X-ray Mapping in a Variable Pressure SEM

Application Note

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 generated 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 for charge accumulation on the surface of non-conductive samples, has permitted the analysis of insulating materials. So successful has this approach been that the majority of SEM systems are now purchased with this capacity.

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. These “skirt” electrons generate X-rays that are not associated with the intended point of analysis on the specimen. These X-rays can significantly complicate qualitative or quantitative interpretation.

Instrumentation

A ZEISS **EVO**® MA SEM fitted with an X-ray micro-analysis system was used to investigate the effect of beam scatter on the spatial resolution of elemental X-ray maps. The design of the **EVO**®’s sharp 80° final lens, EDS port, and BeamSleeve® technology allowed a wide range of operating conditions to be used to investigate the extent of electron scattering. The **EVO**®’s BeamSleeve® 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 (i.e. the distance the electron beam travels through the low pressure gas within the chamber – BGPL) can be reduced to only 2 mm. Without the BeamSleeve® in place the BGPL can be arranged to be as large as 28 mm 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 parameters on electron scattering and its influence on the resulting X-ray maps.

Test Specimen

A conductive sample, a multi-phase aluminium silicon alloy, comprising a primarily aluminium matrix with a second phase of silicon was used. Analysis in high vacuum (no beam scatter) establishes a standard for

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Fig. 2: Photograph of the sharp 80° final lens used on the EVO® MA and LS 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.

comparison with subsequent measurements made where beam scattering occurred. The fine particle size led to the use of a beam energy of 15 kV and a probe current of 200 pA to ensure high resolution X-ray mapping. Figure 3a shows the backscattered image, which clearly shows the higher atomic number silicon containing second phase material. This is clearly comparable to the silicon X-ray map where again the silicon containing phase 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, X-ray mapping gives elemental distributions that reflect the known microstructure of the material with a high degree of accuracy.

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

Figure 4 shows the influence of three choices of BGPLs and three choices of gas pressures on Si 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 10 Pa is often not sufficient for charge compensation in many materials.

A minimum chamber pressure is usually dictated by the need for charge compensation. The only method to reduce the beam scatter is then to minimise the BGPL. With the **EVO®** series proprietary BeamSleeve®, the BGPL can be reduced to only 2 mm.

The benefit of this BeamSleeve® technology is clear from Figure 4 where quality X-ray maps are obtained even at 100 Pa chamber pressure.

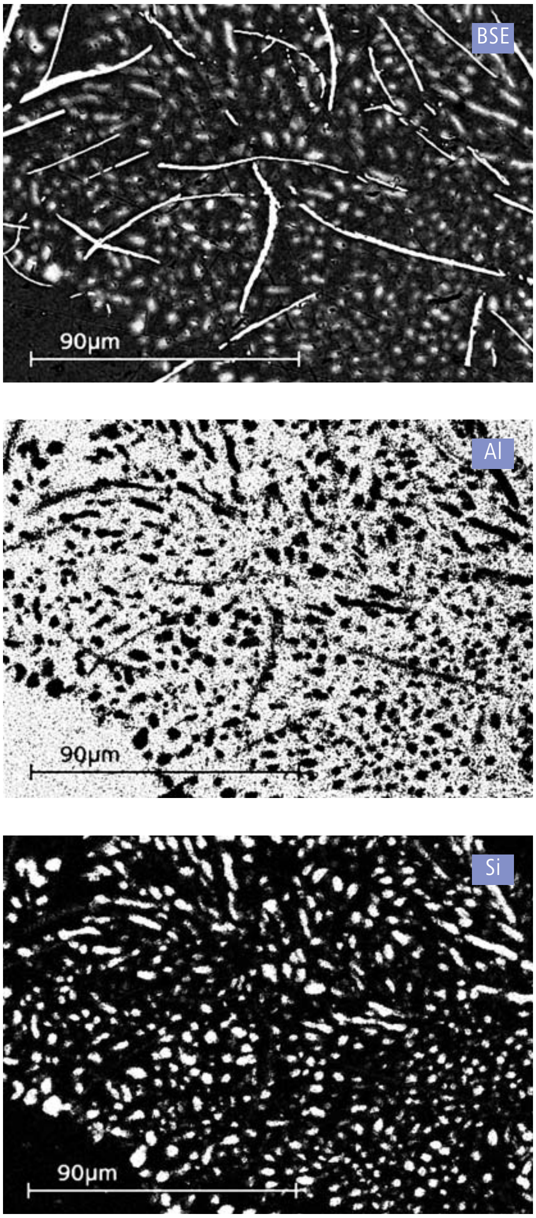

Fig. 3a, b, c: Figure 3a shows the backscattered electron image. The aluminium (Figure 3b.) and silicon (Figure 3c.) X-ray maps are also shown taken under high vacuum using a 15 kV, 200 pA electron beam and a 8.5 mm working distance.

