Low voltage imaging and X-ray microanalysis in the SEM: challenges and opportunities

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Abstract. Low voltage imaging, X-ray microanalysis and X-ray mapping has become very important for the investigation of nanomaterials and their surfaces. This is especially true for low voltage imaging of non-conductive samples and beam sensitive samples. Operating the SEM at lower accelerating voltage allows for greater surface sensitivity, the ability to minimize charging effects, nanometre scale lateral X-ray spatial resolution and nanoscale X-ray depth sensitivity. Determining the correct accelerating voltage for imaging in a SEM is dependent on the instrument’s operating performance at low voltage, the material being viewed, and other factors that limit effectiveness of low voltage microanalysis, which will be discussed in this paper.

1. Introduction

There have been significant advances in the field of scanning electron microscope (SEM), X-ray microanalysis, X-ray mapping (XRM), gunshot residue (GSR) analysis and electron back-scattered diffraction (EBSD) over the last five to ten years. The development of a stable field emission gun (Schottky emitter) and a new generation of secondary electron (SE) and backscattered electron (BSE) detectors has made high resolution SEM imaging on the nanometre level possible (figure 1) [1-8]. With the development of silicon drift detectors (SDD) this has allowed for greater X-ray collection efficiencies with greater sensitivity for X-rays in the low energy part of the spectrum with the SEM operating at lower accelerating voltages.

For decades, high energy accelerating voltages, ranging from 15kV to 30kV, have been used by microscopists for imaging and analysing samples because of the sub-nanometre image resolution and the ability to excite the well-known K-lines in the atomic spectra for the identification of the elements and quantification of their concentrations.

Conventional SEMs that use high accelerating voltages and conventional “lower” or “below-lens” detectors require a thin conductive coating on the sample to avoid charging artefacts if they are non-conductive. When the electron beam impinges on an uncoated non-conductive sample, a charge can build up inside the specimen, which can make imaging unstable, difficult and even in extreme cases, impossible [1]. The development of the variable pressure and/or low-vacuum SEM (VPSEM or LVSEM) and environmental SEM (ESEM) [9-11] allows samples to be imaged in their natural state.
without any sample modification or sample coating. These systems utilize gas ionisation (charge compensation) in the SEM chamber, eliminating charging artefacts usually seen with imaging non-conductive samples under high vacuum conditions [9-11].

More recently, low voltage imaging has become popular and easier to perform due to the development of a new generation of field emission SEM’s (FEGSEM or FESEM). These have better electron optic systems and better performing (new generation) SE and BSE detectors. Operating the SEM at lower accelerating voltages allows for increased surface information (greater surface sensitivity), sometimes reduced beam damage (depending on the sample), and also gives the ability to minimize charging effects (figure 2) [1-4, 6-8]. By operating at low accelerating voltages, the problem of charging can be minimized and even eliminated [1, 12-18] as can be seen in figure 3, which is an image of a non-conductive polymer that is beam sensitive. The polymer was deposited on a silicon wafer, which can also assist in carrying away any charge.

Low voltage operation is very important in the observation of non-conductive charging samples, imaging of beam sensitive samples and obtaining greater information of the sample surface whether it is conductive or non-conductive [1]. Low voltage imaging generally refers to the application of SEM techniques at beam energies below 5 kV. On the other hand, very low voltage imaging or sometimes referred to as ultra-low voltage imaging refers to beam energies below 1 kV [1, 3, 12, 13].

This paper will initially cover low voltage imaging in the SEM and how to determine the ideal accelerating voltage (beam energy) for particular specimen types. Furthermore, the paper will also cover the challenges and opportunities for low voltage microanalysis with particular emphasis on low voltage X-ray mapping and post-processing X-ray maps.

