

# Low kV Multispectral Imaging in a Field Emission SEM

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Successful imaging and characterization of nanomaterials, composites and biological specimens require unsurpassed performance in the low and ultra-low kV range in FEG-SEM for both imaging and analytical modes. The state-of-the-art FEG-SEM should show superb resolution with various detectors at a wide range of accelerating voltages and have the ability to perform fast and reliable microanalysis at low kV using high probe currents, without loss of spatial resolution and with an ease of operation.

The new JSM-7600F Schottky FEG-SEM from JEOL combines two proven technologies – an electron column with semi-in lens detectors and an in-the-lens Schottky field emission gun - to deliver ultrahigh resolution combined with wide range of probe currents for all applications (from 1pA to more than 200nA). It delivers unsurpassed imaging performance at ultra low kV, with 1.5 nm resolution at 1 kV and 5 nm at 0.1 kV.

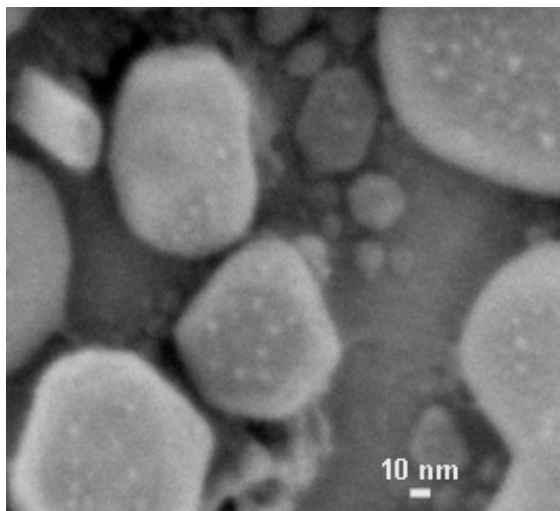


Figure 1. Au on C specimen imaged with JSM-7600F at 100V accelerating voltage.

This paper highlights some of the unique features of the new JEOL JSM-7600F SEM that

enable unmatched imaging and analytical performance, including the Aperture Angle Control Lens (ACL), the new patented LABe detector (low-angle backscatter detector) and the Gentle Beam mode (application of a specimen bias) that allows operation down to 100 volts (see Figure 2).

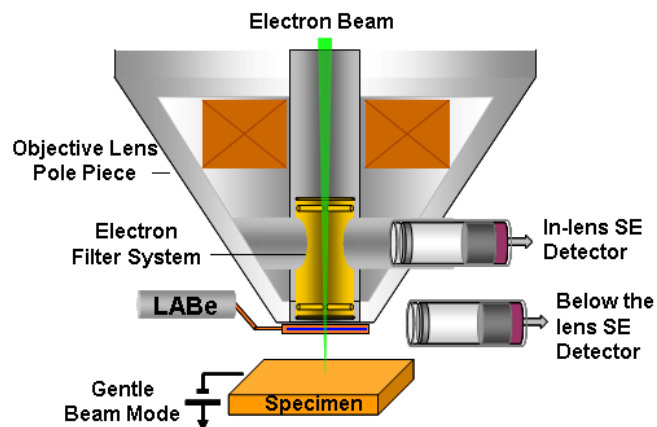


Figure 2. General schematic of the detector system in the JSM-7600F.

In particular, we focus on new analytical performance (EDS, WDS and CL) as well as backscatter imaging data acquired at low and ultra low kV.

## Vector Filtered BSE Imaging

Traditionally BSE imaging required high or moderately high kV due to smaller electron yield in BSE, detector sensitivity and bandwidth at lower probe current, low energy electron sensitivity and a single (typically solid state) detector. Even with all of these limitations BSE detectors have been very useful at low to medium magnifications, especially for highlighting compositional or topographic differences in the samples [1].

Recent developments by JEOL in detector and SEM technology have allowed significant progress towards BSE imaging at low kVs with high resolution. JSM-7600F incorporates a new and unique LABe backscatter detector that is based on this patented JEOL technology.

