

X-ray Mapping in a Variable Pressure SEM

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.



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



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.

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 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

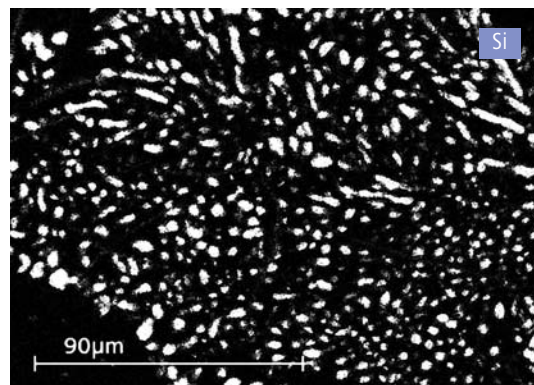
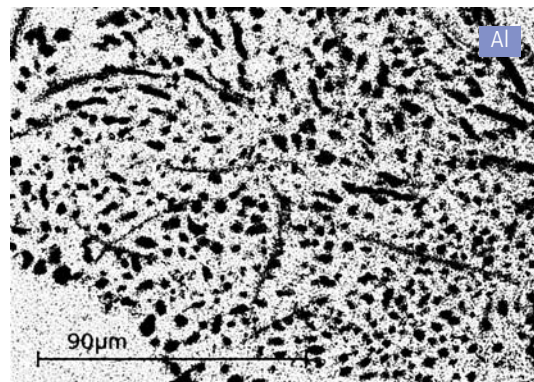
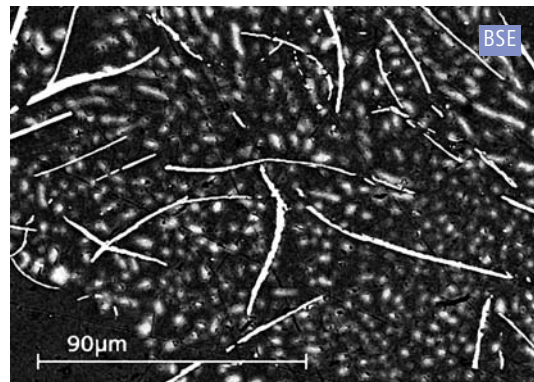


Fig. 3a, b, c: Figure 3a shows the backscattered electron image. The aluminium (Figure 3b.) and silicon (Figure 3c.) X-ray maps are also shown taken under high vacuum using a 15 kV, 200 pA electron beam and a 8.5 mm working distance.

