducing α.
- If your source size is large (e.g., a W hairpin) decrease the accelerating voltage and thus increase λ.

Spatial coherency is more important practically than temporal coherency. A small electron source subtends a small angle at the specimen, and we can help by using small limiting apertures. Small beams are more spatially coherent than large beams. The more coherent and parallel the beam is, the better the quality of the phase-contrast images (see Part III), the sharper the diffraction patterns (see Part II), and the better the diffraction contrast in images of crystalline specimens (see Part III). An in-depth and rather mathematical description of coherency in the TEM is given in the review by Hawkes (1978).

5.2.D. Stability

In addition to the stability of the high-voltage supply to the source, it is also important that the electron current coming from the source is stable. Otherwise, the screen intensity will vary, making it difficult for you to take correctly exposed images, and also making microanalysis impossible in many cases. Thermionic sources are generally very stable except when they are first installed, or when they are about to fail. Typically, you can expect variations of less than 1% per hour in the current. For cold field emission sources, however, the emission current is not very stable, and electrical feedback circuits are required to maintain stability to better than 5%. Stability does improve with better UHV conditions.

To summarize, the important properties of electron sources are their brightness, temporal coherency, energy spread, spatial coherency, and stability. A smaller source size gives higher β and better spatial coherency, but less stability.

Now that we know the critical characteristics required of electron sources, let's examine those used in commercial TEMs.

5.3. ELECTRON GUNS

It's no good just having a source. We need to be able to control the electron beam and direct it into the illumination system of the TEM. We do this by incorporating the source into a gun assembly which in effect acts as a lens to focus the electrons coming off the source. The design of the gun is different for thermionic sources and field-emission sources.

5.3.A. Thermionic Guns

Both tungsten and LaB_6 sources are used as the cathode in a triode gun shown in Figure 5.1. In addition to the cathode, there is a "grid" called a Wehnelt cylinder, and an anode at earth potential with a hole in its center. What these three components look like in practice is shown in Figure 5.2, where they are all separated. The cathode is attached to the high-tension cable, which in turn connects to the high-

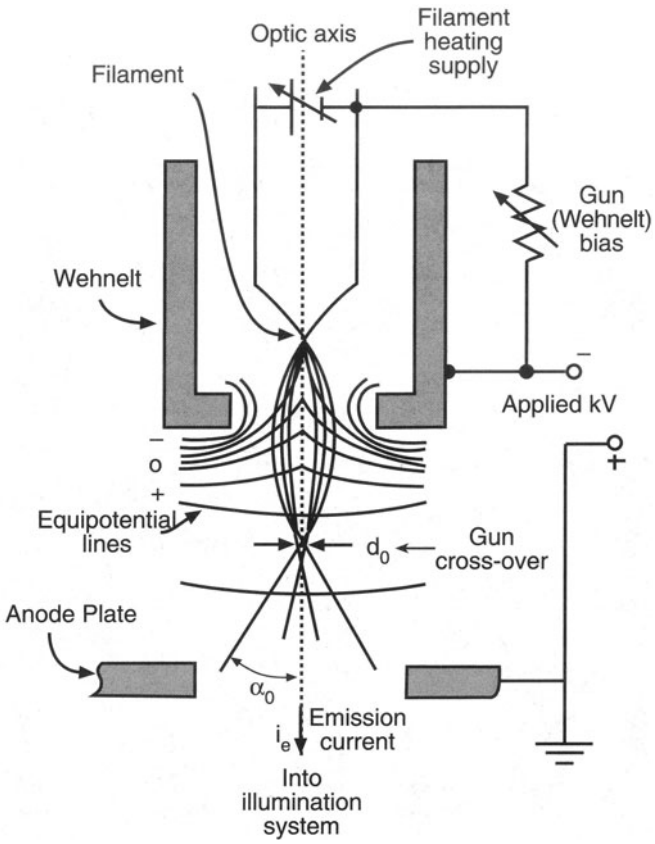


Figure 5.1. Schematic diagram of a thermionic electron gun. A high voltage is placed between the filament and the anode, modified by a potential on the Wehnelt which acts to focus the electrons into a crossover, with diameter d_0 and convergence/divergence angle α_0.

voltage power supply. This cable also connects to the tungsten filament to supply a current to heat the filament resistively to the operating temperature. LaB_6 sources are indirectly heated usually by bonding them to a metal filament such as rhenium, which is resistively heated.

As the filament current (i_f) increases the temperature increases until thermionic emission occurs, and an emission current from the cathode i_e can be measured. Sometimes you'll find this current referred to as the "beam current," but this is misleading, because the true beam current is that which enters the specimen after the electrons have left the gun and gone through the illumination system of the microscope.

When the electrons leave the cathode they have a negative potential of 100 kV with respect to the earthed anode, so they accelerate through this potential difference acquiring an energy of 100 keV, and a velocity of greater than half the speed of light.

Now to get a controllable beam of electrons through the hole in the anode and into the microscope itself, we apply a small negative bias to the Wehnelt cylinder. The electrons coming off the cathode see the negative field and are converged to a point called a crossover between the Wehnelt and the anode as shown in Figure 5.1. We could operate the cathode heating and the Wehnelt bias controls independently, but the electronic circuitry of the gun is designed so that as the emission current increases the Wehnelt bias increases, and this arrangement is called a "self-biasing" gun. The result is shown in Figure 5.3, which plots the filament emission current (i_e) against the current used to heat the filament (i_f). As you can see, i_e reaches a maximum such that further increase in i_f doesn't increase the current going into the microscope. This is the *saturation condition* and all thermionic sources should be operated at or just below saturation. Operating above saturation reduces filament life without any compensating ad-

Figure 5.2. The three major parts of a thermionic gun, from top to bottom: the cathode, the Wehnelt cylinder, and the anode, shown separated. The Wehnelt screws onto the cathode (filament) support and both are attached to the high-tension cable which contains power supplies for heating the filament and biasing the Wehnelt. The anode sits just below the Wehnelt, in the top of the TEM column.

