Low Voltage Scanning Electron Microscopy

David C Joy* and Dale E Newbury**
*University of Tennessee, Knoxville, TN
**NIST, Gaithersburg, MD

Low Voltage Scanning Electron Microscopy (LVSEM), defined as operation in the energy range below 5 keV, has become perhaps the most important single operational mode of the SEM. This is because the LVSEM offers advantages in the imaging of surfaces, in the observation of poorly conducting and insulating materials, and for high spatial resolution X-ray microanalysis[1]. These benefits all occur because a reduction in the energy E_O of the incident beam leads to a rapid fall in the range R of the electrons since $R \sim k.E_0^{1.66}$. The reduction in the penetration of the beam has important consequences. Firstly, volume of the specimen that is sampled by the beam shrinks dramatically (varying as about E_0^5) and so the information generated by the beam is confined to the surface of the sample. Secondly, the yield ⇦ of secondary electrons is increased from a typical value of 0.1 at 20 keV to a value that may be in excess of 1.0 at 1 keV. The yield ↑ of backscattered electrons also varies, although less dramatically, tending to rise for elements of low (Z<30) atomic number and fall for those of high atomic number. Thirdly, because of the large increase in the secondary electron signal the total emitted electron yield can reach unity at certain energies. When this occurs no net charge is deposited in the sample and hence even insulators can be observed without the problem of charge build-up. There are also some potential disadvantages to LVSEM operation. Images will be highly sensitive to the state of the specimen surface and thus contamination can be expected to be a major problem, and the rapid reduction in the size of the interaction volume means that the incident beam dose is concentrated. As a result beam induced radiation damage is enhanced even though the size of the region over which it occurs is reduced.

It is difficult at low beam energies to match the electron-optical performance that can be achieved at higher voltages. This is because the brightness of any emitter varies linearly with the beam energy and so the performance of all electron sources is significantly degraded at low voltage. In addition the combination of the chromatic aberration of the probe forming lens and the energy spread of the beam produces a substantial 'skirt' around the probe which increases its diameter, spoils the resolution, and lowers the contrast in images. At the lowest energies (*i.e.*, below 250 eV) the relatively large wavelength of the electrons still further worsens the resolution because of diffraction, and the depth of field of the instrument is also reduced. Despite these problems current LVSEMs, employing conventional optics can achieve resolutions of 2 nm or so at 1 keV when using a field emission gun. If retarding field optics are employed this level of performance can be maintained to energies below 100 eV (figure 1).

X-ray microanalysis performed in the low voltage regime (E_0 <5 keV) offers special advantages compared to the conventional beam energy range (15< E_0 <30 keV), but it is also subject to constraints that can be severely limiting. On the positive side, the decrease in the electron range at low voltage is complemented by an even sharper drop in the x-ray excitation range, $R_x \sim (E_0^{1.67} - E_C^{1.67})$. The shallow x-ray range results from operating close to the critical excitation potential E for many elements and leads to sampled masses in the femtogram range and lateral resolution below 50 nm. Because of the low overvoltage ($U=E_0/E_c$) and because the x-ray generation is so shallow, often 100 nm or less, the matrix corrections that result from electron scattering and retardation and x-ray absorption tend towards values near unity, in principle reducing the associated error.

On the negative side, the low beam energy severely restricts the atomic shells that are accessible for intermediate and high atomic number elements, and even for those shells, the low fluorescence yield and the low overvoltage result in low spectral peak/background (P/B). The combined impact of these effects is to restrict access to trace concentration levels (arbitrarily defined as C < 0.01), and in some particularly difficult cases, even minor constituents (0.01< C < 0.1) prove difficult to detect. Low voltage microscopy is often effectively applied as a means to avoid, or at least diminish, charging effects in images of uncoated insulators. While stable imaging can often be achieved in such cases, x-ray microanalysis under these conditions must be performed with special care. Because the overvoltage is so low, even minor charging can significantly alter the efficiency of x-ray generation, which depends exponentially upon overvoltage: $I \sim (U-1)^n$, where *1.5* < n < 1.7 (figure 2). To test for charging, the analyst can use the EDS spectrum to monitor the Duane-Hunt limit of the x-ray bremsstrahlung, which is equal to the energy of the beam electrons as they strike the sample. However, charging is often a dynamic process so that monitoring must be performed with adequate time sampling. Otherwise, the observed relative peak heights for a mixture of elements can be extremely misleading[2]. ■

1. Joy, D.C., Joy, C.S., (1996), 'Low Voltage Scanning Electron Microscopy', Micron 27. 247-263
2. Newbury, D.E. "Measures for Spectral Quality in Low-Voltage X-ray Microanalysis," SCANNING, 22 (2000) 345-351.

Figure 1: SE image of Au/PD film recorded At 30 eV beam energy. Field width 2 μm

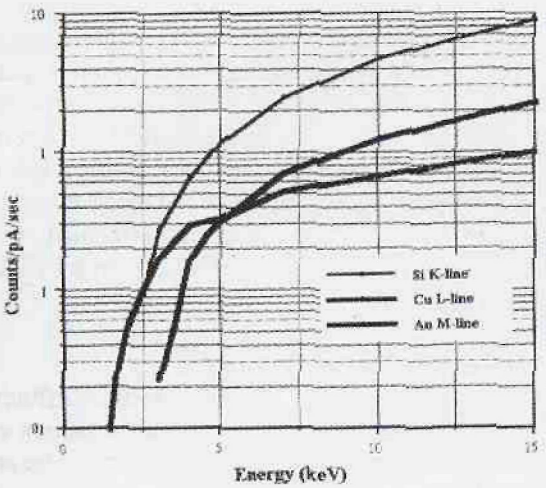


Figure 2: Experimental variation of X-ray generation efficiency with energy for Si K, Cu L, and M lines

Three Techniques, One PC, One System

INCA - *Fast, Integrated Microanalysis*

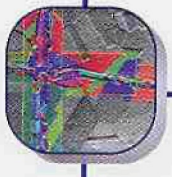
Switch between techniques at the click of a mouse

INCAEnergy

- Superior EDS accuracy
- Rapid results
- INCAEnergy+ EDS speed/WDS resolution

INCAWave

- Powerful WDS
- Ultimate accuracy in SEM
- High sensitivity for trace elements



INCACrystal

- High speed EBSD
- Enhances performance with unrivalled support
- Easy-to-use INCA interface

For your information pack on INCA, e-mail to:
info@ma.oxinst.com or contact us at the address below

Oxford Instruments Inc, 130A Baker Avenue Ext., Concord, MA 01742, USA.
Tel: +1 978 369 9933 Fax: +1 978 369 8287
www.oxford-instruments.com

OXFORD INSTRUMENTS