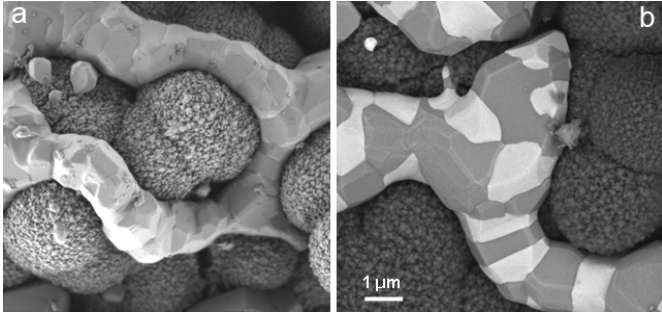


Figure 3. LAbE images of an uncoated ceramic eutectic. (a) 0.2 kV BSE image showing enhanced surface detail, (b) 5 kV BSE image showing more traditional compositional and crystallographic contrast.

The combination of the geometry of the LAbE detector, specimen position (Working Distance) with respect to the detector as well as accelerating voltage and application of specimen bias effectively allow Vector Filtered BSE imaging (Figure 3). The LAbE detector can serve as a conventional BSE detector under “normal” operating conditions i.e. moderate kV and analytical WD. In this configuration it is a high angle BSE detector yielding Z contrast and channeling contrast.

The LAbE detector can also collect *only* the low angle BSEs which due to the angular dependence convey high spatial resolution. Low angle BSE’s escape only from the top few nm although they are generated throughout the entire interaction volume of the specimen. By lowering the accelerating voltage down to few kVs or even few hundred volts the beam/specimen interaction volume is also significantly reduced, yielding true surface information as well as crystallographic contrast which is similar in appearance and information, but fundamentally different in its generation from channeling contrast. Figure 4 shows an image of nano-grained Au imaged at 0.5 kV. The image shows twinning (orientation contrast) in Au crystal with very high spatial resolution compared to a regular BSE image.

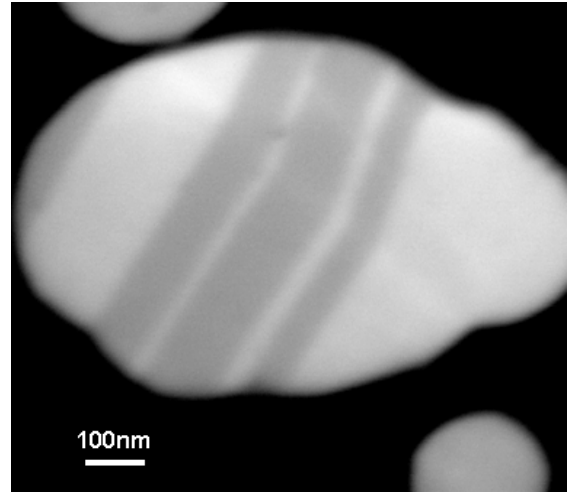


Figure 4. Nano-Au grain imaged at 0.5 kV with the LAbE detector. The sample was prepared using JEOL Cross Section polisher.

Applying variable specimen bias via the Gentle Beam (GB) function in combination with the LAbE detector offers a remarkably new perspective for imaging nano-materials. An example of the combination of the GB and the LAbE for low kV BSE imaging is shown in Figure 5. The 1 kV image clearly shows enhanced surface information (including surface contamination) when compared to the 2 kV image. The images taken with no specimen bias show typical BSE compositional and crystallographic information. An increase in specimen bias (from 0 volts to 2 kV) somewhat suppresses the more traditional compositional contrast and shows more enhanced surface detail as well as additional topographic information. Therefore, choosing a combination of specimen bias and kV will dictate the type of information obtained with the LAbE detector.