2. Low voltage imaging: electron-specimen interactions
In the SEM instrument, lowering the beam energy of the electrons has a profound effect on the interactions that occur within the sample. The elastic and inelastic scattering processes critically depend on the energy of the incident electrons. The mean-free-paths of these elastic and inelastic processes rapidly decrease with decreasing beam energy [18]. At low voltages, both the electron range and the interaction volume are significantly reduced [12, 18]. Figure 4 shows the Monte Carlo simulations of electron scattering in carbon, iron and gold at 20 kV and 5 kV beam energies. At low
Figure 2. a-c) Multilayer carbon coating. Imaged at 2 kV, using an “in-lens” SE detector. Sample was freshly broken and imaged in cross-section immediately. Image c) reveals fine structure detail that would easily be missed operating at higher accelerating voltages; d) Image of “Opal Eye” sample taken at 50 eV on a Zeiss FEGSEM revealing fine surface detail; e) Gold on carbon image taken at 100 eV; and f) Image of material surface taken at 1 kV revealing a thin layer of contamination. Image micrometre bar lengths represent a) 100 nm, b) 100 nm, c) 20 nm, d) 200 nm, e) 100 nm, and f) 200 nm, respectively.

Figure 3. Polymeric material deposited on silicon wafer. Sample imaged at 0.5 kV, using an “in-lens” SE detector.
energies the electron range (Re) falls from the micrometre level at 15 kV to the nanometre range at 500 V beam energies. For example, the typical electron range of 1 kV beam with carbon is 7 nm (figure 4b). This rapid decrease in electron range causes a large characteristic change in the secondary electron yield (δ) for all specimens, which rises from 0.1 at 30 keV to 1 at 1 - 4 keV, and even greater than 1 for some materials [12-14].

| Typical electron ranges (1 kV) |
|-------------------------------|
| Carbon (C)                    | 33 nm  |
| Aluminium (Al)               | 25 nm  |
| Copper (Cu)                   | 7 nm   |
| Silver (Ag)                   | 6 nm   |

Figure 4. a) Monte Carlo simulations [19]. Electron beam diffusion varies with accelerating voltage and specimen density. All are scaled to the same depth and width. b) Table of typical electron ranges for different materials at 1 kV accelerating voltage [19].

Below ~ 1 kV, the secondary electron yield falls as there is less energy available to produce secondary electrons [12-14]. Figure 5a shows the effect on the electron yield as a function of the accelerating voltage [20]. As can be seen in figure 5a, there exists an electron beam energy (E1 and E2) where the secondary yield is unity. These E1 and E2 points are called the crossover energies where the E2 point is a stable operating condition and typically exists between 0.5 kV and 4 kV [14, 19, 21]. The E2 point is dependent on the instrument and material [12, 18, 19].

There are many sources of electron signals in the SEM, not all of which come from the sample, as can be seen in figure 6. Some of these include secondary electrons generated by [19, 21]:
1. The primary beam (SE1).
2. By backscattered electrons (SE2).
3. By backscattered electrons interaction with chamber walls as well as other parts of the SEM (SE3).
   So, with the SEM operating at low voltage:
   1. The spatial resolution improves for all emission signals SE1, SE2, BSE, X-ray, cathodoluminescence (CL) as the interaction volume shrinks as 1/E1.6;
   2. The signal to noise ratio (S/N) improves as secondary yield rapidly increases;
   3. The information carried by all emissive signals is related to the near-surface of the specimen; and
   4. Less background secondary electrons (SE) as SE2 signal are generated from the same volume as SE1.

A clean sample is absolutely critical for low voltage imaging [1, 19]. At low voltage conditions the specimen generally looks more solid than translucent as emitted signals only contain information about the near surface of the specimen. In addition, the reduced electron beam penetration enhances edge-effect emission but reduces the topographic contrast, as most SE signal is generated in the escape depth irrespective of the surface tilt. Therefore, low voltage SEM images appear flatter and less 3D compared to high-voltage SEM images.
Figure 5. a) Electron yield as a function of beam energy, reproduced from [19, 21].
b) Backscatter coefficient as a function of atomic number [1, 21]. c) Backscatter coefficient versus accelerating voltage [1, 21].

Figure 6. Schematic images showing the sources of electron signals in the SEM. Schematic modified from [19, 21]
With regard to the back-scatter signal, the BSE yield ($\eta$) is considered independent of the accelerating voltage above $\sim 2$ keV and is usually shown as in figure 5b [6-8]. However, below 2 keV, the situation is more complex as $\eta$ rises for low-Z materials but falls for high-Z materials (figure 5c) [1, 6-8, 22-24]. At very low voltages, a small amount of charging can significantly change the image contrast, especially if you are looking at backscattered electrons. This is also a point with using a stage bias/beam deceleration, a sharp or pointy area can emit a lot of secondary electrons and with stage bias these can be accelerated up the column and appear as backscattered electrons [6, 7]. When using stage bias and backscattered (in-lens) together, careful interpretation of the images is required.

