The benefits associated with a 1 mm Beam Gas Path Length on the accuracy of X-ray analysis in the Variable Pressure SEM.

V. M. Kugler, S. J. Bean*

Carl Zeiss SMT Ltd, Cambridge, UK

Introduction

The scanning electron microscope (SEM) is extensively used to provide imaging and analysis of specimens, in combination with energy dispersive x-ray spectroscopy (EDS), in both high vacuum and variable pressure (low vacuum) modes. Variable pressure SEM is of increasing importance as the technique permits investigation of non conducting specimens without the need for metal coating.

A fraction of the primary electron beam is scattered by the gas in the chamber influencing both imaging and microanalysis [1,2,3].

Lower gas pressures and shorter beam gas path length (BGPL) values increase the unscattered fraction of the primary beam. Those electrons that are scattered electrons generate x-rays away from the intended location on the specimen.

Experimental method

The EVO® column has been designed to provide the optimum geometry for X-ray analysis at the analytical working distance of 8.5 mm with a take-off angle of 35 degrees (as shown in Figure 1). The benefit of reducing the BGPL from 16 mm to 1 mm was studied for a range of pressures (10 Pa to 2000 Pa), and with two gases (nitrogen and water vapour), in an Carl Zeiss EVO® MA15 electron microscope. An Oxford X-MAX EDS detector was used to measure x-ray signal intensities.

The SEM was fitted with one of two BeamSleeve® designs to provide either a 1 mm or 2 mm BGPL. When used in standard Variable Pressure mode, the BGPL is 16 mm. The BeamSleeve® is designed so that backscattered electrons, secondary electrons, and X-rays can be collected with maximum sensitivity from the specimen. A 20 keV 1.5 nA electron beam was employed.

The specimen was a standard 3 mm diameter Mo aperture disc with a 30 µm central aperture. In high vacuum, the Mo intensity from the carbon tape in the centre of the aperture was negligible. However, in VP a fraction of the electrons are scattered onto the surface of the Mo disc giving rise to a Mo x-ray signal (1). The Mo x-ray signal is used to estimate the fraction of electrons scattered beyond a radius of 15 µm.

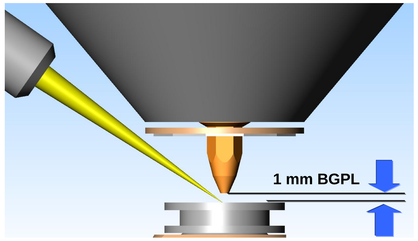

Figure 1. EVO® MA 15 geometry with 1 mm BGPL BeamSleeve® and back scatter detector

Results and Discussion

The Analytical Integrity Metric (AIM) is defined as:

$$AIM_{(30)} = \frac{I\ (unscattered)}{I\ (total)} \times 100$$

where:

I (unscattered) is the probe current not scattered by the gas by more than the radius of the central aperture.
I (total) is the total probe current.

The subscript donates the inner diameter of the Mo disc.

The x-ray signal for Mo has been used to estimate the scattered unscattered fractions. The AIM(30) parameter is plotted below for both the 1 mm BGPL and 2 mm BGPL cases. Measurements in both water vapour and air are presented.

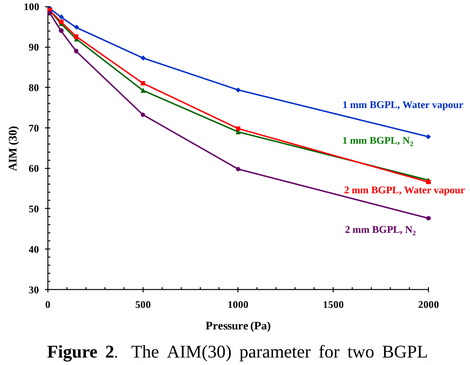

Figure 2. The AIM(30) parameter for two BGPL values, and for air and water vapour, for pressures of upto 2000 Pa.

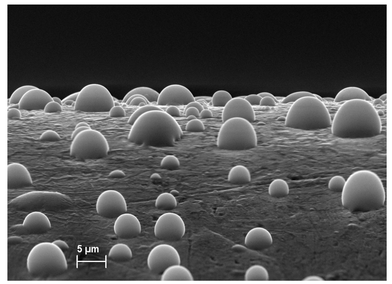

Figure 3. Image of water drops condensed onto a cooled wire. Conditions are: 25 keV, 700 Pa water vapour, and 0.1°C.

It can be seen in Figure 2, that reducing the BGPL reduces the scattered fraction from approximately 20% to 10%. This is important for environmental imaging as used to image liquid water as shown in Figure 3.

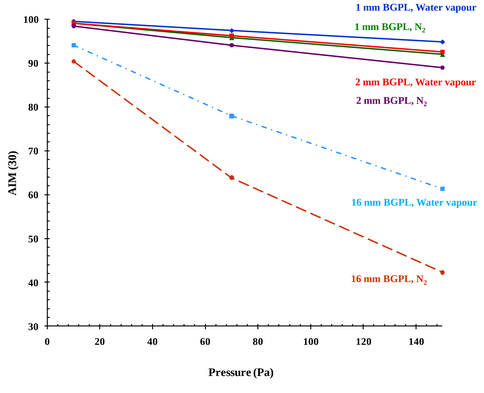

Figure 4. The AIM(30) parameter for the pressure range usually used for the analysis of non conducting materials, for two gas types, and for three BGPL values. The 16 mm BGPL is applicable to standard large field of view imaging in the EVO® microscopes.

Conclusions

- A reduction in the BGPL from 2 mm to 1 mm improves the unscattered fraction of the primary electron beam for all pressures.
- The Analytical Integrity Metric (AIM) provides a transferrable metric for the evaluation of all VP-SEMs.
- Water vapour provides lower scattering than nitrogen.

References

[1] S. Bean, V. Kugler, Microscopy and Microanalysis 2006
[2] S. Bean, V. Kugler, Microscopy and Microanalysis 2007
[3] G. D. Danilatos, Ad Electronics Electron Phys 71 (1988) 109

EVO® MA 15 Variable Pressure SEM

Booth 428