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30 kV STEM-SEM – The Perfect Conditions for Transmission Spectroscopy?

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Scanning Electron Microscopy (SEM) has seen continuous improvements to spatial resolution at both high, 30 kV, and low, 1 kV, accelerating voltages over the last 30 years. This has driven the demand for high spatial resolution Energy Dispersive Spectroscopy (EDS) spectral imaging to match the spatial resolution specification of the microscope. Improvements in EDS technology, transitioning from SiLi detectors to Silicon Drift Detectors (SDD), have resulted in greater X-ray detection efficiency. However, spatial resolution in spectral images is predominantly governed by the interaction volume, a measure of electron penetration and X-ray generation, and not the detection efficiency.

Under normal 20 kV SEM high-resolution imaging conditions, due to the large interaction volume, the spatial resolution of spectral images will be orders of magnitude larger than the size of the electron spot (Figure 1a). Reducing the accelerating voltage will reduce the interaction volume and improve the spatial resolution of the spectral images. Sub 10 nm EDS spatial resolution has been achieved using windowless EDS detection at short working distances and with an accelerating voltage of 1.5 kV [1].

Alternatively, the spatial resolution of spectral images can be improved by utilising electron transparent samples. The lateral broadening of the interaction volume is proportional to the depth of penetration, by reducing sample thickness to < 100 nm we can greatly improve the spatial resolution at high accelerating voltages. Monte-Carlo simulations of the interaction volume calculated using NISTMonte [2], indicate that > 70 % of the X-rays generated at 30 kV originate from within the first 10 nm of sample (Figure 1c). Therefore, high-resolution spectral images of electron transparent samples at 30 kV can achieve spatial resolutions similar to both high-resolution 200 kV STEM and high-resolution 1.5 kV bulk SEM [3].

We have investigated the spectral imaging spatial resolutions achievable for a range of accelerating voltages between 1 and 200 kV. Results highlight that STEM-SEM of electron transparent samples can be utilised to acquire the best spatial resolution spectrum images from an SEM. Furthermore, we have modelled the theoretical X-ray emission as a function of solid angle, beam current and sample thickness, identifying that, for a 100 nm thick sample, 30 kV is a more efficient accelerating voltage for X-ray emission than 200 kV. A result of the interaction probability, with high energy electrons more likely to travel through the sample without any inelastic interaction. These calculations indicate that under the same acquisition settings, a 200 kV TEM would require twice the solid angle to achieve equivalent X-ray count rates to a 30 kV STEM-SEM (Figure 2).

A bulk steel sample, known to include sub 100 nm V-rich inclusions, was measured using a Thermo Scientific Scios FIB-SEM equipped with an Oxford Instruments X-Max^N EDS detector. Initial experiments were performed using 20 kV SEM, at this accelerating voltage, the V spatial resolution was found to be ≈ 100 nm, with no smaller inclusions visible against the background EDS signal (Figure 1b).

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Using a FIB-SEM, a sample was extracted from the bulk and thinned to approximately 100 nm before being imaged using STEM-SEM at 30 kV (Figure 1d). This resulted in a large spatial resolution improvement, bringing the characterisation capabilities of V-rich inclusions from 100 nm to 20 nm. The workflow to analyse an electron transparent sample using STEM-SEM was found to require less time and expertise than that of the workflow to analysing an electron transparent sample in a TEM. The ease of use of STEM-SEM combined with the resolution improvements and enhanced X-ray collection efficiency make STEM-SEM an extremely powerful tool for high spatial resolution spectral imaging. We feel proper utilisation of STEM-SEM could reduce the TEM workload for high magnification EDS analysis.