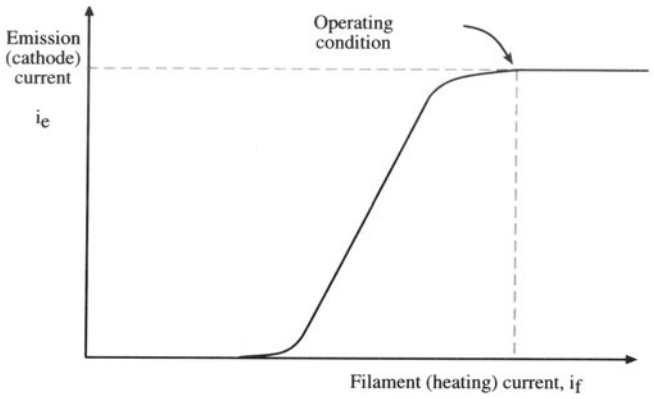


Figure 5.3. The relationship between the current emitted by the electron source (i_e) and the filament heating current (i_f) for a self-biasing gun. Increasing the filament current results in a maximum emission current termed saturation.

vantage; operating significantly below saturation reduces the current into your specimen, thus reducing the intensity of all the signals coming out of your specimen.

The Wehnelt acts as a simple electrostatic lens: the first lens in the microscope.

In addition to optimizing the source life, operating at saturation also optimizes brightness. If you look at Figure 5.1, the crossover is the source size d_0 that we used

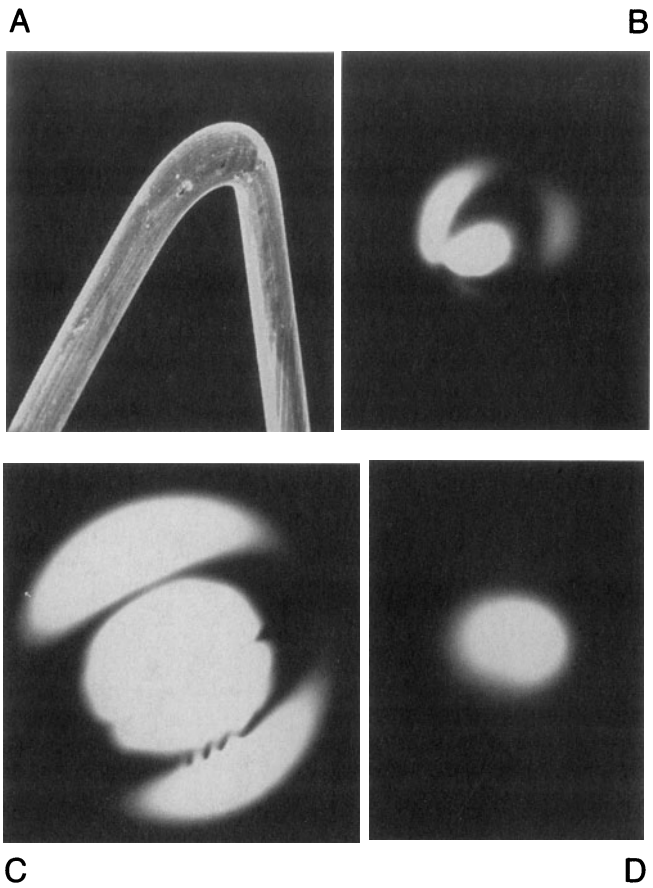


Figure 5.5. (A) The tip of a tungsten hairpin filament and the distribution of electrons when the filament is (B) undersaturated and misaligned, (C) undersaturated and aligned, and (D) saturated.

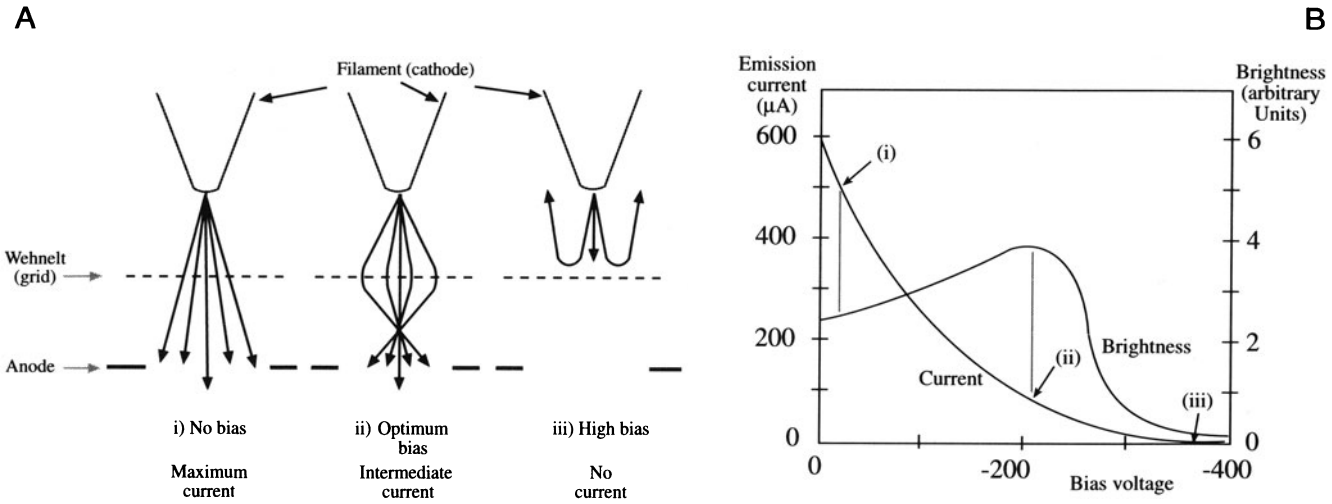


Figure 5.4. (A) The effect of increasing Wehnelt bias (i–iii) on the distribution of electrons coming through the anode. (B) The relationship between the bias and the emission current/gun brightness. Maximum brightness is achieved at an intermediate Wehnelt bias, and an intermediate emission current [condition (ii) in A].

back in the brightness equation (equation 5.3) and the convergence/divergence angle at the crossover is α_0 in that same equation. The current in the crossover is the emission current i_e. Now, as shown in Figure 5.4A, if the Wehnelt bias were too low (i) d_0 would not be very small, and if the bias were too high (iii) the cathode emission current would be suppressed. In either case β would be low. The optimum β is at an intermediate bias setting (ii), as summarized in Figure 5.4B. You might think that the small bias on the Wehnelt acts against the accelerating voltage, so the true beam voltage is the applied kV minus the Wehnelt bias (which may be up to 2 kV), but this is compensated for in the design of the gun.