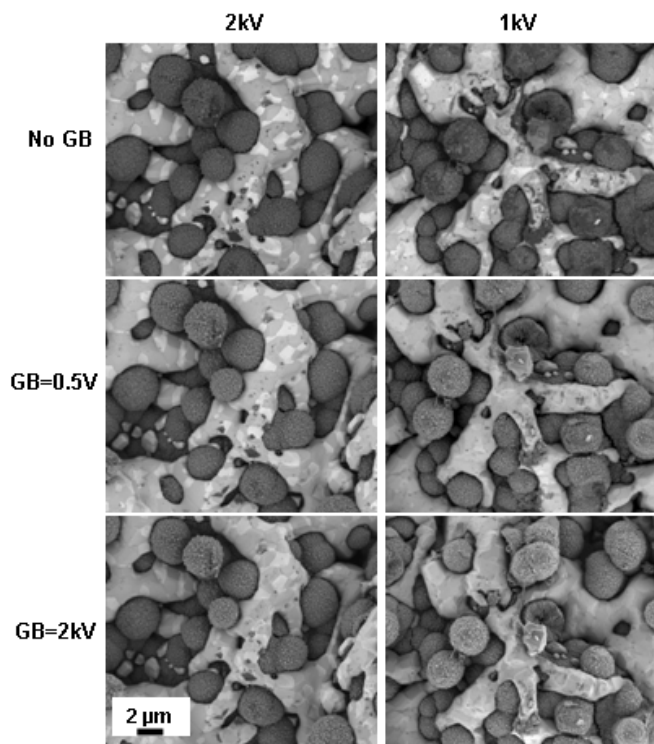


Figure 5. An uncoated ceramic eutectic imaged with variations of kV and specimen bias using the LAbE detector.

### Optimization of the Alpha Angle for Analysis

One of the unique features of the JSM-7600F is the patented Aperture Angle Control Lens (ACL) that automatically optimizes for both high resolution imaging at the lower probe currents and high spatial resolution X-ray analysis at high probe current with a seamless transition between the two, essential for rapid analysis and optimized image quality. The ability to increase the probe current for fast microanalysis, while still maintaining a small spot size and small volume of excitation for high resolution, has been the holy grail of microanalysis in SEM. This is particularly true for low kV microanalysis. The ACL works by taking into account effects of all aberrations (such as spherical aberration and diffraction limitations) on spot size and optimizing the alpha angle accordingly in an automatic fashion.

When the SEM is optimized for the smallest spot size (largest alpha angle) there is some beam tailing that produces X-rays from areas “not in the spot.” For low beam current applications this is

insignificant. The best analytical data comes from the smallest alpha angle. Therefore, when the ACL is optimized for image resolution, the resulting high current image (large alpha angle) has a somewhat ‘hazy’ background but shows great resolution. However, when the ACL is optimized for analytical work the ultimate resolution is slightly decreased, yet the analytical signal is no longer affected by the beam tailing, resulting in smaller analytical signal delocalization (Figure 6).

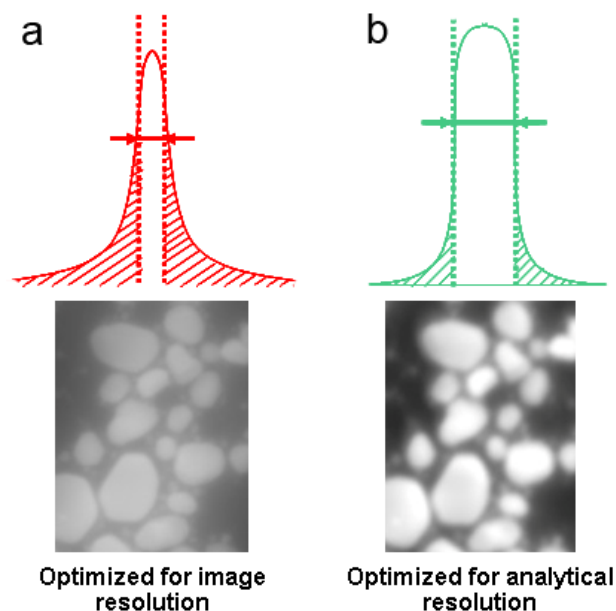


Figure 6. ACL optimization at high beam currents for (a) image resolution, and (b) analytical resolution. Au on C images at 15 kV, 20kX and 198 nA.

