Some Thoughts on Why You Want to Use Low kV Imaging

What makes the difference between a good SEM image and a stellar one? Imaging samples at the appropriate conditions, and that often means at very low accelerating voltage (low kV). It's time to give it a try!

Every modern day scanning electron microscope (SEM) from the top of the line, ultra-high resolution field emission SEMs to the most economical entry level bench-top tungsten (W) thermionic SEMs have the capability of imaging samples at very low accelerating voltage (Low kV). Low kV imaging has many benefits and this easily accessible function should not be overlooked.

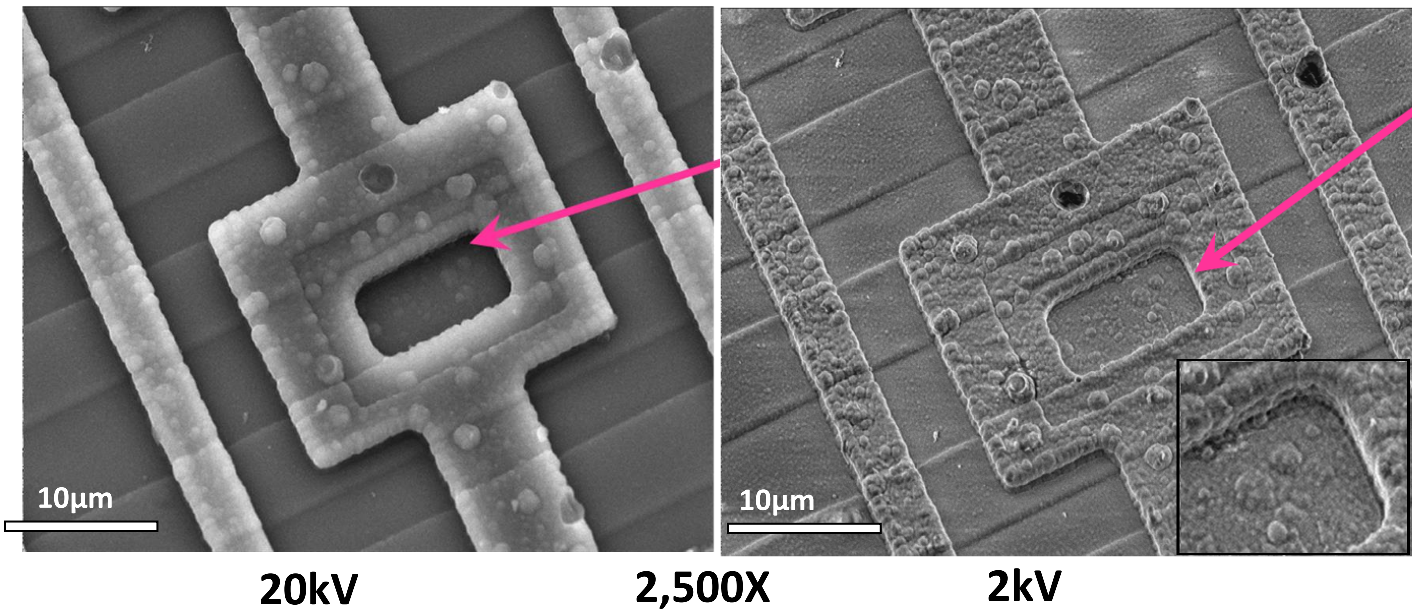


Fig. 1. A pair of images of a defect called a"Sunken Window" on a transistor. One image is 20kV; the other is 2kV. While the 20kV image appears at first to be OK, edge effect washes out all corners and edges and depth of penetration minimizes all surface information. These two factors prevent one from seeing the actual defect (see arrows and insert).