So how do we achieve saturation? One way is to look at the meter which displays the i_e and watch it rise to a maximum as i_f is continuously increased. This method may not be easy because the appropriate readouts may not be available, or if they are, they may not be very sensitive. So the standard way is to look at the image of the filament crossover on the TEM screen; this image shows you the distribution of electrons coming off the filament. As thermionic emission starts the electrons come from both the central tip of the filament and a region surrounding the tip (Figure 5.5A), and so the filament image is as shown in Figure 5.5B or C, and is characteristic of an unsaturated tungsten filament. With increasing emission the halo of emission collapses in on the central bright disk, although some structure may still be visible. The filament is truly saturated when no structure is visible (Figure 5.5D).

Since LaB_6 sources have well-defined crystal facets (Figure 5.6A) they show a slightly different undersaturated image, as you'll see in Figure 5.6B, but in essence the process is identical. It is probably best to operate an LaB_6 source at conditions just below saturation, since this will extend the source life without undue loss of signal. We'll find that there are a few occasions when undersaturated operation can be useful, because the electrons in the halo are more coherent than those in the central bright region. LaB_6 crystals are more susceptible to thermal shock than tungsten, and so you should take care when heating *and cooling* an LaB_6 source. Increasing the heating current should be done slowly, with 10 to 20 seconds' pause between each setting. This is particularly critical after you've installed a new LaB_6 source.

The appearance of the image of the source, such as we show in Figures 5.5 and 5.6, can also be used to align the gun assembly so that the beam is aligned along the optic axis of the microscope. This is the only other thing you have to do to the gun apart from saturating it. The source is usually pre-aligned by the manufacturer, so alignment should be simple when it is put inside the Wehnelt. Typically, the undersaturated source image is asymmetrical as in Figure 5.5B

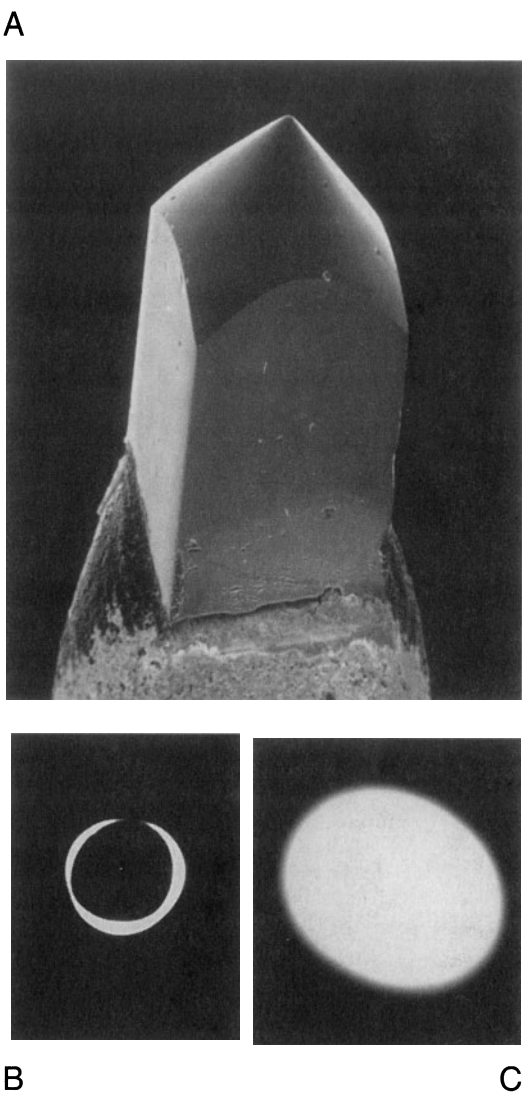


Figure 5.6. (A) An LaB_6 crystal and the electron distribution when the source is (B) undersaturated and aligned and (C) saturated.

and in those circumstances all you have to do is tilt the gun assembly to make it symmetrical as in Figure 5.5C. Detailed instructions will be in the manufacturer's handbook.

> Achieving optimum β is critical in any operations that require a fine beam (<0.1 μm).

In an SEM, which always requires a small probe, the gun is carefully adjusted by the manufacturer to produce optimum β at saturation, and you may not have any external control of the Wehnelt. In a TEM, particularly when you are operating in a broad-beam mode, there is no need to optimize β, but you may need to increase the current density and make the image appear brighter. You can

do this by decreasing the Wehnelt bias, using the "emission" control. When you decrease the bias, you should go back and adjust i_f to ensure you're at saturation, since the saturation condition will change with changing bias. So now you will have a greater current density falling on the screen, but the crossover size will have increased, thus decreasing β. This is not important if you're operating with a broad beam, but if you want to operate at maximum β with a focused beam, as is the case for AEM, then you need to be able to measure β; we'll show you how to do that in Section 5.5.

5.3.B. Field-Emission Guns (FEGs)

In many ways, FEGs are much simpler than thermionic guns. In order to get an FEG to work we make it the cathode with respect to *two* anodes. The first anode is positively charged by several kV with respect to the tip. This is called the "extraction voltage" since it generates the intense electric field-extracting electrons by enabling them to tunnel out of the tip. Increasing the extraction voltage when you first switch on has to be done slowly, so the mechanical shock doesn't fracture the tip. This is the only practical step you have to carry out to run an FEG, and it can easily be computer-controlled.

- Anode 1 provides the extraction voltage to pull electrons out of the tip.
- Anode 2 accelerates the electrons to 100 kV or more.

The electrons are accelerated through the applied potential by the second anode. The combined fields of the anodes act like a more refined electrostatic lens to produce a crossover, as shown in Figure 5.7A. This lens controls the effective source size and position, but it isn't very flexible. Incorporating a magnetic lens into the gun gives a more controllable beam and larger β. The faults (known as lens aberrations) in the gun lens are very important in determining the source size; we'll talk extensively about lens aberrations in Chapter 6.

