

Towards Optimal Imaging and Microanalysis in Variable Pressure and Low Voltage SEM

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Image quality of 'secondary electron imaging' has been an ongoing operational issue with the variable pressure range of scanning electron microscopes (VPSEM), including the extended pressure range environmental or SEM. A second question that has received considerable attention concerns charge cancellation on insulators in VPSEM with particular reference to effects of charging on x-ray microanalytical results.

Optimal imaging

The main parameters affecting the image quality are accelerating voltage, primary electron beam current, working distance, gas pressure, gas type, detector bias, and detector design. In this study, the operating parameters have been measured in terms of their influence on contrast in "secondary electron" imaging of a conductive sample in a variable pressure SEM. Contrast here is used to describe the ratio of image signal levels between two features in a sample and it has been considered in this example as an expression of image quality. There were two aims of the initial phase of this project: a) the characterization of the influence of these parameters from an operational perspective, and b) to identify the optimal imaging

conditions for a "current generation (2003)" LEO 1555 FEG VPSEM and those for a "first generation (1990)" ElectroScan E-3 (tungsten filament) ESEM.

The first test sample was a 300 μm diameter silver sphere mounted on a carbon tab on a conventional Al SEM stub. This specific sample was chosen to provide a high level of contrast variation between the silver and carbon components, because of their widely different SE yields. A simple technique to provide an objective and comparable measurement of the "contrast" was used. For comparative purposes, the average intensities of two 100 x 100 pixel regions (75 x 75 μm) were measured using the histogram feature of Adobe Photoshop. One region was from the "flat" region on the top of the sphere, and the second region was from a featureless area of the surrounding carbon. The collected digital images were 8-bit, allowing a potential range of 0-255 in each pixel. For each measurement series, the 'brightness' and 'contrast' controls on the VPSEM were adjusted so that all images within a data set were within the digital limits.

Data sets for specimen chamber gas pressure (0.2 – 8 torr), accelerating voltage (1 – 30 kV), working distance (4 – 20 mm), and "gaseous secondary electron" detector bias (200 – 650v) were collected for the ElectroScan ESEM. The absolute data for each of these variables are coherent and in absolute terms show the typical gas amplification curves, with pronounced maxima, reported by a range of researchers from VPSEM (figure 1).

The data show different trends when plotted as a signal ratio (figure 1). This ratio is effectively an image 'contrast' measure, noting that by choice of sample, a major difference should exist between the measurement from the silver relative to the carbon-rich substrate. The

