X-ray Analysis in a Variable Pressure SEM

Application Note

Introduction

The Scanning Electron Microscope (SEM) is widely used to provide imaging and analysis of non-conductors and wet specimens in combination with Energy Dispersive X-ray spectroscopy (EDS). Sample charging effects can be eliminated by introducing air, at a few Pa into the SEM chamber. In a high vacuum SEM-EDS combination, the X-rays generated by the primary electron beam are unambiguously associated with the position of the beam on the specimen. However, in a variable pressure (VP) SEM the primary electron beam interacts with gas introduced into the chamber. Electrons are scattered from the primary beam axis to form a “beam skirt”. The Beam Gas Path Length (BGPL) is the distance the electron beam travels through the gas in the specimen chamber. Higher pressures and longer BGPL values increase the significance of the beam skirt. As the contribution from the electrons in the beam skirt increases, the spatial resolution and accuracy of the EDS analysis is compromised [1]. Reliable analyses can only be achieved by minimising the chamber pressure or the BGPL. It is conventionally assumed that reducing the chamber pressure to 10 Pa is sufficient to avoid misleading quantitative analysis. This note explores this assumption.

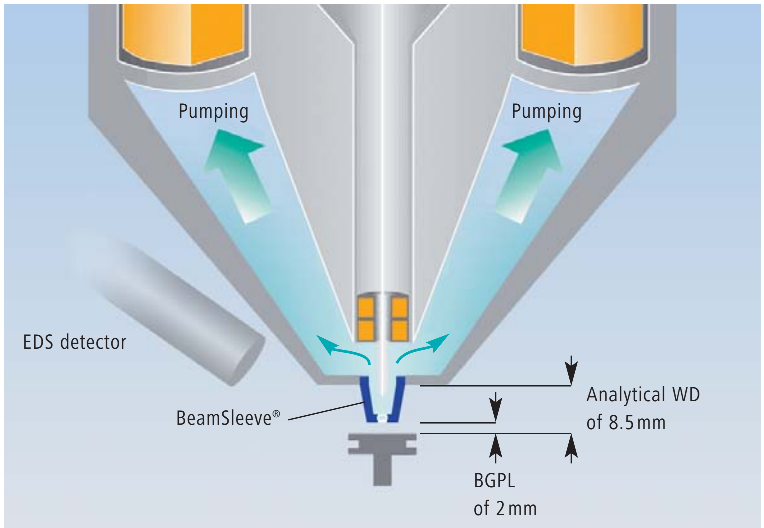

Fig. 1: BeamSleeve® attached to the pole piece of the EVO® LS and MA SEM, reducing the BGPL to only 2 mm at the analytical working distance of 8.5 mm.

Instrumentation

A ZEISS EVO® MA 15 SEM equipped with an EDS system was used to investigate the effect of beam scattering on the spatial resolution and the accuracy of EDS analysis. In this study, an EVO® BeamSleeve® was used, which minimises the BGPL to only 2 mm. Without the BeamSleeve® in place, and at a working distance of 8.5 mm, the BGPL is 16 mm. Figure 1 shows the principle of using the BeamSleeve® when performing X-ray analysis.

Application

For a chamber pressure of 10 Pa, the scattering of 20 keV electrons has been simulated at both 2 and 16 mm BGPLs using Electron Flight Simulator™ Software [2]. At 2 mm BGPL, the beam scattering is only 0.5 % and increases dramatically at 16 mm BGPL to 4 %, as displayed in Figure 2. The specimen used in the present study comprised a pure copper strip on an aluminium surface. The EDS analysis was performed at a beam energy of 20 keV and a probe current of 700 pA. The variable