Electron Detectors and Signals

All SEMs use two basic imaging detectors/signals for most applications. Secondary electrons (SE) are very low energy (<50eV) electrons, and as such escape from only near the surface of the sample. In general they are considered to carry only "topographic" surface information. These electrons are typically collected with an Everhart-Thornley scintillator/ photomultiplier detector with a bias on it to attract the low energy electrons to the detector. Backscattered electrons (BSE) are higher energy electrons (up to nearly the accelerating voltage of the incident beam) and as such escape from a much deeper & wider volume of excitation within the sample. In general they are considered to carry only "Z contrast" (contrast based on differences in mean atomic number or average density). These electrons are typically collected with a solid state or YAG detector placed directly above the sample. Because the BSEs are high energy there is no collection bias on this detector and the detected electrons are "line of sight."

While both of the previous definitions are true they do not paint a complete picture. One of the most common questions asked about any SEM is "What is the resolution?" This is a question with seemingly many different answers and most of them seem non-intuitive! One has to first define what type of electrons you are measuring with (SE or BSE), and what are the SEM operating parameters such as: kV, WD and probe current (often loosely defined as spot size or probe diameter).

Resolution and Contrast Mechanisms

What we were all taught (especially years ago) in SEM 101 was that theory says "as the kV is increased resolution is improved". We were also shown that SE signal is independent of atomic number & depends only on micrometer scale surface topography. This is true only for SE imaging as the SE signal comes only from the surface and at a lateral resolution that is a direct function of the spot size..., or is it? SE signals iare not only generated by the beam at the point of impact, but by BSEs escaping from some depth and breadth below the surface generating SEs on their way out of the sample (called SE IIs). These not only degrade the spatial resolution of the SE signal but they carry no surface topography information and do carry mean atomic number information. There is no reduction in the SE signal coming from the probe at impact; it is just now riding atop a lot of other signal so it is proportionally a smaller part of the total signal. BSE resolution, while limited by probe diameter at high mag (which improves at higher kV), is inversely proportional to kV. This means as the kV is reduced the volume of excitation is reduced, and that reduction in excitation /emitted volume improves the BSE spatial resolution, not linearly but exponentially. This means even a small reduction in kV can have a large impact on BSE or EDS resolution. The BSE signal is a much smaller component than the SE signal so at some point the increase in probe current and its associated larger spot size may negate any advantage of the smaller excited volume as the two approach the same size. This is especially true for W SEMs as opposed to FE SEMs where larger current and lower kV have almost no impact on the smallest spot size one can generate. The addition of specimen biasing at low kV not only improves probe size but also accelerates low energy BSEs that may not have been energetic enough to excite the detector to a voltage that is detectable. These statements do not in any way mean that you can't or shouldn't do low kV imaging on a W SEM! The net benefit of low kV imaging for BSE is obvious due to the smaller excited volume. The net benefit for SE imaging is that as you lower the kV you get an increase in the ratio of the amount of signal generated at the surface (true topography). High kV SE imaging at low to moderate magnifications can often make macroscopic scale surface contrast disappear due to the signal coming from a large depth & breadth within the sample. At higher magnifications micrometer to nanometer scale surface contrast disappears or appears translucent and edges appear washed out due to secondary electrons escaping from the top, sides & bottom of the features of interest simultaneously. This is referred to as edge effect.