3. What is the right accelerating voltage for imaging in a SEM

There is no one correct low voltage that can be used to image a sample surface and balance the charge [12, 13]. The correct low voltage beam energy is dependent on the instrument operating conditions and the material, and should be selected on a case-by-case basis [12, 13, 20]. Imaging of rough samples, multiphase samples, composites and insulators is not simple, as the charge dissipation may be different for the different phases present [1, 12]. As can be seen in table 1, every material has a unique $E_2$ value, which is the point where there is a charge balance (current in equal’s current out).

| Material          | $E_2$ (keV) | Material   | $E_2$ (keV) |
|-------------------|------------|------------|------------|
| Nylon             | 1.18       | PMMA       | 1.6        |
| 5%PB7/nylon       | 1.40       | NaCl       | 2.0        |
| Acetal            | 1.65       | LiF        | 1.9        |
| Polyvinyl chloride| 1.65       | Pyrex glass| 1.9        |
| Teflon            | 1.82       | GaAs       | 2.6        |
| Quartz            | 3.0        | Alumina    | 2.9 - 4.2  |

4. Low voltage imaging of non-conductors: determining $E_2$ charge balance point

With low voltage imaging in a SEM, the goal is to eliminate charging or at least to control and minimise it. The paper written by Joy and Joy [12] has an excellent section discussing and explaining the why and how of low voltage SEM imaging. According to Kirchoff’s law on conservation of charge, currents at a point must sum to zero [7, 8, 12, 13].

$$I_B = (\eta + \delta)I_B + I_{SC} + (dQ/dt) = I_{BSE} + I_{SE} + I_{SC} + (dQ/dt)$$

where $\delta$ and $\eta$ are the SE and BSE yields, $Q$ is the charge deposited in the specimen after some time ($t$), and $I_{SC}$, $I_{BSE}$, and $I_{SE}$ are the currents for the specimen, backscattered and the secondary electrons.

If the sample is a conductor, then it cannot charge and the $dQ/dt$ equals zero. However, with an insulator, $I_{SC}$ is zero and we require the sample not to charge, so $dQ/dt$ equals zero. If this condition is achieved, then a dynamic charge balance exists where:

$$I_B = I_B(\eta + \delta) \quad i.e., \quad \eta + \delta = 1$$

If $\eta + \delta = 1$, then a dynamic charge balance exists;
If $\eta + \delta < 1$, then negative charging will occur;
If $\eta + \delta > 1$, then positive charging will occur.
The variation of the \((\eta + \delta)\) yield curve is much the same for all materials with the \(E_2\) being stable and the \(E_1\) not stable [12, 13].

The method that is employed to determine the \(E_2\) point of a material with low voltage imaging has been well published [1, 12-14, 18]. A schematic of the procedure is shown in figure 7 and basically involves the following [12]:

1. Set up the microscope at a particular accelerating voltage say ~ 2 kV, focus surface and allow image to stabilize.
2. Set the SEM magnification to ~ 300 x at a fast rate (say TV rate) (figure 7a).
3. Increase the magnification to ~ 1,000 x as quickly as possible and wait for around 5 seconds (figure 7b).
4. Then drop the magnification back to the original magnification (~ 300 x) and look at the scan square that is visible in the centre of the screen (figure 7c).

If the area scanned (scan square) in the lower magnification image appears brighter than the background (figure 7f), then the sample is charging negative and the beam energy is greater than the \(E_2\) point. On the other hand, if the area scanned (scan square) in the lower magnification image appears darker than the background (figure 7d), then the sample is charging positive and either the beam energy is less than the \(E_2\) point (figure 7e) or there could be contamination on the surface. Be aware that sample preparation and cleanliness is critical with any low voltage imaging [1, 12, 19].

When performing the above procedure, charge can build up in the sample more easily than it can discharge, which will cause fluctuating electric fields and disturb the electron beam as well as the secondary electrons [12-15]. Implanting charge into the sample must be avoided, and if it does occur, then 1) move to another area of the sample, 2) tilt the sample [12], or better still 3) vent the chamber and then re-pump the chamber as this aids in removal of charge from the sample. To avoid this, prepare two samples (same samples) in the microscope at the same time and also place conductive paint all the way around the sample edges. Use the first sample to determine the low voltage conditions and then go to the second sample to perform imaging. This minimizes the chance of charge storage.

