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The Effect of the Charge Compensating Gas and Beam Gas Path Length on X-ray Analysis in the Variable Pressure SEM

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The scanning electron microscope (SEM) is widely used, in combination with x-ray analysis by energy dispersive spectroscopy (EDS), for imaging and microanalysis of non-conducting materials. Sample charging effects are minimised by introducing gas into the variable pressure SEM chamber.

As the primary electron beam interacts with the introduced gas, a fraction of the electrons are scattered to form a "beam skirt" [1]. The scattered electrons generate x-rays away from the focussed beam adversely affecting the spatial resolution and accuracy of x-ray analysis [2]. Lowering the chamber pressure and reducing the beam gas path length (BGPL) [3-4] is known to improve x-ray maps and spot analysis. The aim of this work was to compare the relative influence of water vapour and air on the accuracy of data obtained by EDS analysis. A further aim of this work is to experimentally check a widely held assumption that a low pressure of 10 Pa in combination with conventional BGPLs is sufficient to avoid misleading analysis.

Figure 1 presents experimental results that indicate that selecting water vapour instead of air reduces primary electron beam scattering by approximately 40%. The fraction of electrons scattered were estimated by the ratio of Mo x-ray signal with the electron beam on, and off, a 3 mm diameter molybdenum Faraday cup aperture.

Figure 2 reveals that a chamber pressure of 10 Pa in combination with long BGPL does lead to inaccurate quantitative analysis particularly of low concentration elements. However, EDS data, equivalent to those obtained in a high vacuum SEM, have been obtained at 10 Pa by reducing the BGPL to 2 mm. The BGPL was reduced to 2 mm by using a pressure limiting aperture (BeamSleeve^{TM}) with an EVO$^{®}$ SEM (Figure 2; left).

In conclusion, the most accurate EDS data are obtained by combining a short BGPL with water vapour as the charge compensating gas.

References

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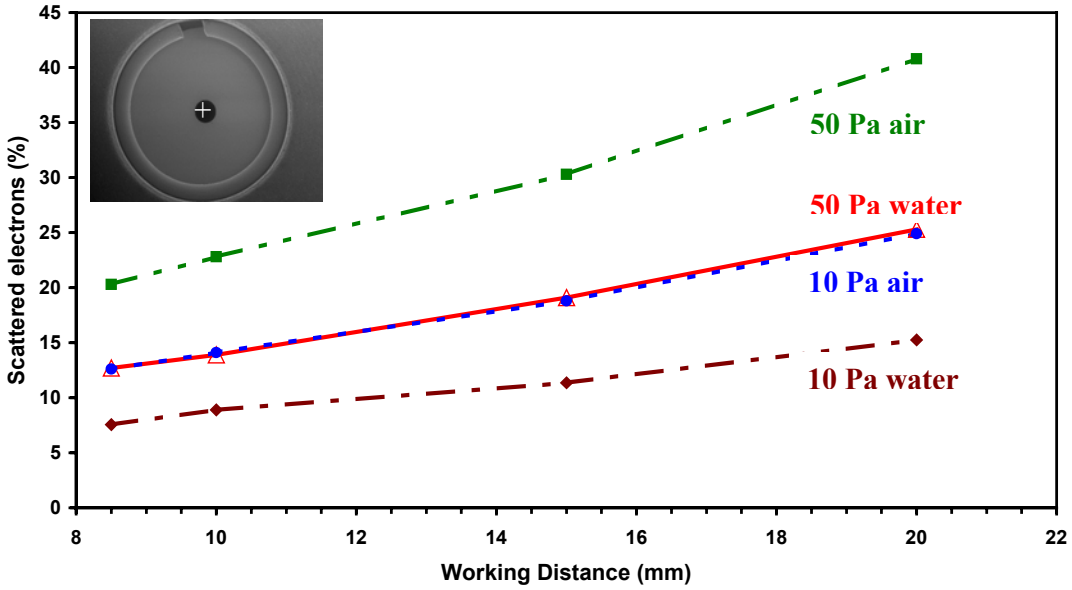

Fig 1. The fraction of electrons scattered by the gas as a function of working distance and pressure in water vapour and air.

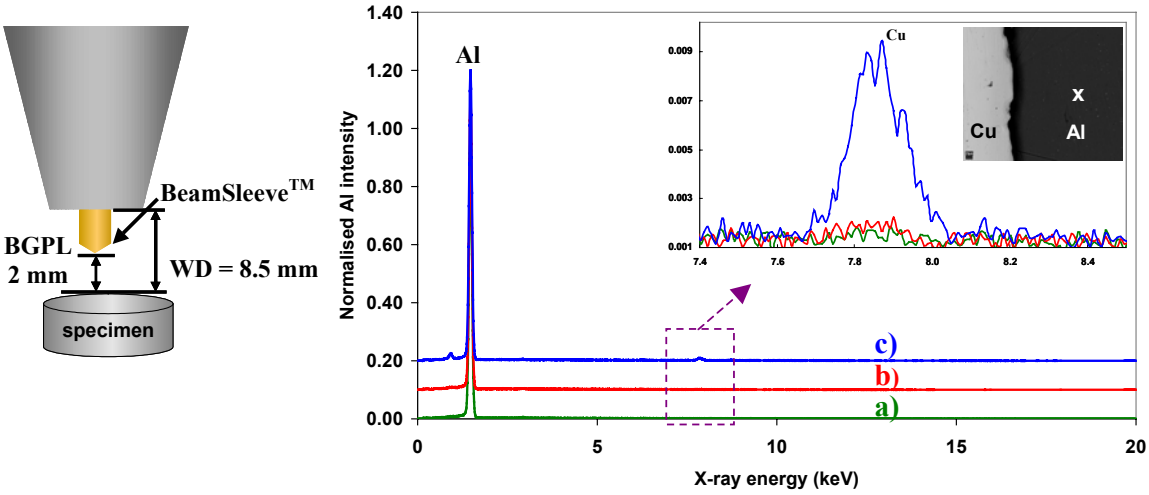

Fig 2 Left: BeamSleeve™ attached to the pole piece of the SEM, reducing the BGPL to 2 mm at the analytical working distance of 8.5 mm.
Fig 2 Right: The backscattered electron image shows the specimen, Cu stripe (bright region) on top of Al surface (dark grey), at 20 keV and 8.5 mm working distance.
The graphic shows x-ray spectrum of Al measured 150 μm from the Cu edge (shown by a cross on the image) at: a) High Vacuum , b) 10 Pa air and 2 mm BGPL, c) 10 Pa air and 16 mm BGPL. The inset shows the intensity of Cu (Kα) peak from the indicated area, magnified for clarity.

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