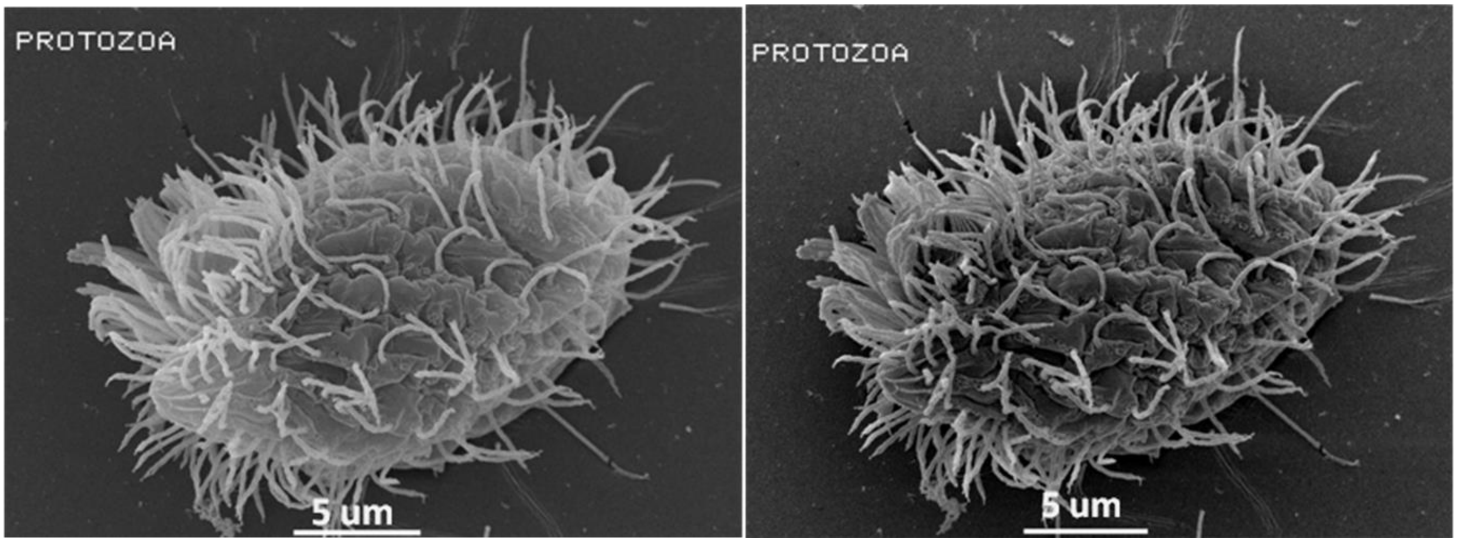
The mindset of yesterday was "How high of a kV do I need to get the resolution I need?" This should be replaced with "How low in kV can I go before I run out of resolution?" As mentioned before with a W SEM there may be some practical limits to low kV high mag imagined due to the fundamentals of the W source (C_c, source size, gun bias voltage, etc.) but within normal imaging parameters we use W SEM at low kV all the time. With a FEG SEM there is almost no limit to how low you can go in kV and still generate phenomenal images. In reality, it isn't always about "resolution". It is often about obtaining the correct signal from the correct region of the sample that yields contextually pertinent information and answers the question that you want to ask.. This is often loosely defined as "image quality."

Conclusions

The benefits of low kV imaging include: 1) There is less sample damage especially on biological or polymer samples, 2) Nonconductive samples can be imaged without the need to apply a conductive metal or C coating which can totally obscure surface information, 3) Thin or nanoscale surface structures that may be invisible due to beam penetration at high kV (surface sensitivity) are now easily visualized, 4) The edge effect artifact which washes out edge, corner and surface contrast is dramatically reduced due to the small excitation volume , 5) Low kV also improves EDS and WDS spatial resolution for the same reasons as it does so for BSE imaging.