The automatic optimization of the ACL maintains high resolution imaging at a wide range of probe currents, from few picoamps to hundreds of nanoamps, as illustrated in Figure 7. The figure shows images of Au on C taken using 5 kV with various probe currents at the EDS working distance. The resolution at 5 kV is 2.4 nm at 50 pA, 4.5 nm at 5nA, and 12 nm at 100 nA. This beam resolution allows very fast acquisition (using high beam current) of EDS or WDS data at low kV with high spatial resolution.

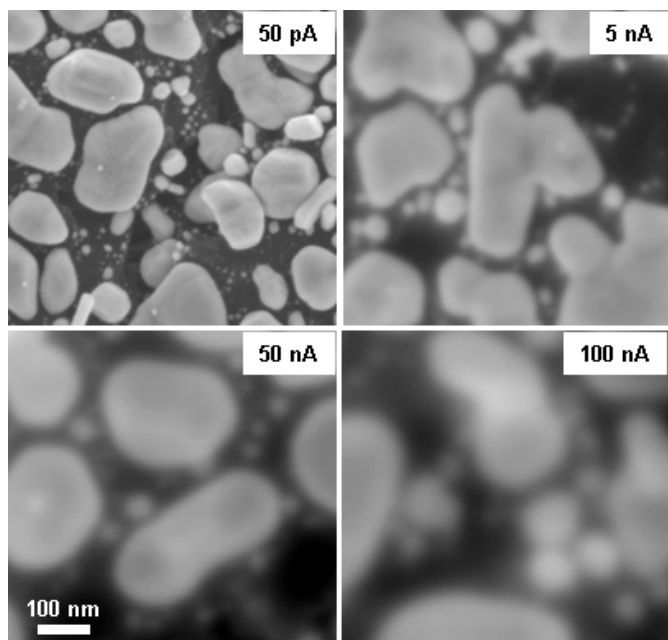


Figure 7. Au on C resolution images at the EDS WD with various probe currents ranging from 50 pA to 100 nA. The resolution at 5 kV is 2.4 nm at 50 pA, 4.5 nm at 5 nA, and 12 nm at 100 nA.

Implementation of the ACL function allows collection of very fast EDS maps at high spatial resolution on bulk samples using low kVs. One such example is shown in Figure 8. The map shows an EDS map of Sphene (yellow), Apatite (blue) in a magnesium aluminosilicate matrix (red) with sub-100 nm resolution. The map was collected at 8 kV for 20 min using beam current of 20 nA.

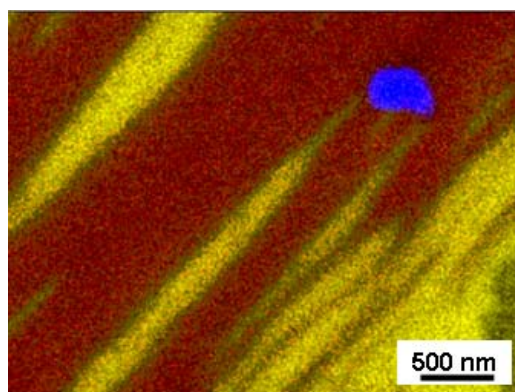


Figure 8. EDS map of Sphene  $\text{CaTi}(\text{SiO}_4)(\text{O},\text{OH},\text{F})$  (yellow), Apatite (blue) in a magnesium aluminosilicate matrix (red) with sub-100nm resolution. The map was collected at 8 kV for 20 min using beam current of 20 nA.

Certain specimens, especially those containing low Z materials, require low kV EDS to correctly identify elemental distribution. Additional advantage of high beam current/small spot size is the ability to collect EDS maps very fast (in a few minutes) even at low accelerating voltages. Therefore, beam sensitive specimens remain damage-free during the analysis.

