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Low kV Scanning Electron Microscopy

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1. Introduction

The voltages typically associated with low-voltage scanning electron microscopy are within the range of 5 kV and lower. The low end cutoff is typically the lowest value at which a user’s microscope can achieve a usable image. The author of this chapter would like to see a new definition of low kV-accelerating voltage begin within the realm of 2–1 kV. With the advent of new column designs, through the lens (TTL) secondary electron (SE) detectors and the use of electrostatic lens, and similar techniques, the low end of the low kV range is typically reaching well below 500 V, and is often approaching 100 V. These new and tried true techniques are illustrated in Figs. 4.1 and 4.2. A recent development is the use of a retarding field to modulate the landing energy of the primary electrons. By simply applying a negative potential (Vb) to the specimen, a retarding field can be generated between the specimen and a grounded electrode above the specimen (the cold finger or the objective lens in a semi-in-lens type FEG-scanning electron microscope [SEM] instrument). In this simple configuration, the specimen itself is part of a “cathode lens” system. The landing energy ($E_L$) of the incident electrons can be varied by changing the applied potential:

$$E_L = E_0 - eVb.$$ 

where $E_0$ is the energy of the primary electron beam. The nature of the electron–specimen interactions is determined by the electron landing energy $E_L$. When $eVb$ is equal to or larger than $E_0$, the incident electrons will not enter the specimen at all; in this particular configuration a mirror image may be formed. Therefore, by varying the potential applied to the sample, we can conveniently extract useful surface information of the sample. The ultimate resolution of ultralow voltage (ULV) SEM images is determined by the combined properties of the probe-forming lens and the cathode lens [1]. The highest resolution secondary images are being obtained using TTL secondary detectors commonly referred to as TTL detectors. These TTL detectors often involve a strong magnetic field projected into the chamber, or a mild electrostatic field. This field is generally used
FIGURE 4.1. Secondary electron image of resist on Si at 1 kV uncoated.

FIGURE 4.2. Secondary electron image of resist on Si at 0.1 kV uncoated.
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To collect the low-energy SEs, which help to provide the highest resolution images. The type of field and detector positioning above the final lens will often depend upon the manufacturer of the system. Often this field type and detector positioning will have a great effect on image quality, as well as overall system performance. To further this point Joy wrote *Through-the-Lens (TTL) Detectors*, which use the postfield of the lens to collect the secondary signal, have not only a much higher efficiency (DQE ~0.8), but also are more selective in the energy spectrum of the electrons that they accept. In the most advanced design the TTL detector can, in effect, be tuned so as to collect either a pure SE signal or a backscattered signal or some mixture of the two. This provides great flexibility in imaging, and avoids the necessity of a separate BSE detector, and permits backscattered operation at very short working distances. The ultimate goal remains a detector that is both efficient and can be used to select any arbitrary energy window in the emitted spectrum for imaging [2]. Low kV SEM can also have profound effect on the results achieved during elemental analysis. To quote Boyes form of microscopy and microanalysis: the use of lower voltages (Boyes, 1994, 1998) to generate x-ray data for elemental chemical microanalysis improves substantially the relative sensitivity for surface layers, small particles, and light elements. The reduced cross sections for x-ray yield can be partially compensated with improved analysis geometries. There remains a need to achieve a critical excitation energy plus a factor of 20–40% (or overvoltage $U$, of $1.2 < U < 1.4$), and this limits analysis of complete unknowns to about 6 kV; 5 kV is inadequate for the elements I, Cs, and Ba. The main light elements (B–F) and those 3d series transition metals with strong L lines are still accessible at 1.5 kV and this can be especially important for the analysis of the smallest particles and thinnest overlayers [3].

This chapter will try to address the concepts and practices of low kV SEM, and its use to attain high-resolution micrographs. (What does the term “high resolution” refer to? In the case of current production SEMs, high resolution would be better than 2 nm.)