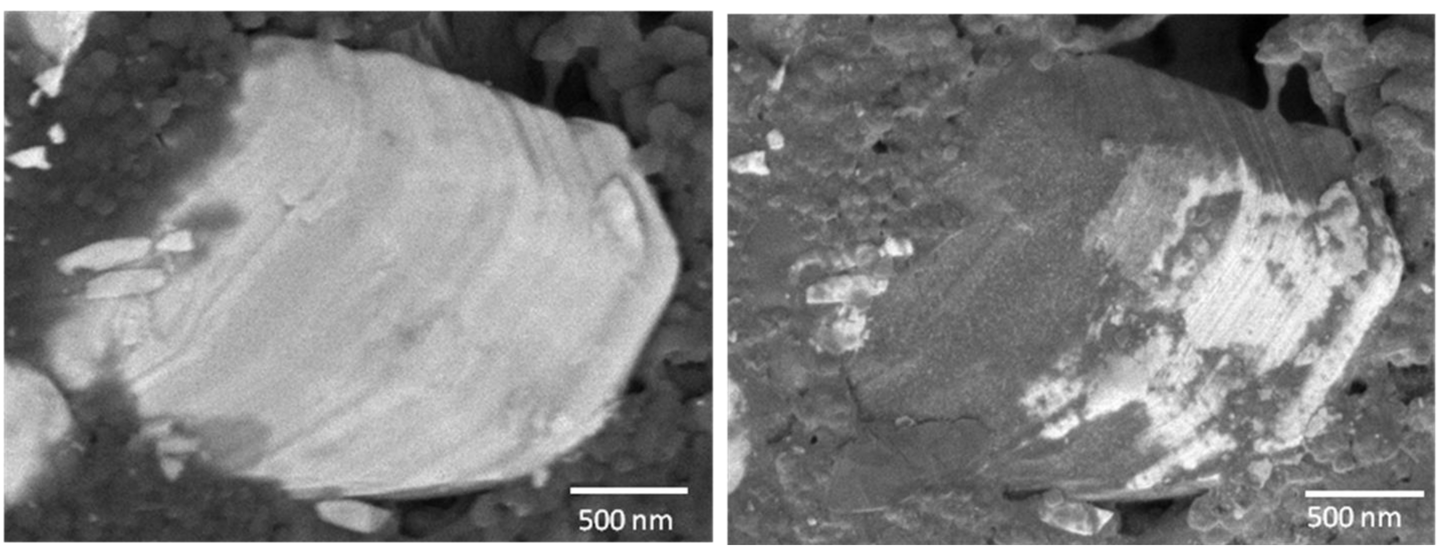
A few examples and comparisons are shown. The bottom line…..

Don't revert to heavily coating the samples and running your SEM at 15-20kV. Low kV is the way to go!.



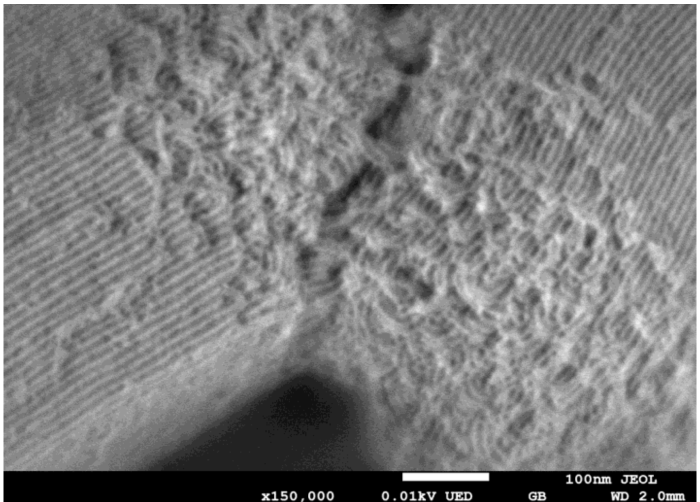
10kV **5kV**

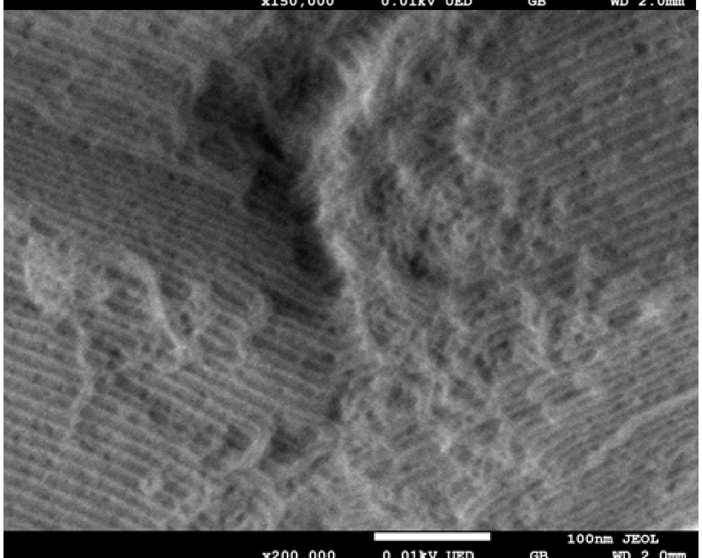
Fig.2 A pair of images of a protozoan that has been Au/Pd sputter coated. Even though the sample does not charge at high kV, edge effect masks all of the contrast from the cell surface and especially on the smaller cilia. The 5kV image has much more surface detail than even the 10kV image.