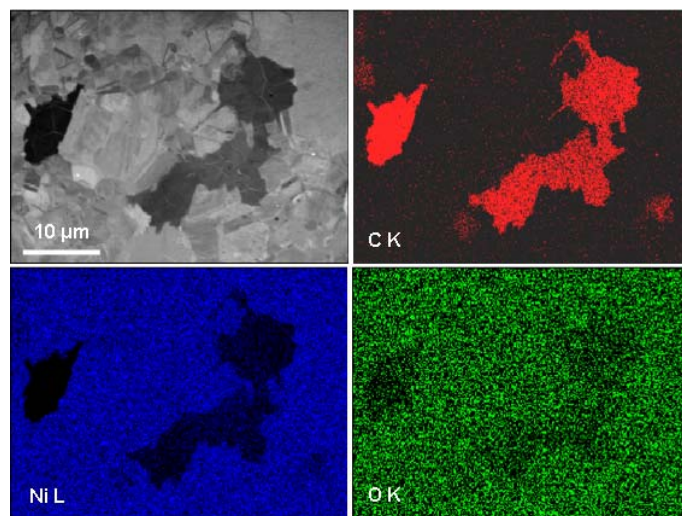


Figure 9. SE image and corresponding EDS maps of graphene layers on a Ni substrate. The maps were collected at 2 kV for 2 min using a beam current of 10 nA.

Figure 9 shows EDS maps of graphene (C) layers on a Ni substrate. Because the graphene layers are only few Angstroms thick it is necessary to minimize the interaction volume as much as possible to eliminate X-Rays from the bulk when conducting the EDS analysis in order to correctly identify the distribution of graphene on the Ni substrate. Additional consideration was given to minimize the analysis time in order to determine the graphene distribution unambiguously without building up of any possible Carbon contamination on the surface which would obscure the analysis. Thus, the EDS maps were collected for 2 min at 2 kV and a beam current of 10 nA. The high beam current used for this analysis ensured adequate data collection for compositional interpretation even with a very short acquisition time.

The ability to deliver high beam current also impacts WDS and CL (cathodoluminescence)

analyses where large beam currents are needed for sufficient signal-to-noise collection.

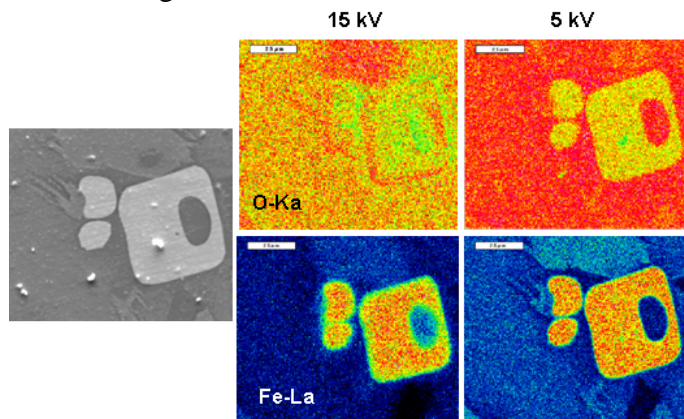


Figure 10. Comparison of WDS maps of Miake Island Ore geological thin section collected using 15 kV and 5 kV (only 2 representative maps shown, O-K $\alpha$  and Fe-La). Scale bar = 2.5 microns.

In WDS, the detector has a small solid angle for detection, therefore high accelerating voltage and the associated high beam currents are usually employed to detect enough of the signal to efficiently obtain good elemental data and maps. Lower accelerating voltages are desirable when thinner or more surface-oriented features are to be investigated. Less signal is generated, normally requiring longer mapping times unless the beam current is increased, resulting in poorer spatial resolution in the maps. In the JSM-7600F, the ability of the ACL to keep the spot size small when the beam current is high results in efficient WDS mapping with high spatial resolution even at lower kVs. The comparison of maps in Figure 9 illustrates the results that are possible with ACL corrected, high beam current operation for WDS applications.