2. Electron Generation and Accelerating Voltage

The first thing that should be addressed is what exactly is “low kV?” To truly understand the term “low kV” the reader must understand how kV is controlled or set. This will require an understanding of an “electron gun.” The basic principles and design of an electron gun and an electron optical column do not vary much from SEM to SEM. The basic design consists of a cathode and anode arrangement. Within this basic design there is an electron source. This source will normally consist of either tungsten or lanthanum hexaboride (LaB6). The electrons are generated by one of a couple of methods. They are either generated thermally or through the use of field emission. In the case of systems where high-resolution imaging is going to be done in the lower kV ranges, the user will more than likely be using a field emission source rather than a thermal source.
In the case of the thermal emitter, the electron source is heated by running a current resistively through a wire. As the current is increased the heating temperature increases. By increasing the temperature of the source, the electrons are liberated due to lowering of the work function of the electron donor material to a point where the electrons are in essence boiled off from the wire. The wire is subjected to temperatures at or close to 2,700 K. This cloud of electrons is gathered up in the Wehnelt cylinder. This Wehnelt cylinder contains the cathode (filament) of the electron gun. The Wehnelt is kept slightly negative in relation to the filament, which provides a focusing effect for the cloud of electrons. The cathode is set to ground potential, 0 V. The voltage of the anode is set to the voltage desired to be the accelerating voltage (e.g., 20 or 1 kV). The anode plate contains an aperture through which the electrons will pass. This difference in voltage provides the potential for acceleration of the electrons down the column [4]. This scenario is called thermal emission (Fig. 4.3). Normally this type of electron emission provides a stable source of electrons, but is frequently associated with lower resolution SEMs (an ultimate resolution of ~3 nm at 20–30 kV).

SEM's that are used at lower accelerating voltages and produce high-resolution images at these “low kV” settings produce their electron beam through the use of one of the two categories of field emission. The two varieties of field emission are thermal and cold. Both have advantages and disadvantages. Generally both are very capable for “low kV,” high-resolution imaging. The basic concept behind field emission is an electron source that has been attached to a tungsten wire. This electron source itself is a piece of tungsten. It has a tip radius smaller than 100 nm. As a negative potential is applied to the source, the small tip concentrates this potential. This concentration of potential at the tip subjects it to a high field. This high field allows the electrons to escape from the source material. The two types of field emission guns, cold and thermal (illustrated below), function in more or.

![Figure 4.3. Thermal emitter.](image-url)
less the same way in respect to the field generated at the tip. If the tip is held at a negative 3–5 kV relative to the first anode, the effective electric field $F$ at the tip is so strong ($>10^7$ V/cm) that the potential barrier for electrons becomes narrow in width as well as reduced in height by the Schottky effect. This narrow barrier allows electrons to “tunnel” directly through the barrier and leave the cathode without requiring any thermal energy to lift them over the work function barrier. Tungsten is the cathode material of choice since only very strong materials can withstand the high mechanical stress placed on the tip in such a high electrical field. A cathode current density as high as $10^5$ A/cm² may be obtained from a field emitter compared with about 3 A/cm² from a tungsten hairpin filament. In a field emitter, electrons emanate from a very small virtual source (~10 nm) behind the sharp tip into a large semiangle (nearly 20° or about 0.3 rad), which still gives a high current per solid angle and thus a high brightness [5]. A second anode is used to accelerate the electrons to the operating voltage. The only real differences fall into the categories of vacuum requirements are long-term source stability and overall I-probe (probe current) that can be generated by a source. Other considerations in regard to column design will have an effect over low kV performance. Figure 4.4 illustrates Schottky field emission source. Table 4.1 compares the different types of electron sources.

3. “Why Use Low kV”?