Figure 7. Schematic representation of procedure for determining the \(E_2\) point for materials. Reproduced from [12].
On a final note, low voltage imaging and reducing charge not only involves adjusting the accelerating voltage to a lower value but also involves lowering the probe current and changing the scan speed to acquire the image [1, 12].

5. Low voltage SEM instrumentation developments

Besides the development and improvement of field emission SEM’s, there has also been a continual improvement and development of a new generation of detectors. These detectors have many generic names including “in-lens” electron detector, “upper” electron detector, and “through-the-lens” (TTL) detector [1, 3, 6, 7, 15, 19]. These detectors, depending on their location and design principles, collect different signals as shown in figure 6.

Another development has been the use of beam deceleration techniques that can provide excellent image resolution at very low landing energies [1, 19]. This is done by maintaining a higher accelerating voltage in the gun and column (and therefore reducing chromatic aberrations and therefore increasing resolution at low voltages) and placing a negative bias on the sample [1, 6]. This firstly slows the beam down (decelerates), therefore, increasing pure surface topographic detail due to reduced beam penetration, and secondly pushes more secondary electrons off the sample improving the signal-to-noise ratio (especially at very low accelerating voltages) as well as reducing charging and contamination [1, 6].

6. Low voltage microanalysis

Traditional microanalysis using energy dispersive X-ray spectroscopy (EDS) involves using higher accelerating voltages. The accelerating voltage of the SEM determines the energy of the electron beam which consequently determines which X-rays are excited during the interaction with the material. The SEM is usually operated at accelerating voltages in the range of 15 kV to 25 kV in order to excite the well-known K-lines in the atomic spectra, however, the use of higher accelerating voltages usually results in larger interaction volumes, which consequently degrades the spatial resolution of the X-ray image and increases the analytical volume (figure 4a).

It is generally not possible to obtain high spatial resolution chemical microanalysis from bulk materials when using high accelerating voltages [18]. However, qualitative X-ray maps can highlight small features on the nanometre level, especially if averaging and filtering techniques are employed to resolve the nano-features.

With the development and advancement of SDD’s, the sensitivity for X-rays in the low energy part of the spectrum has substantially improved. Manufacturers are now producing SDD’s with such large detector areas, that greater X-ray collection efficiencies are possible. This, and high count rate throughput, is now allowing the possibility of operating the SEM at much lower accelerating voltages and subsequently reducing the interaction volume of the electron beam with the material as well as achieving higher spatial resolution information (figure 4). Furthermore, SDD’s can now be purchased with varying windows [30] and also windowless systems [25-29] allowing greater sensitivity of the lower energy X-rays. Figure 8 shows the EDS spectrum from an Amptek detector with two different window materials (C1 and C2). These windows are made from 90 nm and 40 nm silicon nitride (Si3N4) with a very thin aluminium coating to extend the low energy response [26]. The C2 window has excellent transmission for low Z elements with ten times more carbon X-rays (43.9 %) and 29 % transmission of Li [26].

Just becoming commercially available are new soft X-ray spectroscopy (SXES) systems, with a measurable energy range of 50 to 210 eV, an order of magnitude better energy resolution (with 0.3 eV resolution reported), and detection limits for light elements of less than 100 ppm [30].

By lowering the accelerating voltage a major improvement in the spatial resolution and image quality occurs. This forces the selection of X-ray lines with low excitation energy such as L- and M-family lines between 0 - 4 kV, rather than using K- and L-lines between 0 - 10 kV [1, 18, 25]. The
Figure 8. Intensity versus energy spectra from an Amptek Fast123 detector, collected at E₀ = 8kV. a) C1 window of a contaminated Cu-Al grid, b) C2 window of a contaminated Cu-Al grid (same area), and c) Spectra (from C2 window detector) of a white cast iron hard-facing material that has NbC particles present. X-axis represents energy (kV).

measurement of low energy L-line X-rays is complicated by low fluorescence yield, an increase in X-ray absorption, numerous X-ray interferences from other elements within the sample, and less accurately determined mass absorption coefficients [25-29]. The overlaps of K-line X-rays from light elements with L- and M-line X-rays from heavier elements limit the low voltage analysis and mapping capabilities of conventional microanalysis systems [35].