Cathodoluminescence detection in the SEM has been historically associated with high kV, high beam current microanalysis, because of the weak CL signal, and the spot size limitations at low voltages. At high kVs, the CL signal which comes from the same volume of excitation as X-Rays has poor spatial resolution and has often been quite poor due to insufficient probe current in the SEM. When high spatial resolution CL is needed or when

highly charging specimens need to be examined it is crucial to be able to work at low kV while still delivering a large probe current into a small spot. These limitations are a thing of the past with the new JSM-7600F design. Using JSM-7600F we can collect the CL signal from specimens using accelerating voltages as low as 0.5 kV (the maximum beam current at 0.5 kV is ~11 nA, which is more than sufficient for CL). Figure 11 shows CL images of an uncoated diamond specimen collected using 2 kV and 0.5 kV. While CL data at 2 kV clearly has better resolution, it is quite remarkable that the CL signal can be collected at 0.5 kV in cases where the region of interest may be a thin surface film or layer.

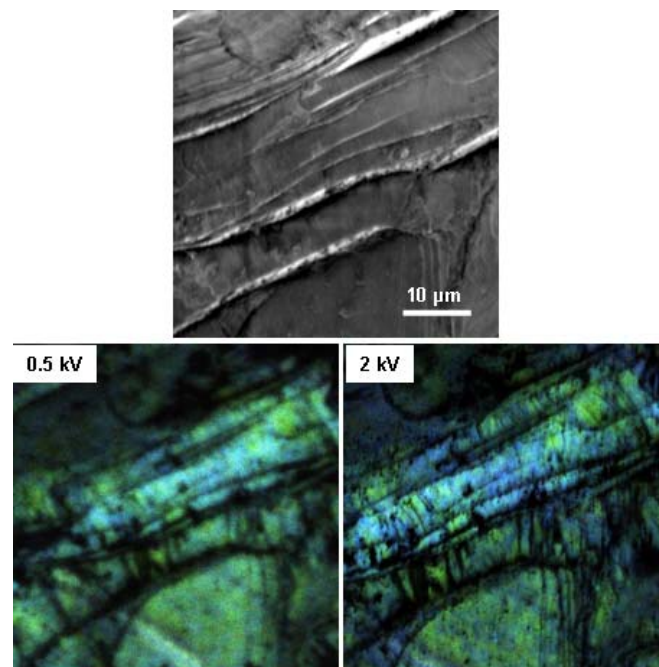


Figure 11. SEM and corresponding CL images from diamond collected using a Gatan Chroma CL.

## Conclusions

JEOL's JSM-7600F delivers the ultimate in a Schottky FEG-SEM. In addition to providing the

state of the art in ultra low kV imaging that is expected in today's high end FEG-SEMs, JEOL's patented ACL technology enables the user to automatically move from high resolution imaging at low probe currents to fast high spatial resolution microanalysis. JSM-7600F can perform very fast microanalysis (EDS, WDS or CL) at low kV and high spatial resolution. The LABe detector offers unique insight on materials through Vector Filtered BSE imaging. This detector also delivers unsurpassed compositional and/or topographic contrast with high spatial resolution at low kV that is required in many nanomaterials applications.

The JEOL JSM-7600F is a highly versatile Schottky FEG-SEM capable of delivering the highest probe currents in the industry for fast microanalysis with superior spatial resolution without sacrificing the ability to yield ultrahigh resolution imaging whether at high or ultra low kVs.

## **Acknowledgements**

1. See for example: J. Goldstein et al, Scanning Electron Microscopy and X-ray Microanalysis, Third Edition, 2003, New York: Plenum
2. All EDS data were collected using a ThermoFisher System 7 with 30 mm<sup>2</sup> SDD.
3. The authors would like to thank SIFCO for providing the sample in Figures 3 and 5.
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4. The authors would like to thank Dr. D.C. Bell from Harvard University for providing the sample in Figure 11.