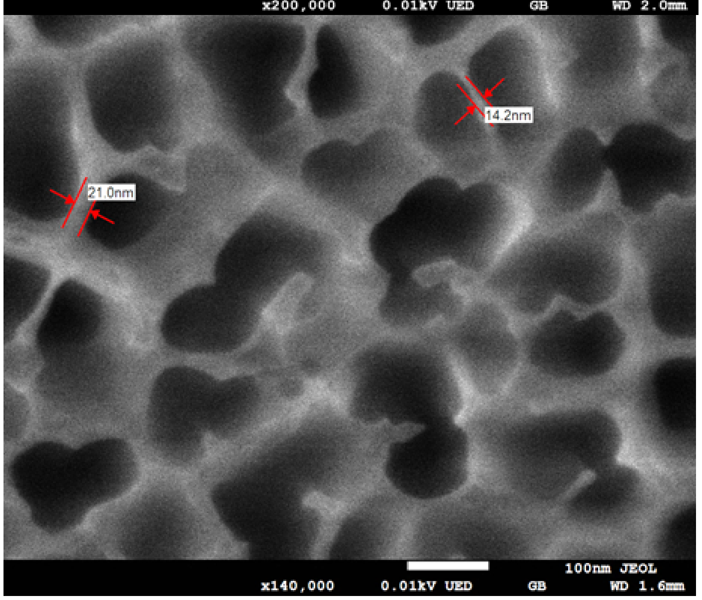


0 kV **WD 2.0mm** **0.5kV**

Fig. 3. A pair of BSE images of a Li ion Battery CoO anode at 3kV (which in days gone by one might have thought of as low kV for BSE imaging) and at 0.5kV (BSE imaging conditions that were impossible not so long ago). The thin carbonaceous layer is clearly visible covering the metal at the lower accelerating voltage.







Sample:
Mesoporous Silica

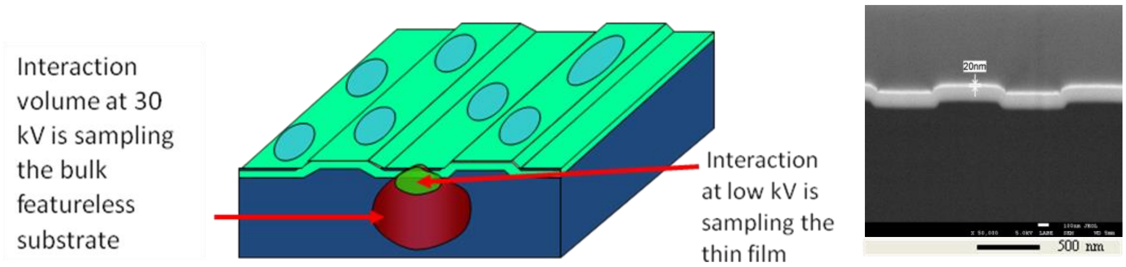
Mesoporous Silica and Alumina Anopore Membranes are: nonconductive, porous, have poor SE yield, charges easily and have structures at the single digit nm scale.