The use of transition element L-lines generally results in poor accuracy as well as poor sensitivity [21, 31, 32]. Furthermore, operating at low voltages has limitations due to the large quantification errors existing on the lower energy X-ray lines [34]. Extensive work is still required on fundamental parameters [31], algorithms and mass absorption coefficients before any accurate quantitative analysis can be performed [32-34], especially with the many overlaps that exist in the low voltage regime of multi-element materials. With multielement materials that have overlaps, at best low voltage microanalysis is only good for qualitative analysis.

For quantitative analysis, standards based analysis is the better way to go [31]. As can be seen in table 2, standardless analysis has many more parameters to be determined for accurate analysis compared to standards based analysis. All low voltage analysis will give better results if using standards based analysis [31], however there are still errors. Wavelength-dispersive X-ray spectroscopy (WDS) also has problems in the light element region due to high order overlaps.

### Table 2. Factors affecting standards and standardless analysis.

| Standards          | Standardless                        |
|--------------------|-------------------------------------|
| Common to both:    |                                    |
| Gain and Zero, kV  | Resolution                          |
| Take Off Angle,    | Fano Factor                         |
| Overlaps, Beam     | Dead Layer                          |
| Current and Count  | Detector Thickness                  |
| Rate               |                                     |
| Dead Time          | Gold Layer                          |
| Live Time          | Window Thickness                    |
| Intensity Factors  | Sum Peak Calculations               |
| Standards          | Escape Peaks                        |
|                    | Carbon Coating Thickness            |
|                    | Incomplete Charge Collection        |
|                    | Other absorbing layers              |
7. Advantages of low voltage X-ray mapping

Characterisation is an essential aspect of materials research and of quality control in materials production. Characterisation of materials frequently involves the determination of point-to-point variation in composition, structure and microstructure, so a variety of imaging and analysis techniques come into play [36, 37]. Understanding the distribution of elements and phases in structures is critical to optimising the performance of all materials. Subtle changes in composition have been studied for many years to determine the properties of many materials and aid in the production of these materials [35-37]. This is quite often done by performing microanalysis and also XRM [35, 40-45]. XRM is an extremely useful problem solving tool which involves the collection of characteristic X-rays as a function of the position of the scanning electron beam on the specimen [36-39]. XRM provides an image related to the distribution and relative abundance of elements within a given specimen and makes XRM particularly useful for: i) identifying the location of individual elements, and ii) mapping the spatial distribution of specific elements and chemical phases within a sample (figure 9) [40]. This is especially true with low voltage X-ray mapping, due to the higher spatial resolution [1], allowing nanolevel features being detected [46, 47]. However, the problems with multi-element materials are that overlaps can exist, especially with the L-lines. As can be seen in figure 8c, the EDS spectra shows the hard facing material of NbC particles in white cast iron, has numerous L-lines overlapping each other. The subsequent generation of X-ray maps collected at 8 kV using K-lines (figure 9b-e) and L-lines (figure 9f, h-j) shows major errors for Mn L-line. Furthermore, the quantification results using the L-lines are totally incorrect. This can minimized by more precise calibration of the detector parameters (due to non-linearities in the detector electronics) to enable better correction of the overlaps in the low energy range.

![X-ray maps taken at 8 kV of hard-facing material with NbC particles in white cast iron.](image)

**Figure 9.** X-ray maps taken at 8 kV of hard-facing material with NbC particles in white cast iron. a) BSE image, b) Carbon K-line, c) Chromium K-line, d) Manganese K-line, e) Iron K-line, f) Niobium L-line, g) Carbon K-line, h) Chromium L-line, i) Manganese L-line, and j) Iron L-line. Notice the Mn X-ray map for the K-line and L-line X-ray maps are totally different, due to the linearity and overlap problems occurring at the L-lines for Cr, Mn, and Fe.

If utmost care is taken with collecting and processing X-rays maps, especially at low voltages, then further post processing (data manipulation) techniques may be performed [39], even if it is only used as a first approximation to determine a qualitative result. These post-processing techniques have been extensively covered by the authors in previous publications and include [36-45]:

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**References:**

[1] (Year), [Title], [Source].

