

Exploring backscattered imaging in low voltage FE-SEM

P Lewis¹, S Micklethwaite¹, J Harrington¹, M Dixon², R Brydson¹ and N Hondow¹

¹ School of Chemical & Process Engineering, University of Leeds, Leeds LS2 9JT, UK

² Hitachi High-Technologies Europe, Maidenhead SL6 8YA, UK

pmpml@leeds.ac.uk (P Lewis)

n.hondow@leeds.ac.uk (N Hondow)

Abstract. Contrast levels in backscattered SEM images were investigated, utilising stage deceleration for low voltage imaging and also electron energy filtering. Image contrast variations are explained via use of Monte Carlo simulations which can predict the optimum accelerating and filter voltages for imaging complex sample mixtures.

1. Introduction

Low voltage scanning electron microscopy (SEM) is advantageous due to both the increased surface sensitivity for imaging and the reduction of specimen charging [1]. The accelerating voltage of a SEM is normally limited to a value such as 0.5 or 1 kV whilst preserving acceptable image resolution, however lower voltages can be achieved by using approaches such as deceleration, where a negative potential applied to the sample stage allows electrons to land at much lower voltages.

In addition to secondary electron (SE) imaging, recording the backscattered electron (BSE) signal can provide useful information which is normally related to the average atomic number at the sample position [2]. However Müllerová and Frank have shown that at acceleration voltages below 2 kV the dependence between backscattered intensity and atomic number (Z) breaks down [3] and light elements more strongly backscatter electrons than heavier elements often leading to a contrast inversion in images [3]. It is also possible to enhance contrast at low acceleration voltages by selectively filtering the energy of emitted electrons thereby permitting the detection of specific energy back scattered electrons. Whilst not as common as energy filtering in a transmission electron microscope, energy filtered SEM has shown application in the imaging and analysis of semiconductors [4,5], in particular in reducing the influence of surface contamination layers in SE images [5]. In further experiments imaging a thermally-oxidized carbon steel and a Zn-55%Al coating, specimen conditions were kept constant whilst the bias voltage was varied [6]. It was found that image brightness decreased with increasing bias voltage and the brightness changed drastically around 0.5 kV for both specimens. Here we investigate the potential for low voltage backscattered imaging of complex mixtures of materials.

2. Experimental

SEM was conducted on a Hitachi SU8230 cold field emission (FE)-SEM equipped with an Oxford Instruments 80 mm² X-Max energy dispersive X-ray (EDX) detector. The Hitachi SU8230 FE-SEM is



equipped with numerous detectors allowing the simultaneous recording of various electron signals and energies (figure 1). Imaging was conducted using incident beam energies of between 2 and 0.25 kV, with energies less than 2 kV achieved via deceleration, together with EDX analysis at 20 kV.

Three samples were examined; a mixed aluminium-gold (Al-Au) sample prepared by Au coating an Al stub; a mixed particulate sample of ~25 nm ceria (CeO₂) and ~25 nm titania (TiO₂) nanoparticles made by allowing a drop of each nanoparticle dispersion in methanol to dry on an Al stub; and finally a mixture of carbon nanotubes (CNTs) and TiO₂ nanoparticles deposited on an Al stub. The Al-Au and CeO₂-TiO₂ samples were imaged using a photodiode-backscattered electron (PD-BSE) detector at beam energies of between 1.5 and 0.25 kV, achieved via deceleration of an initial 2 kV electron beam (figure 1a). The CNT-TiO₂ sample was imaged at 2 kV using the upper (figure 1 b) and top (with and without filter; figure 1c) detectors.

The Monte Carlo simulation software Casino Flight Simulator (Version 2.4.8.1) was used to calculate values of backscatter co-efficient for electrons hitting the surfaces of samples of different composition. Each simulation used the Mott cross section for interpolation with one million electrons. Simulations were run on Al and Au at acceleration voltages of 0.1 to 1.0 kV, in 0.1 kV steps, and on CeO₂ and TiO₂ at acceleration voltages of 0.1 to 1.5 kV, in 0.1 kV steps.

