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Understanding your Material Better – Low Voltage Imaging, Analysis and X-ray Mapping - Applications and Points to Consider

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The application of low voltage imaging, X-ray microanalysis and X-ray mapping (XRM) has become very important for the investigation of materials and their surfaces [1-6]. Operating the SEM at lower accelerating voltage allows for greater surface sensitivity, the ability to minimize charging effects, nanometer scale lateral X-ray spatial resolution and nanoscale X-ray depth sensitivity [1]. 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 [1-4].

Currently there is a high demand from researchers and industry for determining surface variations on the nanoscale, location and compositions of micro to nano precipitates, chemically determine boundaries and interface compositions (especially variations in composition at the interface boundary), finding and imaging contaminations as well as determining their composition. Figure 1 shows the surface morphological change with imaging at lower accelerating voltages. These fine surface details imaged at 1kV revealed important information about the particle surface as well as oxides present, which are implicated in premature material failure of subsequent sintered final products.

There are many things needed to be considered when performing low voltage imaging and microanalysis. 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 can appear flatter and less 3D compared to high voltage SEM images [7], although surface detail is increased (Figure 1).

With regard to microanalysis; by lowering the accelerating voltage, this forces the selection of X-ray lines with low excitation energy such as L and M family lines between 0-4kV, rather than using the traditional K and L lines between 0-20kV [1, 3]. The 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, less well known L subshell emission line ratios (eg. L_l , L_n and L_α) and similarly for the M emissions and less accurately determined mass absorption coefficients [1, 3]. 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 [3, 8]. Extensive work is still required on fundamental parameters, algorithms, L and M line ratios and mass absorption coefficients before any accurate quantitative analysis can be performed [3, 8-10], especially with the low voltage regime of multi-element materials [3]. These multi-element materials that have many overlaps, are only good for qualitative analysis. Recent work by Matthew et al [11], showed that low voltage quantification of a Uranium metal sample with a 15nm surface oxide layer produces anomalously high totals even after correction of the oxide layer (Figure 2). Poorly constrained corrections parameters were proposed as contributing factors [11].

There is no one correct accelerating voltage for imaging in a SEM and for microanalysis. The correct low voltage beam energy is dependent on the instrument operating conditions, type of material, elements present and should be selected on a case-by-case basis [12]. There are also many other factors that limit effectiveness of low voltage microanalysis. Further to this, different strategies have to be considered that also include considering microanalysis calibration and utilizing different standards. All of these points will be covered in this presentation.

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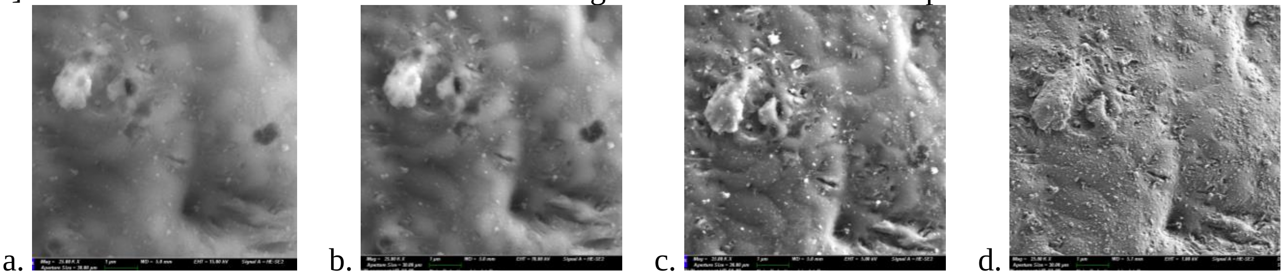

Figure 1. Metal particles imaged at various accelerating voltages [13]. Note that surface detail is increased at low kV (high kV to Low kV, left to right). a) 15kV, b) 10kV, c) 5kV and d) 1kV.

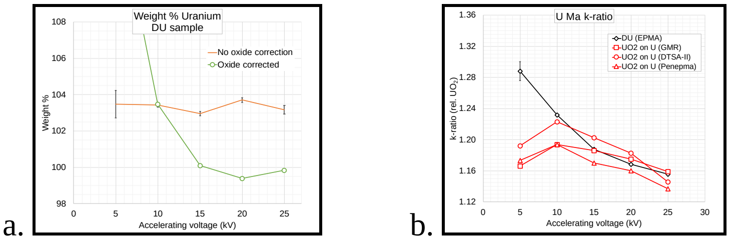

Figure 2. a) Change in composition totals as a function of accelerating voltage for UO on U and b) k-ratio modelled with different programs – measured continues to increase; all models show decrease [11]. Images reproduced from reference [11] with permission.