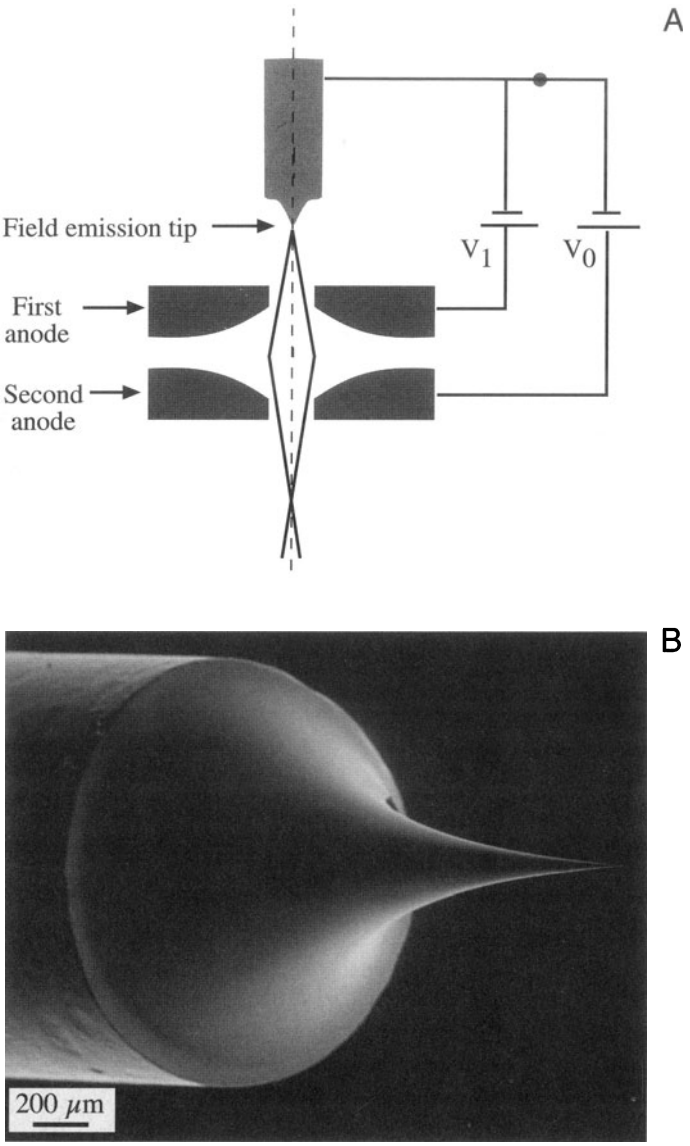


Figure 5.7. (A) Electron paths from a field-emission source showing how a fine crossover is formed by two anodes acting as an electrostatic lens. Sometimes an extra (gun) lens is added below the second anode. (B) An FEG tip, showing the extraordinarily fine W needle.

In a vacuum of 10^{-7} Torr, one monolayer of contaminants will form on a substrate in less than a minute. At 10^{-10} Torr, it will take 7 hours to form a monolayer.

We have already noted that field emission requires a pristine surface and, even in UHV conditions, surface contaminants build up on the tip. With time, the emission current falls and the extraction voltage has to be increased to compensate. But eventually it becomes necessary to remove the contamination by "flashing" the tip. This just means reversing the potential to the tip and "blowing off" a surface layer of atoms, and/or heating the tip quickly to ~5000 K to evaporate the contaminants. In most FEGs flashing occurs automatically, when the extraction voltage increases to a certain predetermined level. Thermally assisted FEGs do not form the same surface contamination

layer and so don't need flashing. A typical FEG tip is shown in Figure 5.7B.

5.4. COMPARISON OF GUNS

All the important characteristics of the three guns we've talked about are summarized in Table 5.1. Tungsten sources are the worst in most respects, but for routine TEM applications they are excellent, reliable sources and are cheap, robust, and easily replaceable.

LaB_6 is a more useful source for several reasons. While it is not as refractory as tungsten, LaB_6 has a much lower value of Φ, and since Φ appears in the exponential in the Richardson equation, its effect on the current density is dominant. LaB_6 crystals can be produced with a fine tip about 1 μm in radius, which accounts for the smaller crossover size. As a result LaB_6 current densities are considerably higher than for tungsten. The brightness is typically 10 times that of tungsten, even though LaB_6 is usually operated at a much lower T to increase operating life. The decreased source size also results in improved coherency and the energy spread can be as little as 1 eV.

The drawback to LaB_6 is purely economic. LaB_6 sources cost several hundred dollars each while tungsten filaments are so cheap that the manufacturer often provides them free. Because LaB_6 is a highly reactive material, the gun vacuum has to be 10–100 times better than for tungsten, and is correspondingly more expensive to construct. So if the cost is not the criterion, LaB_6 guns are *the* recommended thermionic source, for all aspects of TEM, but particularly AEM. The increased brightness, higher coherency, and longer life are tremendous advantages. But you as the operator have the most control over its performance and you can most easily destroy it by careless heating and cooling and oversaturation. So treat LaB_6 sources gently and you will be well rewarded. If users are not careful, your TEM supervisor may try to extend the life of the LaB_6 to the point where it behaves no better than a W filament. LaB_6 sources don't die, they fade away.

In FEGs, the current density is enormous and β is correspondingly high. The values in Table 5.1 are all for 100-kV accelerating voltage and you should remember that for the tungsten and LaB_6 sources, β increases linearly with kV, so there are advantages to using 300 and 400 kV instruments, although the thermionic source brightness at 400 kV still does not approach β of an FEG at 100 kV. The extremely small source size means that the beam is highly spatially coherent and the resulting energy spread is minuscule for cold FEGs; thermally assisted FEGs give a larger energy spread. So for all applications that require a bright, coherent source, the FEG is best. This is the case for AEM, HRTEM, and such special applications as electron holography and Lorentz microscopy (for looking at magnetic domains). However, as we'll see later, the coherence of the source may produce a new complication: we must interpret the image!

For routine TEM, an FEG is far from ideal because the source size is so small. It is thus not possible to illuminate large areas of the specimen without losing current density, and therefore intensity, on the screen. Under these circumstances, a thermionic source is better. This limitation to FEG applications may be overcome by the larger *p-n* FE sources, which use small (≈ 1–10 μm) Si semiconductor crystals, but this is still a new and developing technology.

Another drawback to FEGs is the need for UHV conditions. UHV technology is expensive and requires a much higher level of operator competence. As a result, FEG TEMs are relatively rare. But in the SEM field there is a whole new generation of computer-controlled low-voltage instruments, and it will only be a matter of time before FEG TEMs are common.

TABLE 5.1. Characteristics of the Three Principal Sources Operating at 100 kV

	Units	Tungsten	LaB_6	Field Emission
Work function, Φ	eV	4.5	2.4	4.5
Richardson's constant	A/m^2K^2	6×10^5	4×10^5	
Operating temperature	K	2700	1700	300
Current density	A/m^2	5×10^4	10^6	10^{10}
Crossover size	μm	50	10	<0.01
Brightness	A/m^2sr	10^9	5×10^{10}	10^{13}
Energy spread	eV	3	1.5	0.3
Emission current stability	%/hr	<1	<1	5
Vacuum	Pa	10^{-2}	10^{-4}	10^{-8}
Lifetime	hr	100	500	>1000

5.5. MEASURING YOUR GUN CHARACTERISTICS

This section requires that you know how to operate a TEM. If you're a novice, you should skip this part of the chapter for now because we are going to refer ahead in the book for much of what you need to know.

