doi:10.1017/S1431927620020681
Microsc. Microanal. 26 (Suppl 2), 2020

High Resolution Low kV SEM EDS – Breaking Convention with Low Working Distance EDS

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Energy dispersive X-ray spectroscopy (EDS) is a well-established technique analysing characteristic X-rays generated as a by-product from the electron beam interaction within a scanning electron microscope (SEM). The conventional approach to EDS combines large area silicon drift detectors (SDD) with high working distances, typically 10 – 15 mm, to maximise the number of X-rays being detected. The working distance is derived from the microscope set up, specifically the geometry of the detector, and is required to perform accurate quantitative analysis [1]. This results in a loss of surface sensitivity due to the large interaction, generated by high energy electrons penetrating deep into a material.

Over the last twenty years there has been a drive to improve the resolution of SEMs. This has resulted in FEG SEMs that are now capable of achieving sub 1 nm resolution. The resolution of a SEM is predominantly governed by the size of the electron spot/probe and the interaction volume of the electrons with a material [2]. Therefore, to achieve the best spatial resolution you must minimise the working distance, accelerating voltage and beam current of the SEM. In these HR imaging conditions little or no EDS signal is detected from a conventional EDS configuration, if the working distance is increased to boost the EDS signal, then the image is lost as the resolution of the microscope degrades.

HR EDS analysis has been achieved by optimising a detector to match the same microscope conditions that are required for HR imaging. The two operational changes required for HR SEM are the reduction in working distance and the reduction in accelerating voltage. The reduction in the working distance can be accounted for, by modifying the detector geometry. As the X-ray yield has been decreased by the reduction in accelerating voltage, it is essential that the SDD sensor is positioned as close to the sample as possible. This is achieved by reducing the length of the electron trap within the detector and by implementing a racetrack shaped SDD. This greatly improves the solid angle of the system, resulting in a greater count rate at low accelerating voltages. The requirement to use lower accelerating voltages results in a much greater emphasis on the sub 5 kV region of the EDS spectrum. This region of the EDS is suppressed as low energy X-rays are more strongly absorbed by the polymer window within conventional EDS detectors, therefore windowless EDS detection is necessary. This new approach is generalised through a graphical interpretation comparing a conventional EDS configuration and a HR EDS configuration in Figure 1.

In this study we have compared the results obtained from a conventional windowed EDS configuration with an Oxford Instruments Ultim Extreme HR EDS configuration[3], an example of the data achieved is shown in Figure 2. When used to image at high magnification, the conventional EDS configuration, Figure 2a, results in a low resolution SEM image and Cr K EDS map. This is due to the broad electron beam and large interaction volume, generated by the high working distance and large accelerating voltage, resulting in diffuse Cr nanoparticle resolution with very little edge definition. In contrast, the quality of the HR SEM image, in Figure 2.b), propagates through with resolution improvements observed in the Cr L map. Here the reduced interaction volume and spot size enhance the definition of the Cr nano particles. The improvement in low energy X-ray detection is apparent in the acquired N maps, with the windowless detector easily resolving sub 35 nm N nanoparticles. This is also observed in the Cr maps, in which the Cr L line (573 eV), is easily resolved. In this study, we will also discuss the utilisation of L, M and N energy lines and the challenges of low voltage quantification.