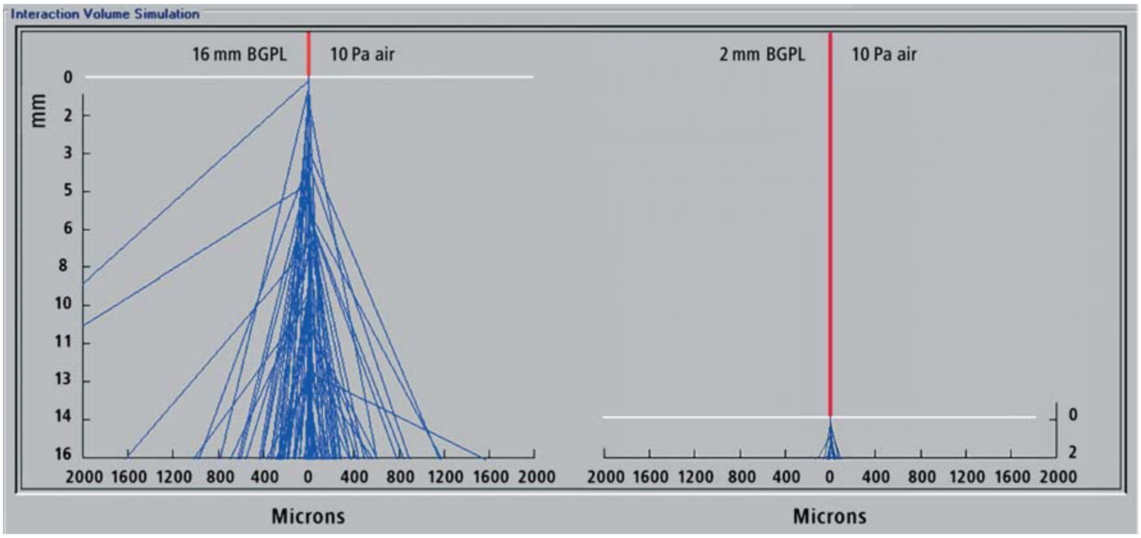

Fig. 2: Monte Carlo simulation of 20 keV electron beam trajectories at 16 mm and 2 mm BGPL at 10 Pa air. 2500 trajectories are plotted for each condition.

parameters were the BGPL and chamber pressure. The BGPL values were 2 mm and 16 mm. The chamber pressures selected were high vacuum, 10 Pa, and 80 Pa.

Figure 3 shows the backscattered image of the Cu region (bright region) adjacent to the Al surface (dark region). Line scan and point spectra were collected from the centre of the Cu region and extending onto the Al surface.

At high vacuum, the intensity of the Cu signal drops sharply as the electron beam is scanned across the Cu edge onto the Al surface, as shown in Figure 4. However, at chamber pressures of 10 Pa and 80 Pa, the Cu intensity does not drop to zero when the electron beam is scanned across the Cu edge. Even 10 Pa of chamber air pressure scatters electrons into the beam skirt. With the BeamSleeve® in place, the Cu Kα intensity drops to zero for both 10 Pa and 80 Pa chamber pressure. This is due to the fact that at a short BGPL the effect of beam scattering is negligible.

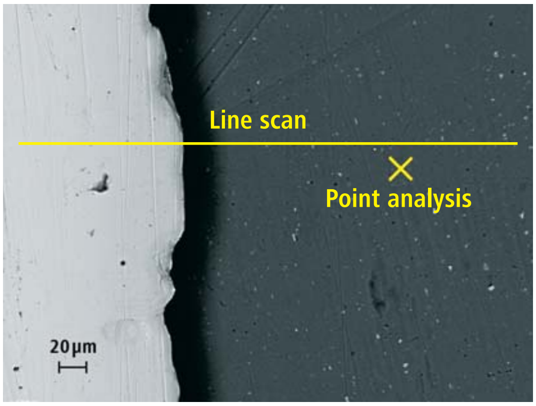

Fig. 3: Backscattered electron image of the Cu strip (bright region) on top of the Al surface (dark grey), at 20 keV, 700 pA electron beam and a 8.5 mm working distance.

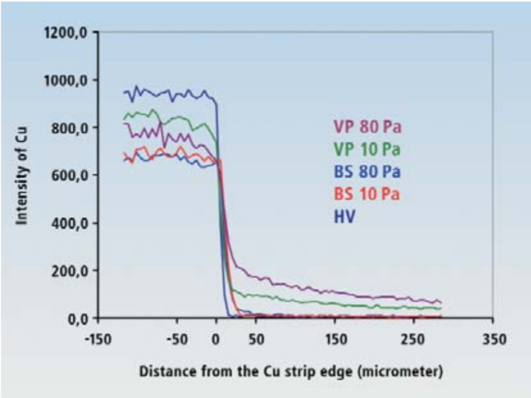

Fig. 4: The intensity of Cu as a function of distance from the Cu edge. VP = Variable Pressure, BS = BeamSleeve®, HV = High Vacuum