For conventional TEM imaging and diffraction and many other routine uses, all you need to do is saturate and align the gun and then ignore it. There are, however, times when we need to be able to measure the brightness and coherency. The source brightness is a most important parameter to measure in an AEM since, if the gun is not operating at its maximum β, then the quality of the analytical information that is generated will be poor. Similarly, knowing the energy spread of your source is important for electron spectroscopy, and having a measure of the beam coherency can be important for some more advanced techniques that we've just mentioned. So let's see how we can measure the various parameters that we've just discussed. We'll start with β, then ΔE, and finally the coherency.

By measuring the three variables in equation 5.3, i.e., the beam current, the beam diameter, and the semiangle of convergence, we can determine β. However, while we can easily get a measure of the emission current at the gun, it is more difficult to measure d_0 and α_0 there. So we make the approximation that, if we neglect lens aberrations, β is constant throughout the electron optical system so it doesn't matter where it is measured. It is easiest, practically, to determine β at the plane of the specimen and we'll now show you how to do this.

5.5.A. Beam Current

You can measure the beam current at the specimen i_b directly using a Faraday cup in a specimen holder. A Faraday cup consists of a small aperture above a relatively deep hole in an earthed metal block. If the aperture is small enough (e.g., about 50 μm) and the metal block deep enough (about 2 mm), and made of something light like Al to minimize backscatter, then it is a reasonable assumption that no electrons escape back out of the entrance aperture. All the electrons going into the aperture therefore go to earth, and you can measure the electron current using a picoammeter in the earth line. Ideally a Faraday cup should be available permanently in the column of a TEM, and this would permit constant monitoring of the beam current. You can also calibrate the Faraday cup measurement against the TEM screen exposure meter or the electron energy-loss spectrometer shield current. This procedure permits you to make a more rapid estimate of i_b at any time you need it.

As we'll show in Chapter 9, i_b is a strong function of the beam size. Therefore the current is controlled by the first condenser (C1) lens strength, and the size of the final beam-limiting aperture in the second condenser (C2) lens. If you look ahead to Figures 9.10 and 9.11 you will see the variation of i_b as a function of C1 lens strength and the effect of C2 aperture size on α.

- The beam current is usually in the range from nanoamps to picoamps.
- The emission current is typically several microamps.

Most of the current from the gun is lost in the illumination system, as we'll see in Chapter 9.

5.5.B. Convergence Angle

You can easily measure the convergence semiangle α from the convergent-beam diffraction pattern, which you can see directly on the TEM screen. (You will need to read Chapter 21 on convergent-beam diffraction in order to find out how to generate such patterns.) In the schematic diagram in Figure 5.8, the total convergence angle 2α is proportional to the width of the diffraction disks, a. This width can easily be calibrated if the specimen has a known Bragg angle $2\theta_B$ (see Chapter 11), since $2\theta_B$ is proportional to the distance, b, from the 000 disk to the $hk\ell$ disk. Thus

$$2\alpha = 2\theta_B \frac{a}{b} \qquad [5.6]$$

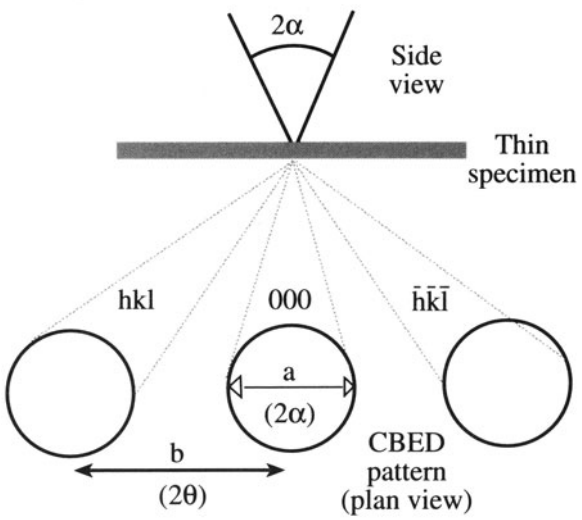


Figure 5.8. The distances on a convergent-beam diffraction pattern from which you can measure the beam-convergence semiangle, α, which is proportional to the width of the diffraction disk.

The convergence semiangle is not only important in the brightness equation, but we'll see that it also plays a major role in convergent-beam patterns, in STEM imaging, and in EELS. Knowledge of α is useful in many aspects of TEM. The value of α is controlled by the size of the final limiting aperture in the illumination system and we'll see how this works in Chapter 6.

5.5.C. Calculating the Beam Diameter

While it is a relatively simple matter to measure i_b and determine α, the measurement of d, the beam diameter, is not so straightforward. However, d is a major factor in all aspects of TEM where we use a fine focused beam, such as AEM and STEM imaging. We can either calculate d or measure it experimentally.

The first problem with determining d is that there is no universally accepted definition of the beam diameter. The manufacturer will give you a list of nominal beam sizes for each setting of the C1 lens. These values are *calculated* and may differ from the actual beam size by large amounts. The calculation assumes that the electron intensity distribution in the beam is Gaussian, and the beam diameter is defined as the full width at half maximum (FWHM) of the Gaussian distribution, defined in Figure 5.9. To approach a Gaussian intensity distribution, the beam must be well aligned, any astigmatism in the condenser lenses corrected (see Chapter 9), and all apertures in the illumination system accurately centered. Even under these conditions you cannot obtain Gaussian conditions for every possible beam size. For example, there may be six different C1 lens excitations, each of which gives a different calculated beam size, but there are invariably fewer than six C2 apertures available, so each beam size cannot be correctly apertured; spherical aberration effects will then broaden the beam size beyond a true Gaussian. If you select too small an aperture, then the intensity distribution will be truncated at a fraction of the full Gaussian curve.