There are many interesting reasons why a microscopist or researcher might use low kV. First and foremost would be charge reduction. SEM users have often fought the charging influences of an electron beam. Often users coat their samples with a conductive coating if the samples happen to be of a nonconductive
variety. The advantages and disadvantages of coating have long been argued and this author does not wish to wade into this discussion. Many samples that are non-conductive often have a point where they reach equilibrium. This equilibrium is the point where the charge into the sample equals the charge out of the sample. The charge out of the sample will be from SEs, backscattered electrons (BSE), Auger electrons, x-rays, and whatever current is absorbed and then transmitted through the sample to ground. This is referred to as a state of unity. Charging comes in several species, and often exhibits several characteristics. These species are either positive or negative charging. When the sample charges positively the image will appear bright, and when there is a negative charge the image will appear dark. Charging can also be frequently seen as streaks of light or dark on an image. As the charge of a sample changes a user will often also see the image drift across the screen. Users frequently attribute this to stage or sample instability. These two causes can be ruled out as a cause by raising or lowering the kV. If the drift either stops or changes direction, charge balance is the culprit, not stage instability or poor sample mounting. The effect that is being seen as image drift is in fact the buildup of charge in the sample pushing the electron beam away from the area to be imaged; hence the appearance of drift. If the charge on the sample becomes great enough, the result can be a reflection of the primary e-beam by the charged sample causing a scan of the chamber to result. This will happen when the charge of the sample is greater than the charge of the primary beam (e.g., a primary beam of 2.5 kV and a charge on the sample of 20 kV). The SE image that is produced as a result of the excessive charge on the sample (a plastic sphere) can be seen in Fig. 4.5.

<table>
<thead>
<tr>
<th>Emitter type</th>
<th>Thermionic</th>
<th>Thermionic</th>
<th>Cold field emission</th>
<th>Schottky field emission</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cathode material</td>
<td>W</td>
<td>LaB₆</td>
<td>W (310)</td>
<td>ZrO/W (100)</td>
</tr>
<tr>
<td>Operating temperature (K)</td>
<td>2,800</td>
<td>1,900</td>
<td>300</td>
<td>1,800</td>
</tr>
<tr>
<td>Cathode radius (nm)</td>
<td>60,000</td>
<td>10,000</td>
<td>≤100</td>
<td>≤1,000</td>
</tr>
<tr>
<td>Effective source radius (nm)</td>
<td>15,000</td>
<td>5,000</td>
<td>2.5 (a)</td>
<td>15 (a)</td>
</tr>
<tr>
<td>Emission current density (A/cm²)</td>
<td>3</td>
<td>30</td>
<td>17,000</td>
<td>5,300</td>
</tr>
<tr>
<td>Total emission current (µA)</td>
<td>200</td>
<td>80</td>
<td>5</td>
<td>200</td>
</tr>
<tr>
<td>Normalized brightness (A/cm²sr*kV)</td>
<td>1 × 10⁴</td>
<td>1 × 10⁵</td>
<td>2 × 10⁷</td>
<td>1 × 10⁷</td>
</tr>
<tr>
<td>Maximum probe current</td>
<td>1,000</td>
<td>1,000</td>
<td>0.2</td>
<td>10</td>
</tr>
<tr>
<td>Energy spread at the cathode (eV)</td>
<td>0.59</td>
<td>0.4</td>
<td>0.26</td>
<td>0.31</td>
</tr>
<tr>
<td>Energy spread at the gun exit (eV)</td>
<td>1.5–2.5</td>
<td>1.3–2.5</td>
<td>0.3–0.7</td>
<td>0.35–0.7</td>
</tr>
<tr>
<td>Beam noise (%)</td>
<td>1</td>
<td>1</td>
<td>10</td>
<td>1</td>
</tr>
<tr>
<td>Emission current drift (%/h)</td>
<td>0.1</td>
<td>0.2</td>
<td>5</td>
<td>&lt;0.2</td>
</tr>
<tr>
<td>Minimum operating vacuum (hPa)</td>
<td>≤1 × 10⁵</td>
<td>≤1 × 10⁶</td>
<td>≤1 × 10⁸</td>
<td>≤1 × 10⁸</td>
</tr>
<tr>
<td>Cathode life (h)</td>
<td>200</td>
<td>1,000</td>
<td>&gt;2,000</td>
<td>&gt;2,000</td>
</tr>
<tr>
<td>Cathode regeneration</td>
<td>Not required</td>
<td>Not required</td>
<td>Every 6 to 8</td>
<td>Not required</td>
</tr>
<tr>
<td>Sensitivity to external influences</td>
<td>Minimal</td>
<td>Minimal</td>
<td>High</td>
<td>Low</td>
</tr>
</tbody>
</table>

