Elemental distributions determined by X ray Mapping in the extended variable pressure SEM

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Introduction

Energy dispersive X ray analysis (EDS) within the scanning electron microscope (SEM) has become a widely used and technologically very important analytical technique. The collection and analysis of X rays generated from a single point on the sample surface fluoresced by the primary electron beam allows the elemental composition to be identified. Additionally, by collecting data from an array of points across a sample surface, elemental distributions can be generated that indicate chemical composition across the sample. In both these examples, the assumption has been made that the detected X rays are generated from a single point source on the sample directly under the primary electron beam. The interpretation of any X ray map is more difficult if this assumption is not true.

Modern SEM systems, in which a gas is introduced into the chamber to compensate the charge accumulation on the surface of non-conductive samples, have allowed the analysis of insulating materials not previously amenable to analysis in the SEM. So successful has this approach been that the majority of SEM systems are now purchased with this facility.

However, the introduction of gas into the path of the primary electron beam can lead to scattering of the primary electrons from the optical axis of the microscope. These deflected electrons form a "skirt" around the primary beam. When these "skirt" electrons strike the specimen X-rays are generated that are not associated with the intended point on the specimen. This contribution of X-rays from material away from the intended analysis point can significantly complicate any interpretation of either qualitative or quantitative results as the underlying assumption is no longer valid.

Instrumentation

An EVO 50XVP SEM fitted with an X-ray microanalysis system was used to investigate the effect of beam scatter on the resolution of elemental X ray maps. The design of the EVO's sharp 80° final lens, EDS port, and sleeved beam technology allowed a wide range of operating conditions to be used to investigate the extent of electron scattering. The EVO's sleeved beam technology provides a good vacuum environment for the primary electron beam almost to the specimen itself. This design ensures that the Beam Gas Path Lengths (-the distance the electron beam travels through the low pressure gas within the chamber – BGPL) to be reduced to only 2mm. Without the beam sleeve in place the BGPL can be arranged to be as large as 28mm whilst still keeping the EDS detector aligned with the sample surface. This range of both BGPL, and chamber pressures, has allowed a systematic study of the influence of these two principle parameters on electron scattering and its influence on the resulting X-ray maps.

Figure 1.
Photograph of the sharp 80° final lens used on the EVO SEM. The EDS detector is shown at a take off angle of 35° and a working distance of only 8.5 mm. The Everhart-Thornley SE detector is visible behind the EDS detector.

Test Specimen

A two-phase aluminium nickel alloy, comprising a primarily aluminium matrix with a second phase of nickel aluminide precipitates. A conductive sample was used so that a control analysis was possible in high vacuum so that any beam scatter could be eliminated and a standard established for comparison with subsequent measurements made where beam scattering occurred. The very fine particle size (~1μm) led to the use of a low energy beam (10keV) and a low probe current (500pA) to ensure high resolution X ray mapping.

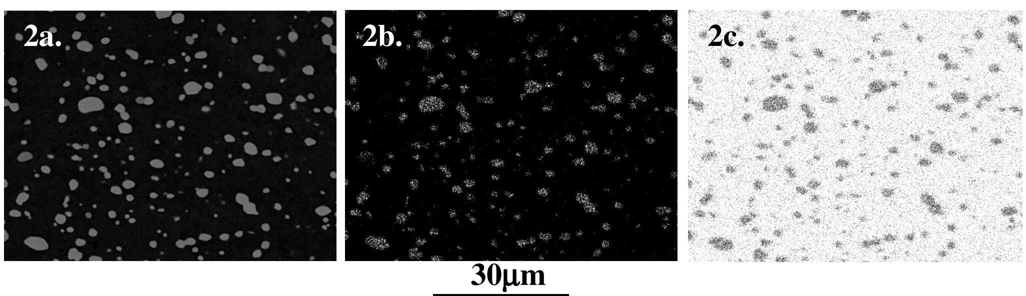

Figure 2.
Figure 2a shows the backscattered electron image. The nickel (Figure 2b.) and aluminium (Figure 2c.) X-ray maps are also shown taken under high vacuum using a 10 keV, 500 pA electron beam and a 8.5 mm working distance.

Figure 2a shows the backscattered image, which clearly shows the high atomic number nickel containing second phase material. This is clearly comparable to the nickel X-ray map where again the nickel containing particles are easily resolved. The particles are also visible in the aluminium X ray map as dark regions due to the lower aluminium content and hence lower aluminium X ray emission. Thus it can be clearly seen that in high vacuum, with minimal electron scattering, that X ray mapping gives elemental distributions that reflect the known microstucture of the material with a high degree of accuracy.