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**Details:**

- **Type of Document:** Conference paper
- **Conference:** EMAS 2015 Workshop
- **Series:** IOP Publishing
- **Volume:** 109
- **Issue:** 1
- **Year:** 2016
- **DOI:** 10.1088/1757-899X/109/1/012019
1. Generating scatter diagrams and rotational scatter diagrams [39, 40] for locating phases.
2. Pseudo colouring (figure 10) for locating phases. The colours of the elements can be rotated, which can often reveal further information (and other phases) in the material. Indeed, the rotating of colours between elements at low voltage X-ray mapping has aided revealing fine precipitates and small boundary interfaces that would be otherwise missed.
3. Filtering to make phase selection easier, to produce cleaner images and aid in trace element location as well as aiding in finding small precipitates.
4. Elemental ratio maps. This is a powerful technique that helps to reveal subtle chemical variations in a sample. Ratio maps can also be created from different combinations of elements and from the different background regions near elements of the spectra [38, 39].
5. Totals mapping. The totals at each point of the map can be calculated and mapped (when standards based analysis is performed). These total maps give an idea of the quality of the analysis and also make it easy to determine problems such as missing elements and interaction volume effects [39, 43, 45].

![Figure 10](image)

**Figure 10.** Pseudo coloured X-ray map of Cr (red), Nb (green) and Fe (blue). Pseudo colouring is a simple method for determining elemental associations and is also called “Primary Colour Imaging”. This technique is where three elemental maps have been assigned the colours red, green and blue. Because the colours red, green and blue form specific colours when combined on a computer graphics monitor, elemental associations are clearly shown in a single “pseudo” colour image. Note, that the original X-ray maps used prior to colouring are normalized to produce good grayscale images.

8. Future improvements
The future SEM will be able to operate in many different modes, whether it is VP SEM, ESEM, low voltage and even high voltage SEM. The new Schottky emitter field emission gun microscopes are capable of 1 nm resolution or better at 15 kV and can operate in VP mode and the emitters can produce greater beam currents (> 100 nA) that allow these instruments to have excellent analytical capabilities, which is of great importance for EDS, WDS, XRM and EBSD analysis. By incorporating different column designs, larger apertures, greater beam currents can be obtained, with minimal resolution loss. Obtain better and faster EBSD maps than with conventional tungsten instrument with XRM and GSR analysis completed a lot faster. With the new imaging detectors, new SDD detectors and new EBSD detectors working at closer working distances, higher resolution images and maps can be obtained no matter what mode the SEM operates in. There is currently a whole generation of detectors available that have better efficiency and can collect many different type of signals. Some of these detectors can operate under various conditions. New SEMs should be able to simultaneous collect images from all detectors BSE, SE and gaseous secondary (GSE), specimen current imaging, in-lens BSE/SE and X-rays.

Besides instruments capable of operating in a number of modes with different detectors, there are still a lot of advances for the instrument to be developed. Systems that have energy filtering, spectroscopy imaging, multi-detector systems (SDD, WDS, low energy detectors, CL) as well as correction systems on SEM columns need to be developed [48-50]. More importantly, systems have to be developed that allow for self-correcting microscopes (automated system calibration) [48-50].
9. Conclusion
There have been significant advances in the field of scanning electron microscopy, especially with electron column design, stable emitters, different SE and BSE detectors and different X-ray detectors with and without windows.

Low voltage imaging and X-ray analysis in the SEM have many challenges ahead but, there are many more opportunities and applications being developed. Low voltage microanalysis has a distinct disadvantage in that fewer X-ray lines are available and the ones that do exist quite often overlap with lines from other elements. However, if a careful approach is taken, some excellent results can be obtained on a nanometre level, even if it is only a qualitative approach.

Acknowledgements
The authors would like to thank the many that have contributed information and advice towards this paper. These include Ron Rasch (UQ), Jo Michaels (Sandia National Laboratories), Brenden Griffin (UWA), Matthew Phillips (UTS), Natasha Erdman (JEOL USA), Heiner Jaksch (Zeiss), David Bell (Harvard University), David Joy (Oak Ridge National Laboratory), Raynald Gauvin (Mc Gill), Dale Newbury (NIST), Ken Mason (Eastern Analytical), Darren Attard (Zeiss), FEI, Zeiss and JEOL. The authors wish to thank the Advanced Materials Characterisation Facility (AMC Facility) at UWS.

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