Source: Adapted from Carl Zeiss SMT Literature.
The use of low kV leads to reduced beam interaction volume. It has been noted by Joy and Newbury [6] that as the energy \((E_0)\) of the incident beam is reduced the range \((R)\) of the electrons falls significantly \((R \approx k.E_0^{1.66})\). This reduced interaction volume results in the production of an SE signal from closer to the surface of the sample. Signal development from closer to the surface leads to more surface information. Low kV operation also benefits image quality of low atomic number (low Z) samples. At low kV the penetration depth of the electrons into low Z materials is much less than higher kV electrons on the same samples. Frequently low Z samples will have a ghost-like or semitransparent look at higher accelerating voltages. By lowering the kV of the beam, the resulting image will be less transparent, and display more information about the material. Often this reduction of interaction volume has the result of improving the resolution of x-ray microanalysis. The user must make sure that there is sufficient overvoltage to excite the peaks required for analysis of the samples of interest. It is advisable to do the analysis at higher kV settings to identify the materials present. If the spatial resolution is not satisfactory, a lowering of the accelerating voltage is recommended provided that peaks for elements required for either the mapping or line scans are available. Boyes mentions that the use of low kV and high kV for

**Figure 4.5.** A secondary electron image formed by charging a plastic sphere at 20 kV, and then imaging the sample at 2.0 kV. The resulting image is formed by the low kV primary beam being reflected about the chamber by the sample that has been charged to a higher kV.
microanalysis is convergent analysis and will lead to a better understanding of the 3D structure and chemistry. Figures 4.6, 4.7, 4.8, and 4.9 are SiO$_2$ nanoparticles and Figs. 4.10, 4.11, 4.12, and 4.13 are a Ti fracture sample. These images illustrate how lowering the kV can reveal more surface information. This reduction of interaction volume also has some other effects on the signal being produced. One of these effects is the increase of SE emission. This increase in the emission of SEs is the direct result of the shrinking of the interaction volume. As the interaction volume is brought closer to the surface, the SEs have a better opportunity to escape from the sample since they are being produced in a region where they are more likely to escape than to be reabsorbed by the sample. It is important to remember that SEs are “defined purely on the basis of their kinetic energy; that is, all electrons emitted from the specimen with an energy of less than 50 eV... are considered as SE.” SEs are produced as a result of the interaction of the primary electron beam and electrons within the elements of the sample. This interaction is generally a collision between a high-energy electron of the beam, and an electron within the specimen. There is a transfer of energy from the beam electrons to the specimen electrons. This energy transfer results in the final energy of the SE that escapes from the sample. Most SEs have an energy, which is less than 10 eV. SEs are of two types: SE1 and SE2 electrons. The SE1 electrons are generated closer to the surface by the interaction of the primary e-beam and specimen electrons. The SE2 electrons are generated by the interaction of BSEs and

![Image of SiO$_2$ nanoparticle at 20 kV on an Al stub](image)

**Figure 4.6.** SiO$_2$ nanoparticle at 20 kV or an Al stub.
FIGURE 4.7. SiO₂ nanoparticle at 10 kV or an Al stub.

FIGURE 4.8. SiO₂ nanoparticle at 5 kV or an Al stub.
FIGURE 4.9. SiO$_2$ nanoparticle at 1 kV or an Al stub.

FIGURE 4.10. Ti fracture sample at 15 kV.
Figure 4.11. Ti fracture sample at 2 kV.

Figure 4.12. Ti fracture sample at 0.25 kV.
specimen electrons. The SE2 electrons are of higher kinetic energy, which allows them to escape from deeper within the sample due where they are being generated. These SE2 electrons result in a somewhat lower resolution image when compared with an image with a higher number of SE1 electrons [4]. Another interesting point made by Müllerová and Frank must also be considered: The active depths of SE and BSE signals become similar and hence both signals are sensitive to the surface cleanliness. Also the widths of the response functions and hence the image resolutions approach each other. The SE signal grows relatively to the BSE one and exceeds this, and at some critical energy no charge is dissipated in the specimen. When the penetration depth of primary electrons approaches, somewhere between 150 and 700 eV—the escape depth of SE—the edge effect disappears together with signal enhancement on inclined facets. Consequently, micrographs appear more “flat,” with limited perception of the third dimension. The topographic contrast is more surface-sensitive and from among relief details those just filled by the upper half of the interaction volume are the brightest. Surface films of so defined (or smaller) thickness become opaque, including contamination islands. [7]

4. Using Low kV

The actual practice of using lowered accelerating voltages is not much unlike using high kV. The user of the microscope will have several things they will have
to consider. These considerations include no specific order: sample preparation and microscope preparation.