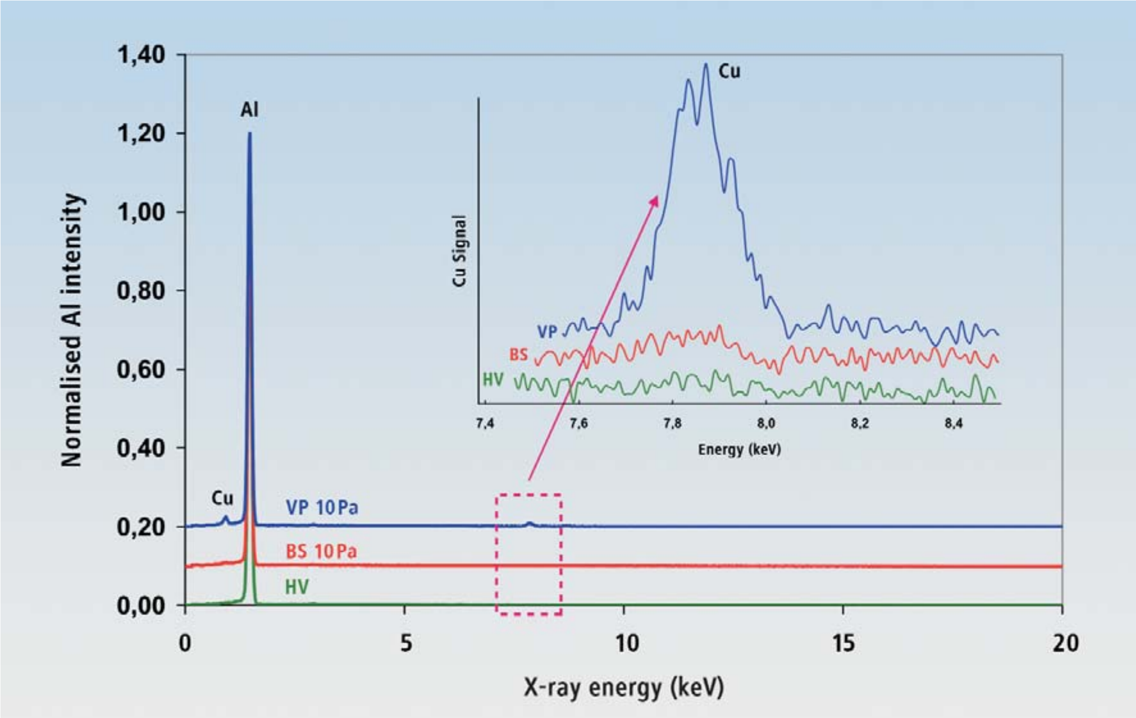

Fig. 5: The X-ray spectrum of Al measured 150 µm from the Cu edge at: a) High Vacuum (HV), b) Variable Pressure mode, at 10 Pa pressure, with the BeamSleeve® (BS), c) Variable Pressure mode, at 10 Pa pressure, without the BeamSleeve® (VP). The inset shows the intensity of the Cu peak from the indicated area, magnified for clarity.

Figure 5 shows EDS spectra collected on the Al surface 150 µm from the Cu edge. The emitted X-ray intensities (Cu Kα) at high vacuum, and 10 Pa pressure with and without BeamSleeve® are plotted as a function of X-ray energy. Without the BeamSleeve®, the spectrum obtained at 10 Pa clearly shows the Cu (Kα) peak at 8 keV. With the BeamSleeve® in place those spectra obtained at high vacuum, 10 Pa, and 80 Pa are equivalent.

Table 1 shows the percentage of Cu measured at 150 µm distance from the Cu edge. With the BeamSleeve® in place, Cu concentrations similar to the high vacuum results were obtained. Without the BeamSleeve®, erroneous Cu concentrations of 2.9 % and 4.9 % were measured at pressures of 10 Pa and 80 Pa, respectively.

Cu concentration (At %)	Vacuum mode
0.33 %	HV
0.32 %	BS 10 Pa
0.47 %	BS 80 Pa
2.90 %	VP 10 Pa
4.90 %	VP 80 Pa

Table 1: The measured Cu concentration at the point shown on Figure 3.

Conclusion

It has been shown that in low vacuum and variable pressure SEMs a chamber pressure of only 10 Pa leads to grossly inaccurate quantitative analysis of low concentration elements. The use of the BeamSleeve® in the ZEISS EVO® MA and LS SEM series provides accurate analysis of non-conductors by SEM-EDS.

References:
1) D. E. Newbury, J. Res. Natl. Inst. Stand. Technol. 107, 567 (2002)
2) Electron Flight Simulator™ Software, version 3.1 E Beta; Small World LLC

Fig. 6: Inside view of the EVO® chamber, showing the BeamSleeve® in place on the objective lens.

Global Solution Provider

The Nano Technology Systems Division of Carl Zeiss SMT provides its customers with complete solutions featuring the latest leading-edge EM technology. The company's extensive know-how, meticulously acquired over 60 years in the field of e-beam technology, has brought many pioneering innovations to the market. Our global applications and service network ensure fast, reliable and high quality support sharply focused on customer requirements. Combined with dedicated upgrade strategies, this will protect your investment for its entire lifetime. The core technology embedded in our innovative products enables us to provide solutions which add value to our customers' businesses.

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