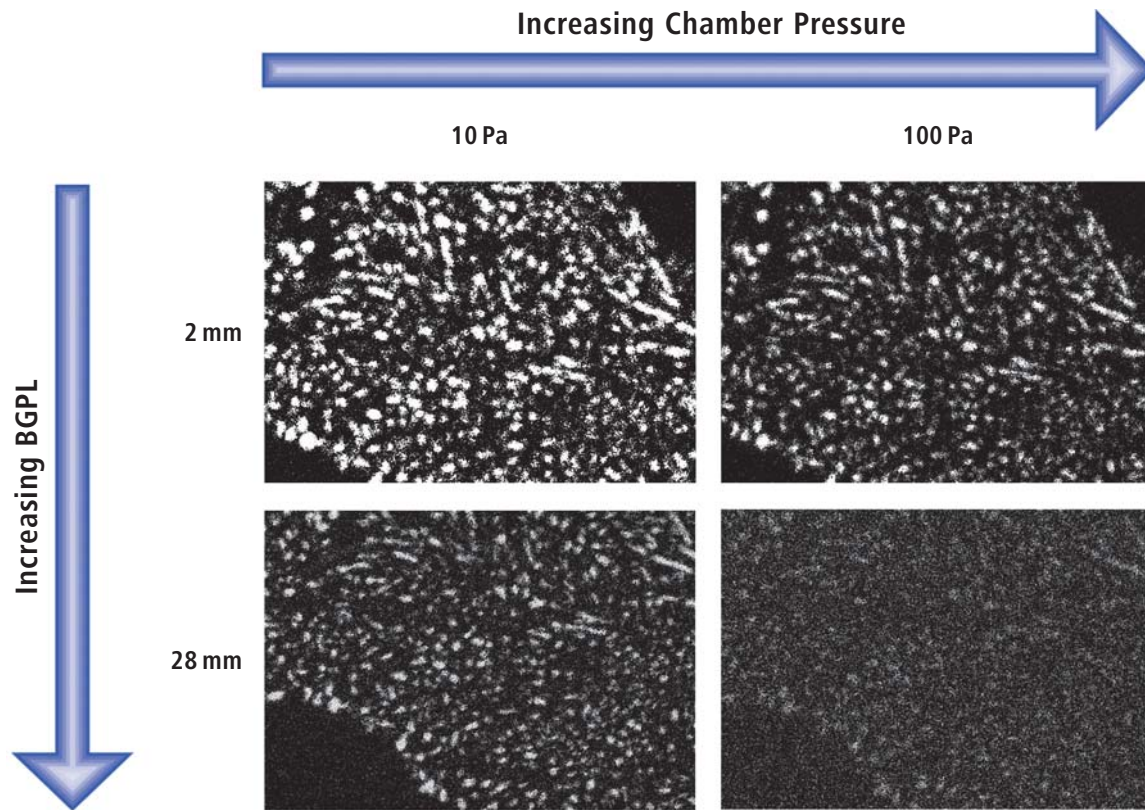


Fig. 4: Silicon X-ray maps (100 μ m field of view) showing the effect of BGPLs and chamber pressures on the resolution of the silicon rich particles.

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

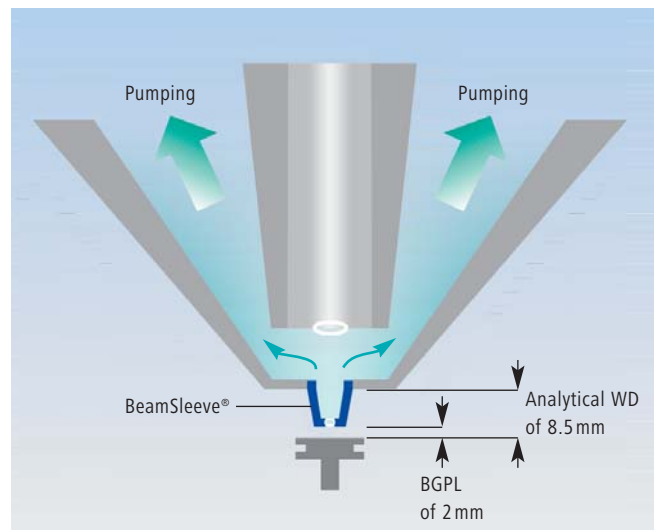


Fig. 5: BeamSleeve® attached to the pole piece of the EVO® LS and MA SEM, reducing the BGPL to only 2mm at the analytical working distance of 8.5 mm.

Useful Links

Free "Casino" Monte Carlo electron scattering package.

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



EVO® Series



EVO® Series					
Applications		Conductors	Non Conductors	Hydrated Specimens	Wet Dynamic Processes
Materials Analysis	MA	✓	✓	Future Assured	Future Assured
Life Sciences	LS	✓	✓	✓	✓



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.

Customer feedback is always welcomed and gathered by collecting valuable information at trade shows, in workshops, in user meetings and upon instrument installation. The Division's business services also include outstanding support from sales consultancy to technical service options even when the warranty has expired.

Carl Zeiss NTS GmbH
 A Carl Zeiss SMT AG Company
 Carl-Zeiss-Str. 56
 73447 Oberkochen, Germany
 Tel. +49 73 64/20 44 88
 Fax +49 73 64/20 43 43
 info-nts@smt.zeiss.com

Carl Zeiss SMT Ltd.
 511 Coldhams Lane
 Cambridge CB1 3JS, UK
 Tel. +44 12 23 41 41 66
 Fax +44 12 23 41 27 76
 info-uk@smt.zeiss.com

Carl Zeiss SMT Inc.
 One Corporation Way
 Peabody, MA 01960, USA
 Tel. +1 978 / 826 7909
 Fax +1 978 / 532 5696
 info-usa@smt.zeiss.com

Carl Zeiss SMT S.a.s.
 Zone d'Activité des Peupliers
 27, rue des Peupliers -
 Bâtiment A
 92000 Nanterre, France
 Tel. +33 1 41 39 92 10
 Fax +33 1 41 39 92 29
 info-fr@smt.zeiss.com

Carl Zeiss SMT Pte Ltd.
 Jean Yip Building
 50 Kaki Bukit Place #04-01
 Singapore 415926, Singapore
 Tel. +65 65 67 / 30 11
 Fax +65 65 67 / 51 31
 info.sea@smt.zeiss.com

Plus a worldwide network
 of authorised distributors

www.smt.zeiss.com/nts