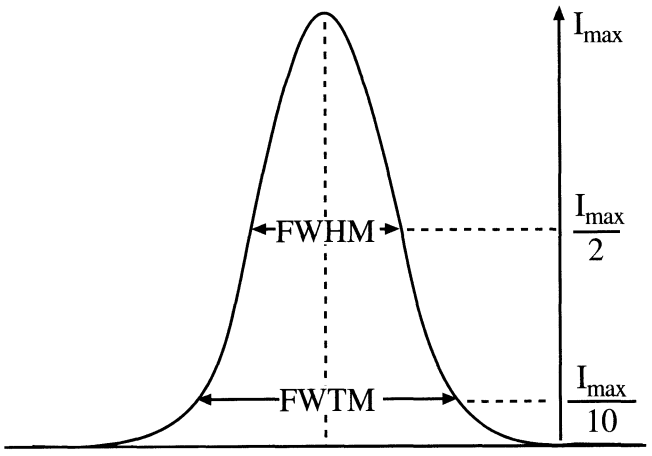


Figure 5.9. The definition of the full width at half maximum (FWHM) and the full width at tenth maximum (FWTM) of a Gaussian intensity distribution which is typical of a well-aligned beam.

To make a complete calculation of the beam size, we assume that it is determined by an initial Gaussian diameter at the gun (d_g). This diameter is broadened by the effects of spherical aberration in the beam-forming lens (d_s) and diffraction at the final aperture (d_d). All these terms should be added in quadrature to give a total, calculated beam size, d_t

$$d_t = \left(d_g^2 + d_s^2 + d_d^2\right)^{1/2} \qquad [5.7]$$

This equation gives us only a first-order estimate, since it is not clear that all the contributions are Gaussian. We'll now briefly discuss the origin of each of these terms.

The value of d_g is a function of β, and a value of β has to be assumed for the purposes of calculation. The expression for d_g is

$$d_g = \frac{2}{\pi}\left(\frac{i}{\beta}\right)^2 \frac{1}{\alpha} \qquad [5.8]$$

We have already defined i, β, and α.

The disk of minimum confusion caused by spherical aberration has a diameter given by

$$d_s = 0.5\, C_s\, \alpha^3 \qquad [5.9]$$

where C_s is the spherical aberration coefficient, which we discuss in detail in Chapter 6. This is the full diameter containing 100% of the beam current. Clearly, this term is not Gaussian unless the beam is correctly apertured which, as we just discussed, is not always possible. The calculated diameter due to diffraction is

$$d_d = 1.22\, \frac{\lambda}{\alpha} \qquad [5.10]$$

which is the Rayleigh criterion which we discussed in Chapter 1. Although all these definitions do not define the same diameter of the electron distribution, they are all combined to give a first approximation of the FWHM of the beam. Clearly, it is more reliable, but more time-consuming, to measure d experimentally. Figure 5.10 shows the result of calculations of the three contributions to the beam diameter in a VG HB501 STEM.

5.5.D. Measuring the Beam Diameter

To measure the beam size in a TEM/STEM, you must form an image of the beam on the TEM viewing screen under conditions where you know, or can calibrate, the magnification. This is a nontrivial exercise and you may need to

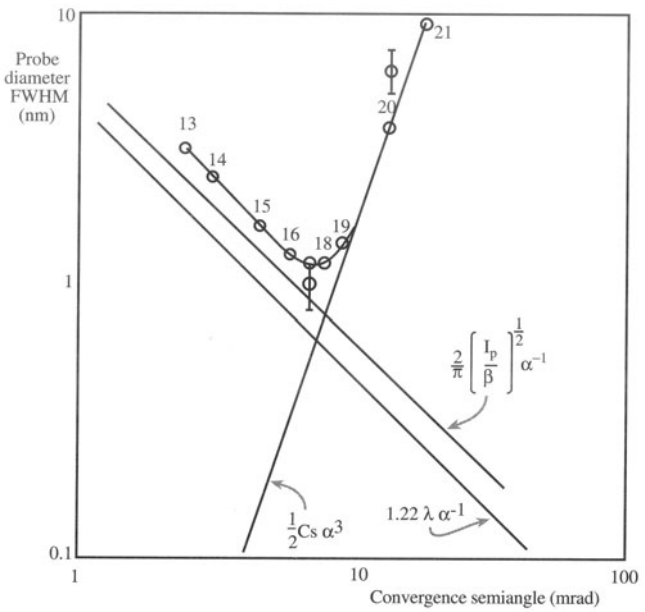
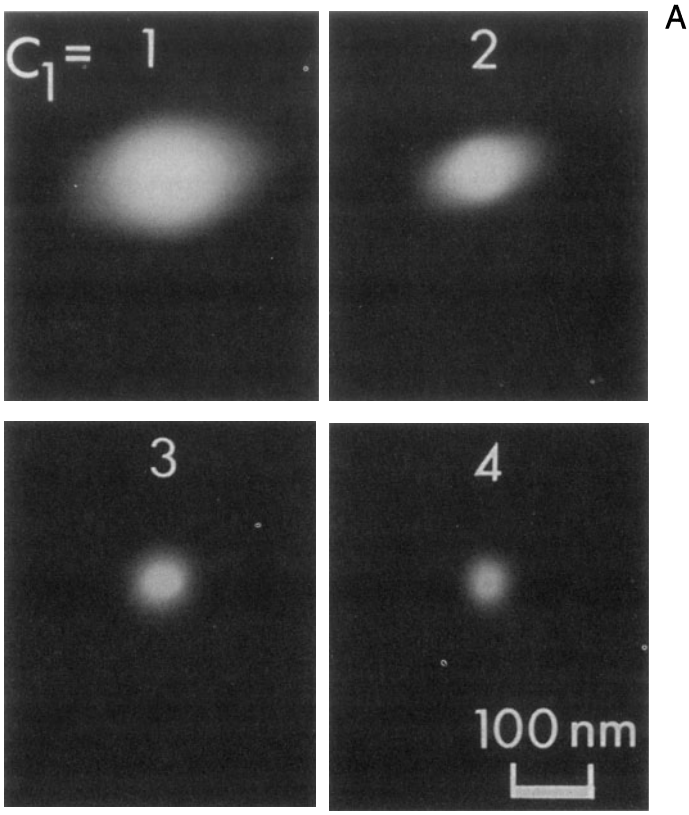


Figure 5.10. Calculations of the three contributions to the probe size as a function of the convergence semiangle α in an FEG STEM with a probe current I_p of 0.85×10^{-8} A. Two experimental measurements are shown, at condenser 1 lens settings 17 and 20. The minimum probe dimension is ~1 nm with $\alpha < 10$ mrads.



consult the manufacturer to be sure that you are doing it correctly. You can then photograph the beam and determine the intensity distribution from a microdensitometer trace across the image, as shown in Figure 5.11.