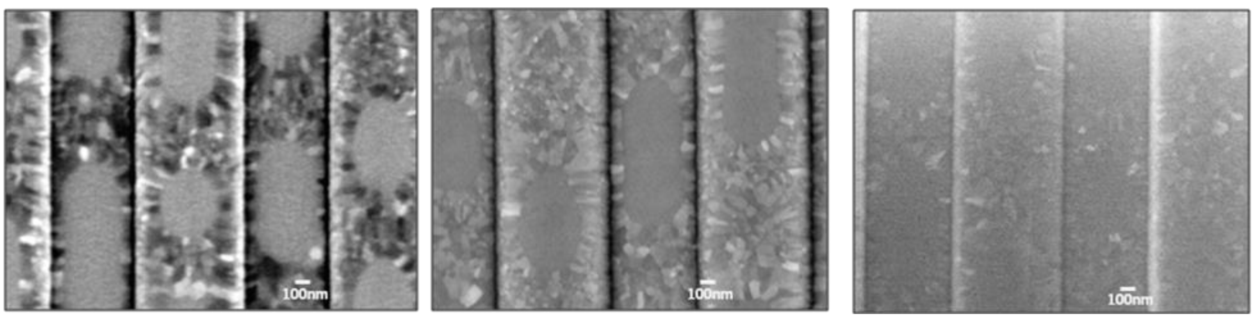
These types of samples need to be imaged at high mag and ultra-low voltage to eliminate charging and edge effect.

Any conductive coating is not an option!
At these kVs the beam will not penetrate the coating, change the size of the structures you are imaging and completely obscure any surface topography.

Sample:
Anopore Al_2O_3 Membrane Filter

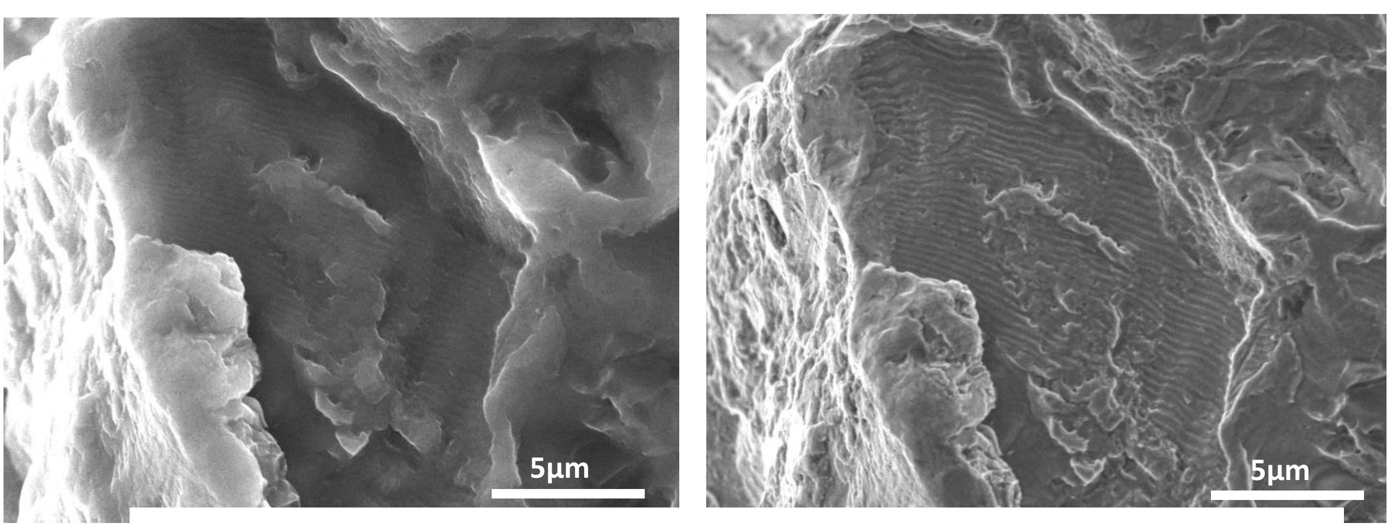
All Images at 10 Volts
at 140,000X -200,000X





0.8 kV **5kV** **30kV**

Fig.4. Backscatter images of crystal orientation contrast (ECP or electron channeling contrast) in the 20nm thick surface film that carries the data on a DVD. High kV BSEs emanating from below the surface carry little or no surface contrast and add a large component to the signal. These two factors greatly reduce the ability to see the grain orientation contrast.



25kV **4,500X** **5kV**

Fig. 5. An aluminum fatigue failure imaged at high and low kV. Because Al is a light (low atomic No.) metal, and self oxidizes, using a high kV penetrates deep into the metal and the signal that comes from below the surface masks the fatigue striations on the fracture surface.