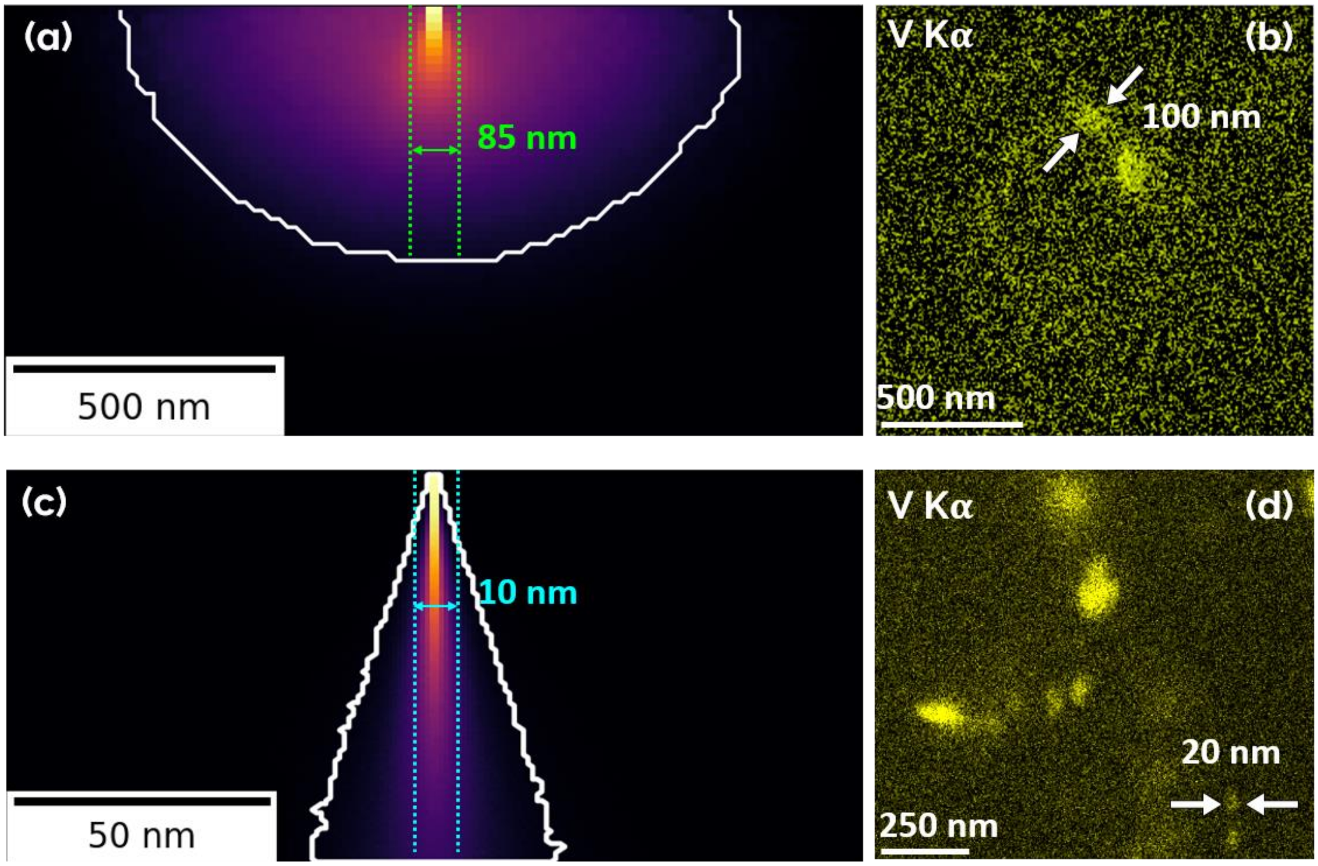

Figure 1. Monte Carlo simulations of V Kα X-ray generation for a bulk SEM sample analysed at 20kV, a), and a 100 nm thick STEM-SEM sample analysed at 30kV, c). The effective spatial resolution has been highlighted on each simulation. Experimental V Kα X-ray maps call attention to the differences in spatial resolution. Data was acquired from a steel sample at b) 20 kV (SEM) and d) 30 kV (STEM-SEM) with 30 minutes acquisition time.

Accelerating Voltage	Fe Kα Counts/(s.nA.msr)	Fe Lα Counts/(s.nA.msr)
2 kV	0	701.65
30 kV	3167.54	917.54
200 kV	1352.73	246.78

Figure 2. Theoretical X-ray detection of Fe Kα and Lα for at 2, 30 and 200 kV accelerating voltage.

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