- The FWHM contains 50% of the integrated intensity. It is the value used by the manufacturers when they report beam sizes. It is also the important dimension when considering the effect of d on the STEM image resolution.
- The full width at tenth maximum (FWTM) contains 90% of the integrated intensity. It is a more relevant dimension because the Faraday cup measures the current in the total beam which is closer in size to the FWTM.

When you insert the beam diameter in the brightness equation, either the FWHM or the FWTM can be used. The FWTM is equal to 1.82 x FWHM and this is also shown in Figure 5.9. You should note, therefore, that you overestimate β if you use the smaller FWHM. Use of the FWTM is also the preferred beam size when calculating the spatial resolution of microanalysis, as we describe in Chapter 36.

In a dedicated STEM you can't image the beam directly, since there are no post-specimen lenses to magnify

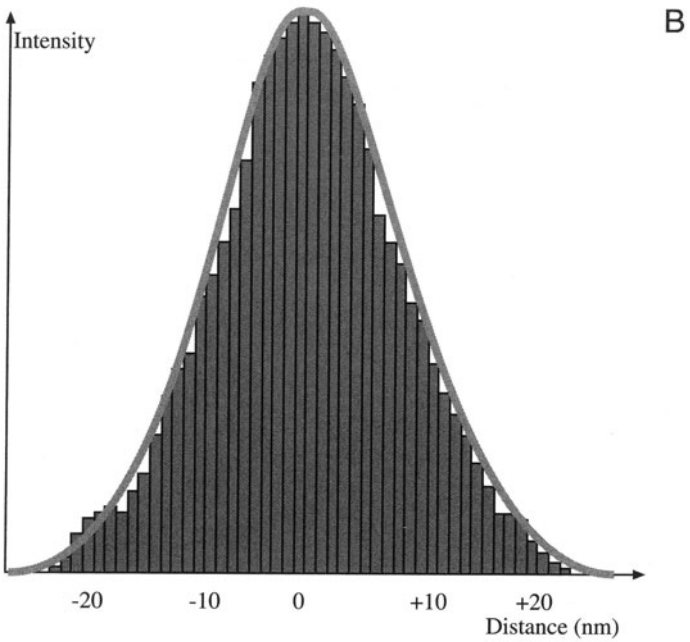


Figure 5.11. (A) Four images of the beam formed on the TEM screen at different condenser 1 lens settings and (B) the corresponding microdensitometer trace across spot #3, confirming the Gaussian nature of the intensity distribution.

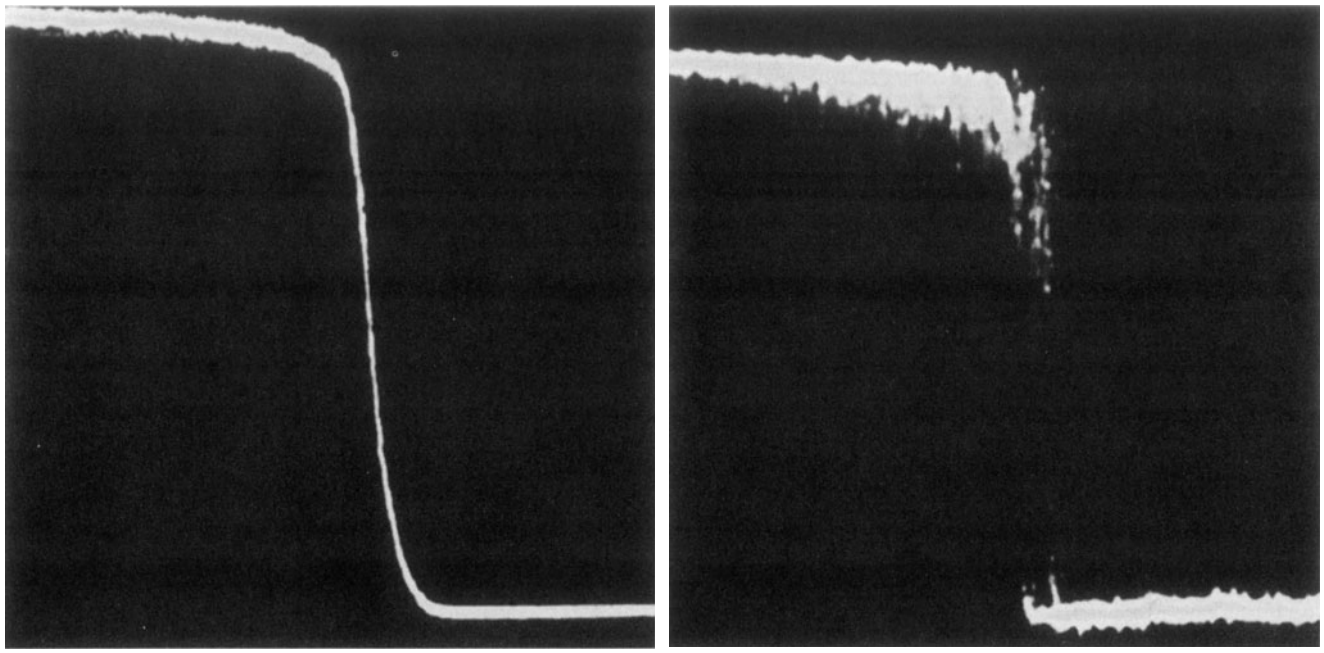


Figure 5.12. Intensity profiles obtained by scanning a fine beam across a sharp edge of a cube of MgO. The measured probe size (FWTM) in (left) is 7.4 nm (magnification 1×10^6) and in (right) 1.8 nm (magnification 11×10^6). The smaller probe contains a much smaller current and is therefore a noisier trace.