Sample preparation will include the choice of whether or not to coat the sample to make it more conductive, if it is not conductive. A user will also need to consider what type of analysis or imaging they are trying to achieve from the system. Often users will decide on a type of mounting that will accommodate the type of sample they are evaluating. It should be thought that the sample needs to be further processed down the line after evaluation, or is this destructive testing. Are ultra high-resolution images the goal, or are low to moderate resolution images acceptable for this project. Frequently, all of the questions will influence a user’s choice of mounting and preparing a sample.

To mount a sample several techniques can be offered. For high-resolution images a user needs to select the most mechanically stable mounting media. The best suggestion is to avoid any type of tape or “sticky tabs.” These will often present an image that will display a drift at high magnifications as the tabs or tape out gas in the vacuum. Often the material from which they are made rebounds after a sample has been pressed into the tab or tape. They are not mechanically stable. They do provide a quick mounting solution, and are often excellent for quick and dirty microscopy when an answer is required immediately. An excellent choice for mounting samples well is a carbon paint/dag or silver paint/paste. These types of mounting media do take some time to dry and are not perfect for all samples, but when it comes to a rigid, stable mount they do provide a good solution as long as speed is not of the essence. There are also many different mechanical sample holders such as vices, and spring-loaded holders that are excellent for bulk type samples. When it comes to mounting samples such as particles, carbon nanotubes, and liquid suspensions which can be dried down, the author has found that carbon-coated formvar TEM grids provide an excellent solution. They provide a nice even black background without the topography often encounter with Al or C graphite sample stubs, by mounting the sample on a grid and by placing it into a sample holder that suspends the sample. Artifacts from an SE or BSE signal being returned from the mounting stub will not influence the image being produced of the sample. The image in Fig. 4.14 is on a carbon-coated formvar TEM grid, the image in Fig. 4.15 is on a piece of Si wafer, and the image in Fig. 4.16 is on an Al stub.

The question of coating is one that inspires a great deal of discussion. The first topic is whether or not to coat the sample. If the decision is made to coat the sample, the next topic of discussion will often be what should be used. Often users are restricted to the type of specimen coater they currently own. In most situations this coater will not always be the latest state of the art coater with all of the bells and whistles. This fact frequently influences a user, to make the decision not to coat their sample because of the perceived poor quality of their coater. There is a rational fear of overcoating the sample, and thus obscuring the beautiful details thereafter at the “low kV” settings. The argument can be made that in many cases a user can flash down 1–5 s of a coating with their old out dated coater and still get excellent high resolution results without the artifacts commonly associated
Figure 4.14. SiO$_2$ nanoparticles on a carbon-coated formvar TEM grid mounted on an STEM sample holder imaged at 1.2 kV.

Figure 4.15. SiO$_2$ nanoparticles on a piece of Si, imaged at 1.2 kV.
with overcoating a sample. This thin or light coating is used to stabilize the sample enough in order to take the sample from impossible to possible. I always suggest a user attempt to image a sample uncoated first, and when all else fails then coating is an apt solution. The user will still have to employ many of the strategies for “low kV” and “low probe current” imaging which have yet to be outlined. Most important will be the proper adjustment of their electron optical column. This of course varies from column to column. In the simplest set of adjustments a user will need to adjust, focus, stigmation and aperture alignment. On more complex columns, user will need to contend with gun alignments, condenser lens adjustments, aperture alignments, biases of the samples, and other adjustments that may influence the landing energy of the beam. To run a microscope at “low kV” and to produce the best quality high-resolution images it is important to be very familiar with the system’s controls, and the parameters that need to manipulated to produce the type of image required to tell the specimen’s story. Looking to the manufacturer for this information is often the best place to begin.