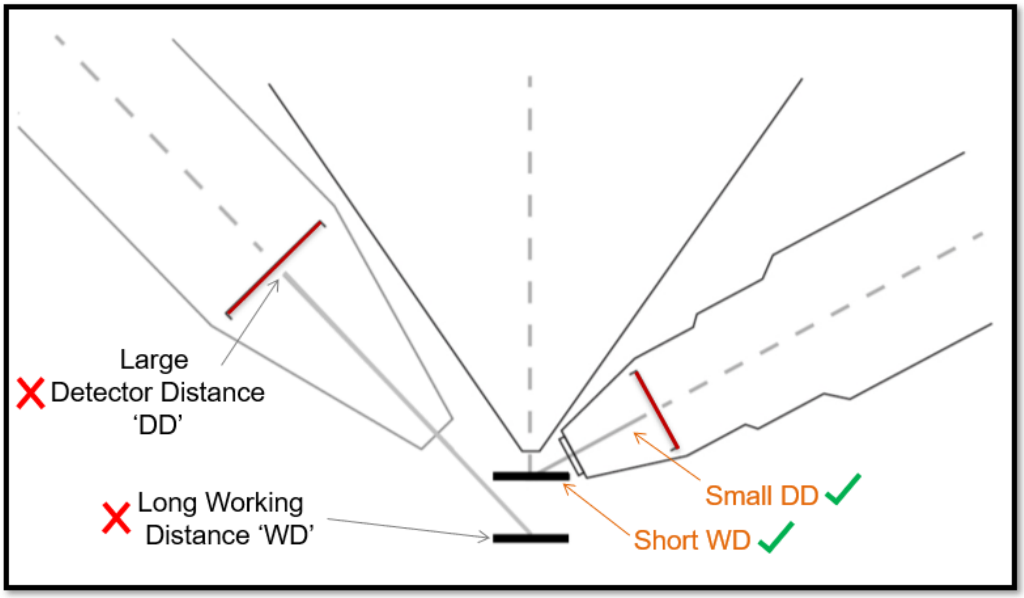
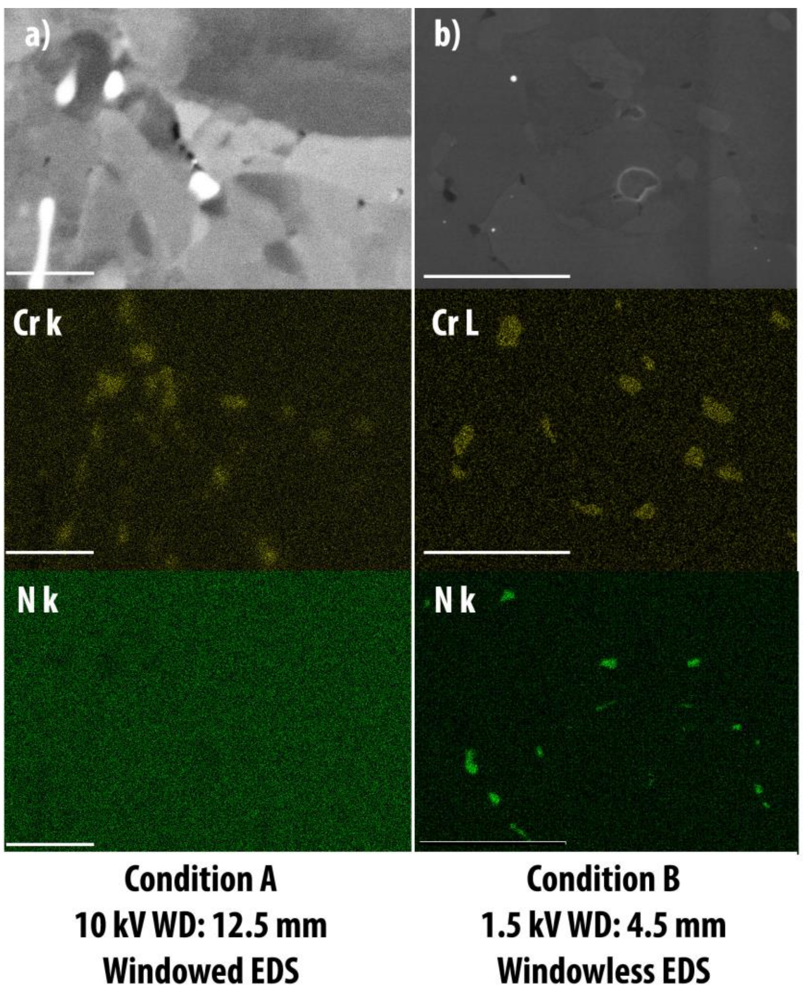


Figure 1. A graphical interpretation of a conventional EDS configuration compared to a high-resolution EDS configuration.



https://doi.org/10.1017/S1431927620020681 Published online by Cambridge University Press

Figure 2. Comparison of conventional EDS, a), to HR EDS, b), through the analysis of precipitates in steel. The effect of reduced accelerating voltage and working distance are observed in the resolution improvements of the electron image and Cr map. The removal of the polymer window in b) results in enhanced low energy X-ray detection with sub 50 nm N particles clearly resolved. Scale bar = 1 μm

References
[1] P. Statham X-Ray Spectrometry 5.1 (1976): 16-28.
[2] D Joy and J. Pawley. Ultramicroscopy 47.1-3 (1992): 80-100.
[3] S Burgess et al. Microscopy and Analysis 6 (2013): S8-S13.

https://doi.org/10.1017/S1431927620020681 Published online by Cambridge University Press