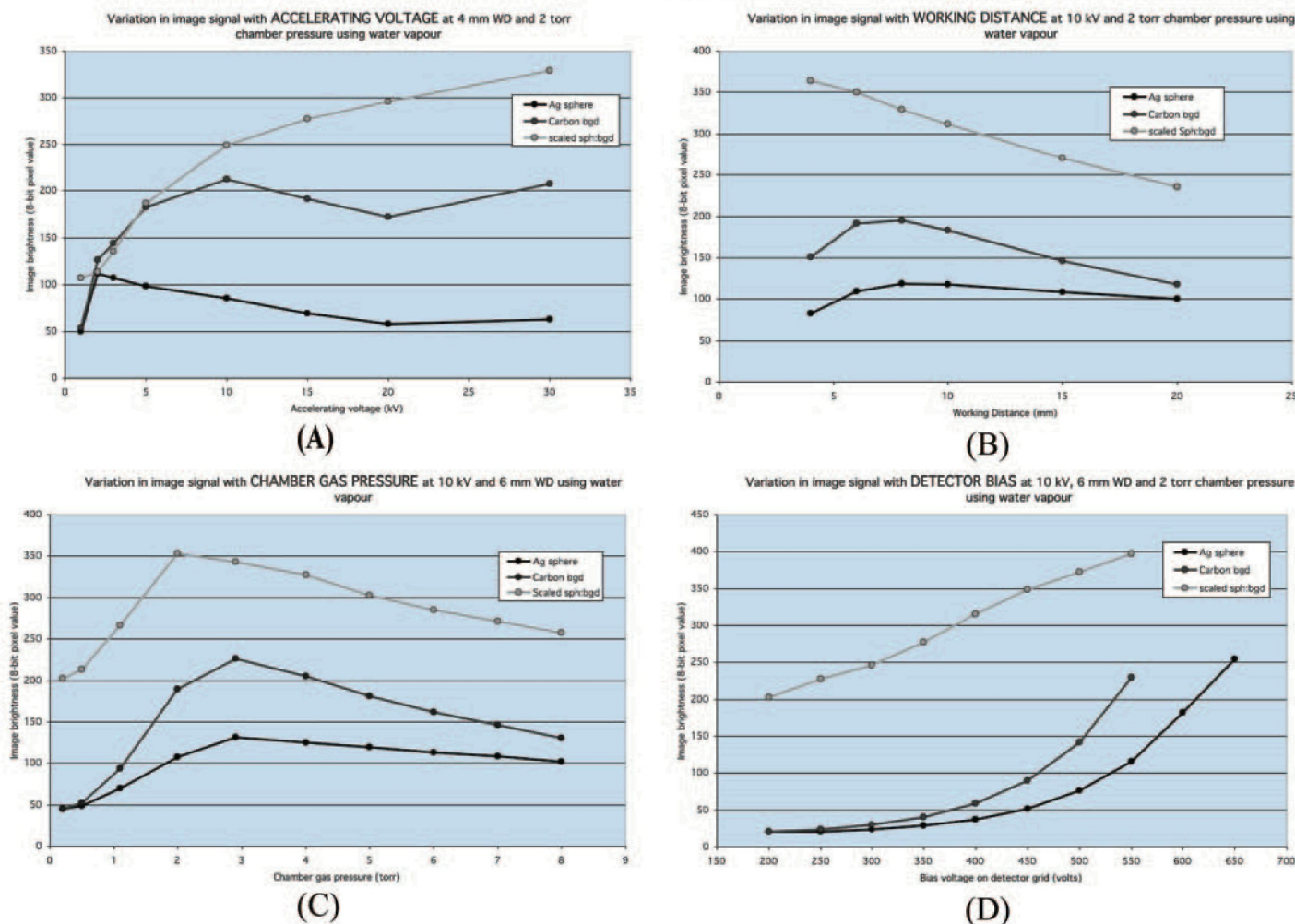


Figure 1: Image contrast variation with accelerating voltage (A), working distance (B), gas pressure (C), and detector bias (D) under conditions as noted from a silver sphere on a carbon tab background in an ElectroScan E-3 ESEM.

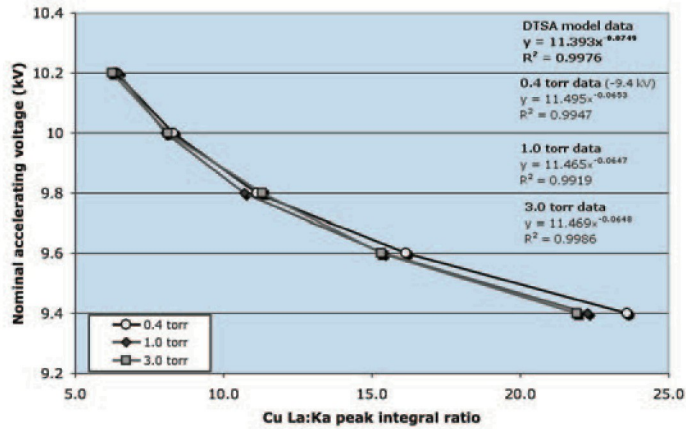


Figure 2: Variation in Cu L α :K α peak ratio with accelerating voltage and gas pressure from EDS x-ray spectra from a grounded polished Cu sample in an ElectroScan E-3 ESEM and DTSA model data.

variations in detector bias and working distance gave linear changes in this contrast ratio, whereas, the effect of change in accelerating voltage is a power function and the response to gas pressure variation is a typical gas amplification curve. Notably, the maximum of the contrast ratio for variation in gas pressure occurs at a different pressure to those for the individual contrast data.

Towards optimal microanalysis

The second phase of this project was to evaluate the potential of using an elemental L:K x-ray peak intensity ratio as an indicator of charging rather than the Duane-Hunt Limit (DHL). Three reasons prompted this work: (a) the large error associated with the DHL measurement due to the non-linear decay of the background and the poor measurement precision as very low counts are usually recorded in this part of the spectrum, (b) the inability to measure the DHL when using high accelerating voltage (e.g. 30 kV) as most systems are set to an upper x-ray collection limit of 20.48 keV (1024 channels @ 20ev/ch), and (c) a desire to study time related effects and so the need for quick and accurate measurements.

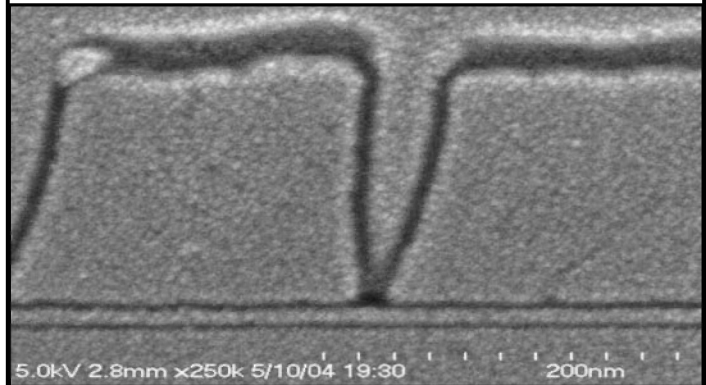
The concept was that that the intensity of an x-ray emission is highly sensitive to the overvoltage ratio (the ratio of the accelerating voltage to the excitation energy). Therefore any reduction in the accelerating voltage, and more exactly the primary electron landing energy, will have a significant and measurable effect on an x-ray peak whose excitation energy is close to, but below the accelerating voltage in use. If a sample is used that has x-ray emissions near, and also well below the accelerating voltage, the ratio of the peak integrals will be a measure of sample charging. This assumes that the reduction in landing energy is a consequence or the build-up of a net negative sample surface charge. Copper analyzed at an accelerating voltage of 10 kV satisfies these conditions, with the copper L α peak at 0.93 keV and the copper K α peak excitation energy at 9 keV.

Two approaches have been followed. Data has been gathered from simple modeling using the NIST DTSA software. A series of (bulk) copper spectra have been generated and copper L α :K α ratios obtained from simple peak integrals for a range of accelerating voltage / primary electron beam landing energy ranging from 9.4 to 10.2 kV. Similar data has been experimentally collected using an Oxford Instruments ISIS EDS on the ElectroScan ESEM using a pure copper sample. The experimental data was also collected at a range of different chamber gas pressures, using water vapor as the chamber gas.

The DTSA model data and experimental measurements have been completed and indicate that the impact of small variations in the overvoltage ratio on suitably selected emissions for a particular accelerating voltage can be very high (figure 2). Both model and measured data indicate a sensitivity of ~4 ev per 0.05 change in Cu L α :K α peak integral value. This is a significant improvement on direct DHL measurements. ■

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