The influence of gas pressure and BGPL on X-ray map quality

Figure 3 shows the influence of three choices of BGPLs and three choices of gas pressures on Ni X-ray map quality. It is clear that the best images are obtained with the lowest gas pressures and the shortest BGPLs. With a BGPL of 28 mm, it is almost impossible to obtain useful data even at the lowest gas pressures. It is important to note that 15 Pa is often not a sufficient gas pressure to charge compensate many materials.

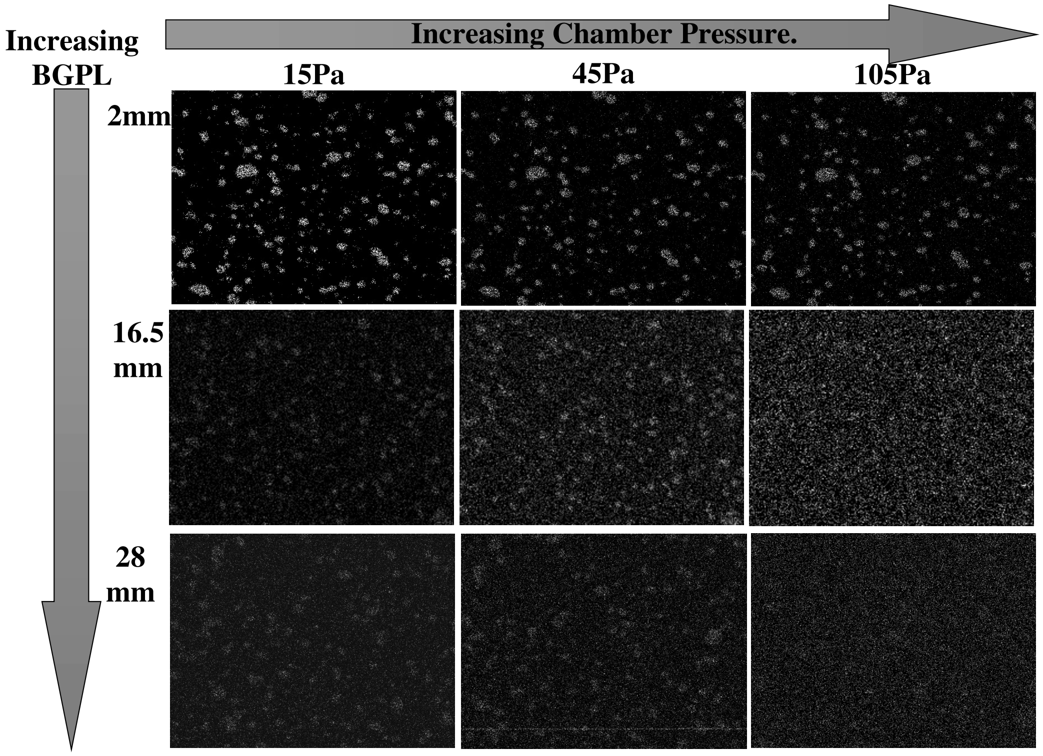

Figure 3.
Nickel X ray maps showing the effect of BGPLs and chamber pressures on the resolution of the nickel rich particles.

A minimum chamber pressure is usually dictated by the need to charge compensate the specimen. The only method to reduce the beam scatter is then to minimise the BGPL. With the EVO series proprietary sleeved beam technology, the BGPL can be reduced to only 2 mm.

The benefit of this sleeved beam technology and its associated 2mm BGPL is clear from Figure 3 where quality Xray maps are obtained even at 100 Pa chamber pressure.

Conclusions

It has been shown that Xray maps are influenced by both the pressure in the specimen chamber and by the beam gas path length (BGPL). The most accurate X-ray maps are obtained if the BGPL is reduced to 2 mm. If the BGPL is as large as 28 mm, as it is in some commercial microscopes, then quality Xray mapping of insulators is not possible.

In many cases, a minimum chamber pressure is required to provide stable imagingand so the only parameter available to minimise this electron scattering is to reduce the BGPL the distance over which the electrons interact with the chamber gas.

Key Points

- High Electron Scattering is an inescapable consequence of the electron beam passing through a gas.
- Electron scattering leads to a larger spot on the specimen surface and a defocusing of the image.
- High chamber pressures lead to electron scattering
- Long beam gas path lengths lead to electron scattering
- Both effects lead to the creation of a beam skirt
- Sleeved beam technology permits a BGPL of only 2 mm
- A BGPL of only 2 mm provides the best quality X-ray distributions

Links

Free “Casino” Monte Carlo electron scattering package

http://www.gel.usherb.ca/casino/