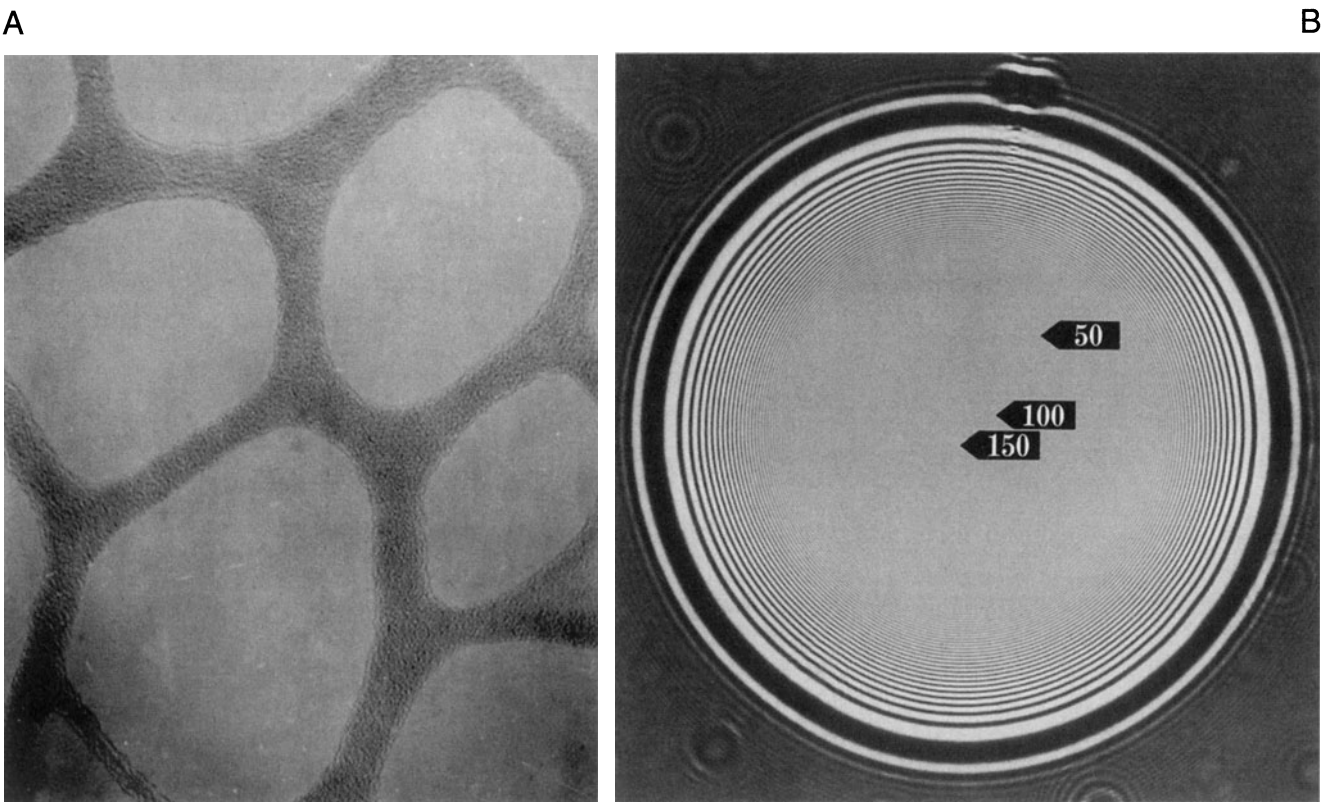


Figure 5.13. Fresnel fringes from (A) a thermionic source with poor coherency and (B) an FEG with high coherency.

its image and no photographic film to record it. The value of d must be determined indirectly, as in other scanning instruments. The best method involves scanning the beam across a knife-edge specimen and monitoring the intensity change which occurs, for example, by recording the output from the annular dark-field detector. This approach yields an integrated intensity profile, as shown in Figure 5.12. In order to extract a value of the FWHM or FWTM from the profile, you must make measurements between various points determined by integrating the intensity from one side of a two-dimensional Gaussian to the other. In Figure 5.10, two experimental beam-size measurements are shown; they show reasonable agreement with the calculated values from the brightness equation.

The measurement of d is clearly not a simple procedure. You can find a full description of the problems in the paper by Michael and Williams (1987).

5.5.E. Energy Spread

Remember that the energy spread (ΔE) of the electron beam is a measure of the temporal coherency. This spread is important in EELS and, in fact, the only way to measure the energy spread is to use an electron spectrometer. Under conditions where the spectrometer itself is not limiting the resolution of the spectrum, the value of ΔE can be simply measured by collecting a spectrum of electrons without a specimen in the way of the beam. The spectrum then consists of a single Gaussian peak and the resolution of the spectrum is defined as the FWHM of this peak. You can find out how to do this in detail in Chapter 37. Typical values of ΔE for the various electron sources are also given in Table 5.1.

5.5.F. Spatial Coherency

It's difficult to measure the coherency of the beam experimentally although, as we've discussed, small sources ensure spatial coherency. One practical way of measuring the coherency is to form an image of the edge of a hole in a specimen, such as a thin holey carbon film. When you operate slightly out of focus you see alternating dark and bright fringes, called Fresnel fringes, as shown in Figure 5.13A. Typically for a thermionic source only one or two fringes are visible. These fringes are a phase-contrast effect (see Part III). We can also use them to correct the astigmatism in the objective lens, as we'll see in Chapter 9. The number of visible fringes is a measure of the beam coherency. Figure 5.13B shows the enormous number generated by an FEG.

5.6. WHAT kV SHOULD YOU USE?

For the materials scientist, this is usually an easy question to answer. You always operate at the maximum available kV, unless there is a definite reason to use a lower kV. Of these reasons, the most obvious is avoiding beam damage, but we'll see others later in the book, so don't forget that you can always operate a 300-kV machine at 100 kV. Remember, it's like being able to change the wavelength of a monochromatic light source in a visible-light microscope (VLM). The threshold for beam damage for most metals is less than 400 kV, which is the highest available voltage on "off-the-shelf" TEMs. For lighter and more beam-sensitive materials, such as some ceramics and polymers, lower voltages may be better, but there is not much use going below 100 kV since the images will be rather dim and you'll have to make extraordinarily thin specimens to see anything useful. The reasons for choosing the highest kV are:

- The gun is brightest.
- The wavelength is shortest; the resolution is potentially better.
- The cross section for inelastic scatter is smaller; the heating effect is smaller.

CHAPTER SUMMARY

Most TEMs use thermionic sources and, if you have the choice, use an LaB_6 source and run at the highest kV. Take care when heating and cooling the LaB_6 crystal and always operate just below saturation to maximize the lifetime of a source. If you're going to be doing AEM, get some idea of the beam current that you can get from your source under typical operating conditions. Also, measure the beam size and convergence angle to give a measure of β, and if you're doing EELS then the energy spread is essential information. If you have an FEG you'll most likely be doing fine probe analytical work, in which case all the above characteristics must be measured, and if you're going to do high-resolution imaging, then the degree of coherency is important too. Always treat the source carefully when changing it, aligning it, saturating it, or switching it off. There's nothing more annoying than losing your source, since it usually happens at some critical point during your work.

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