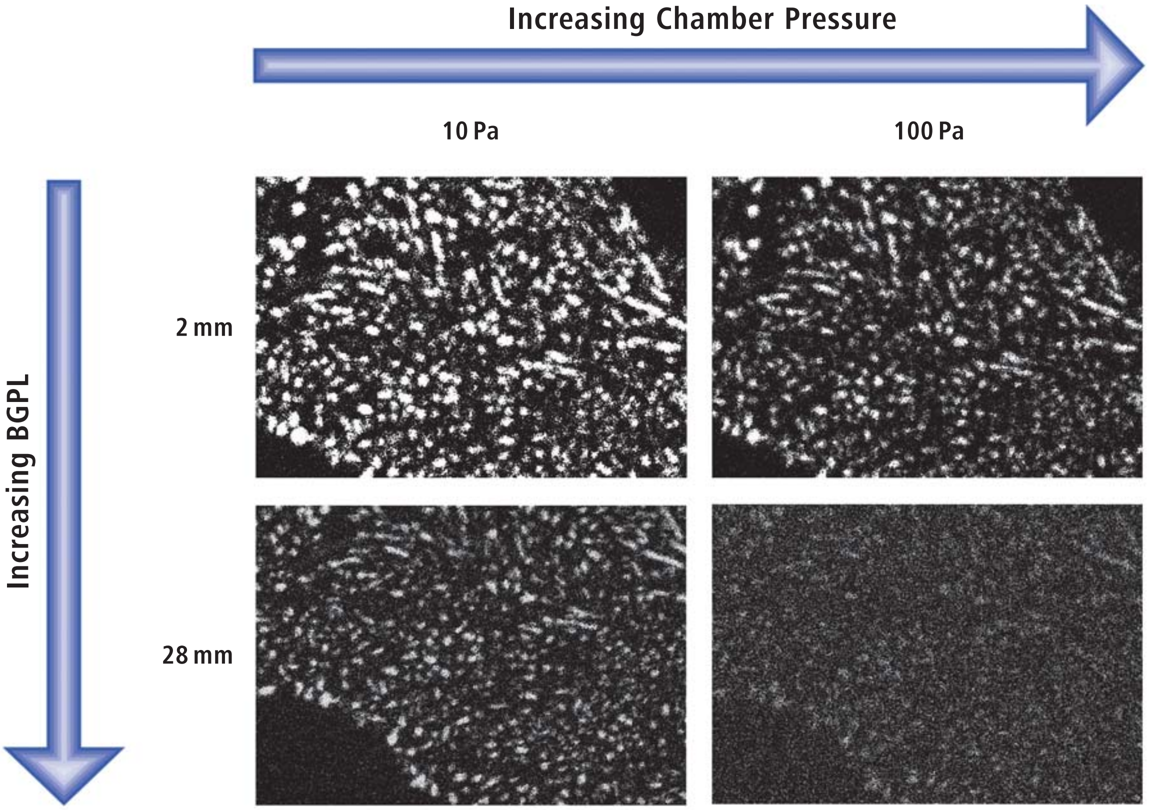

Fig. 4: Silicon X-ray maps (100µm field of view) showing the effect of BGPLs and chamber pressures on the resolution of the silicon rich particles.

Conclusions

It has been shown that X-ray 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 larger than 28 mm, as it is in some commercial microscopes, then quality X-ray mapping of insulators is not possible.

- Electron scattering is an inescapable consequence of the electron beam passing through a gas.
- Electron scattering leads to the formation of a beam skirt.
- Higher chamber pressures lead to more electron scattering.
- Longer beam gas path lengths lead to more electron scattering.
- The BeamSleeve® technology permits a BGPL of only 2 mm and the most accurate X-ray distributions.

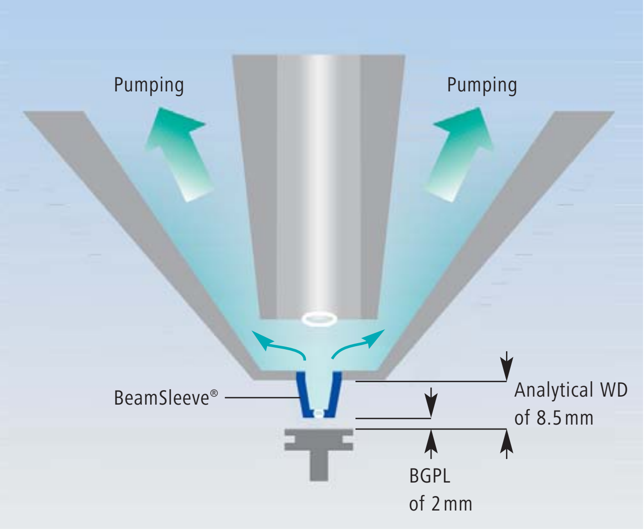

Fig. 5: BeamSleeve® attached to the pole piece of the ***EVO***® *LS and MA SEM, reducing the BGPL to only 2mm at the analytical working distance of 8.5 mm.*

Useful Links

Free "Casino" Monte Carlo electron scattering package.

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

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