When approaching an unknown sample, one that the user has never before imaged, it is a good idea to pick an arbitrary starting point. Often this is either a set of conditions where the user has had success with a similar sample, or a configuration where the user feels comfortable with the performance of their microscope. A good choice for the operation of a field emission SEM at low kV might be 1 kV. If the user knows that there are requirements to do x-ray analysis this

![SEM Image of SiO2 nanoparticles on an Al stub, imaged at 1.0 kV.](image)

**FIGURE 4.16.** SiO2 nanoparticles on an Al stub, imaged at 1.0 kV.
type of “low kV” setting might not be desired. It is always a good idea to ask if the sample is conductive, or nonconductive. Is it beam or charge sensitive? These types of questions will also have an effect over starting parameters. If the sample is charge or beam sensitive a user will often need to reduce the I-probe (probe current). This will be done in most systems by changing condenser lens settings. On certain microscope designs changing condenser settings can be disregarded, and a smaller aperture will often be all that is needed. Once the baseline for starting conditions has been established, and the sample has been prepared and inserted into the microscope, the user can begin to work out the best conditions for imaging the sample. This is a relatively unglamorous process, one that amounts to trial and error. Beginning with the baseline settings it is good to decide whether or not to increase or decrease the kV setting in response to a less than perfect image. Once this decision has been made the user can experiment with the kV settings until the state of equilibrium has been reached. This experimentation amounts to changing the kV, and observing the response of the sample. If there is more charging of the sample as a result of the choice to increase or decrease the kV setting then the user should reverse course, and find a point that is halfway between the starting point, and the last adjustment. If his adjustment does not yield better results then the user may consider going to a point that is greater or less than the starting point by the amount adjusted (e.g., 1, 0.5, 0.75, and 1.5 kV). If the charge appears to be negative, then an increase in kV is what is required, and if the charge appears to be positive, then a reduction in kV is the answer. Often very small difference in kV can result in a charge balance being reached. A good example is shown in Figs. 4.17 and 4.18. These images are of uncoated latex spheres. The difference in kV is 390 V. This point of charge equilibrium was found by stepping through the kV range in 10 V steps until the charge is dissipated. Frequently this trial and error will be a cycle that will eventually result in a charge balance on the sample. Sometime a reduction in I-probe will also be required once the equilibrium has been reached. If it is hard to find this point of equilibrium, coating may be the answer to imaging the sample provided that it is an option. Figures 4.19 and 4.20 illustrate how a small amount of coating can improve the image significantly without influencing the sample. One of the other parameters that often influences the sample charge balance and must be considered is the dwell time of the beam on the sample. Dwell time will often have an effect on the charge balance of the sample. The shorter the dwell time the better for samples which is nonconductive, or suffer from poor conductivity. A drawback to a shorter dwell time is a reduction in the signal produced by the interaction of the beam with the sample. When deceasing the dwell time leads to an image considered unusable to illustrate the characteristics of the sample a user will need to find an appropriate method to improve the image being produced. Normally this will require the use of features specific to the microscope in use. Many systems have the ability to do noise reduction. This is when the image is filtered through the use of averaging to remove the noise that results from the lowered signal production.
FIGURE 4.17. Uncoated latex spheres at 1.47 kV.

FIGURE 4.18. Uncoated latex spheres at 1.08 kV. A difference of 390 V.
FIGURE 4.19. Uncoated filter sample at 0.25 kV.

FIGURE 4.20. Filter sample coated for 5 s with Pt/Pd, imaged at 1 kV.
5. Conclusion

Low kV is not a realm where users should fear to tread. It is a gateway to the revelation of new and interesting features. It will help users discover new properties of their nanomaterials, which have been hidden by the overpowering of high kV, high beam penetration, and high-resolution SEM. Dr. Oliver Wells in the May/June 2002 Microscopy Today wrote about Jack Ramsey’s principle, “There is no best way of doing anything” [8]. This is a very fair statement, and one that is very accurate when it comes to the idea of performing scanning electron microscopy at low kV. Low kV is not always the answer to the best possible imaging of every sample. Samples often give the user clues as to where to go for the best imaging and analytical conditions. Users need to keep their eyes open for these clues, take the time to investigate the sample, and be very familiar with the controls and performance of their particular SEM. This familiarity with their system will allow them to exploit relationships of electron microscopy so as to produce the results that will best tell the story of their samples and research. After all the SEM is a tool for making the invisible, visible. Without the skills and understanding of the relationships the results will not help to elucidate the hidden world.

References