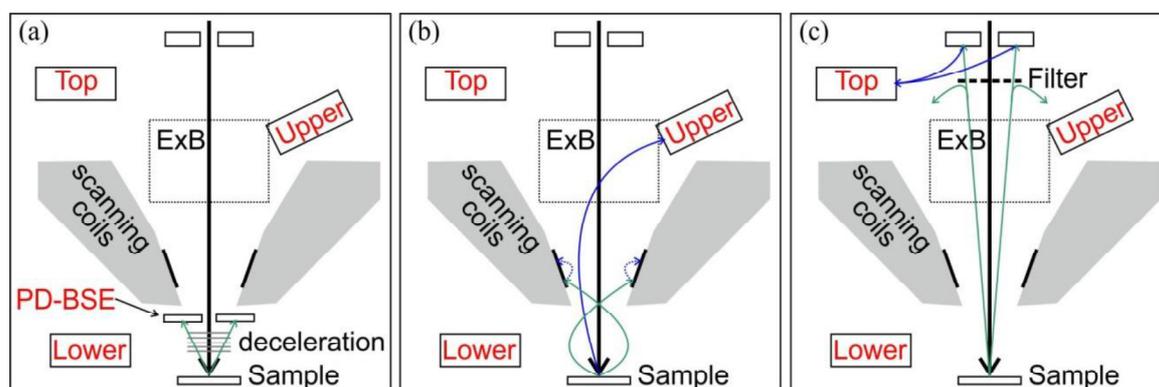


Figure 1. Schematic of the Hitachi SU8230 FE-SEM with detectors labelled in red, SE in blue and BSE in green. (a) PD-BSE detector to form BSE images. (b) Upper detector used to form SE images. (c) Top detector used to produce BSE images, the energy of which can be filtered.

3. Results and Discussion

3.1. Backscattered electron imaging at low voltages

The Hitachi SU-8230 FE-SEM used in this study can operate at a minimum accelerating voltage of 0.5 kV. To operate at lower voltages, stage deceleration is used. While this can allow imaging as low as 10 V, we have used an initial operating voltage of 2 kV which is then dropped to an effective voltage of between 1.5 and 0.25 kV by applying a potential of between 0.5 and 1.75 kV to the sample stage. The advantages of deceleration are that the electron beam retains the lower aberration and higher brightness associated with higher acceleration voltages but also has the smaller interaction volume of lower voltages, this along with the focusing effect caused by the magnetic field which is created around the sample by the deceleration voltage greatly increases the resolution of the system.

The backscattered signal can be detected using the PD-BSD and allows the formation of images that permit the discrimination between materials of different atomic weights. When examining the Al-Au sample at 1 kV (figure 2a), there is a darker level of contrast in the lower atomic number Al ($Z=13$) relative to Au ($Z=79$), as would be expected by the standard Z-contrast. This contrast is consistent at 0.5 kV (figure 2b), however at 0.25 kV (figure 2c) there is a contrast inversion, with the Au now darker than the Al. Confirmation of element location is provided by the EDX map (figure 2d), and in addition images exhibit more topographical contrast as the deceleration voltage is increased.

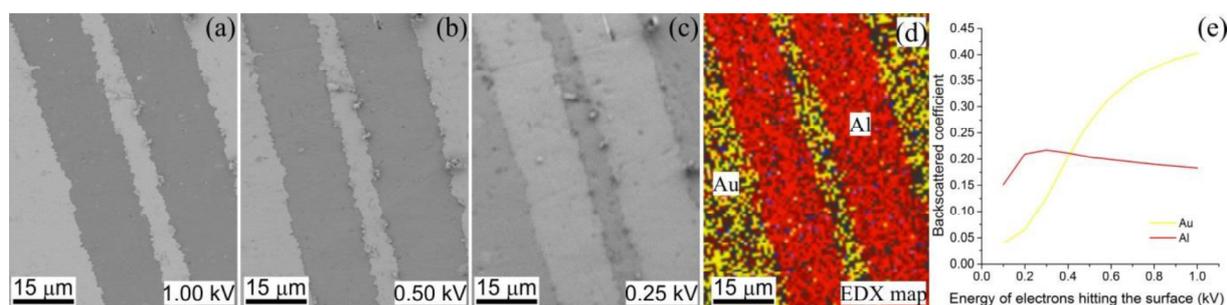


Figure 2. SEM images and analysis of the Al-Au sample. As the voltage is reduced a contrast inversion is observed between Al and Au. Backscattered electron images recorded at (a) 1.00 kV, (b) 0.50 kV and (c) 0.25 kV. (d) EDX map showing the distribution of Al and Au. (e) Plot of the predicted backscatter coefficient of Al and Au against voltage; data generated using Monte Carlo modelling. The intersection point, and therefore theoretical contrast inversion point, is at 0.41 kV.

To further understand this contrast inversion, Monte Carlo simulations were conducted to determine the backscatter coefficients for each element at the appropriate effective accelerating voltages. Using appropriate conditions for the simulations, a contrast inversion is predicted to occur at 0.41 kV for Al and Au (figure 2e), which matches the experimental results (figure 2a-c).

The ceria-titania nanoparticle sample was examined under similar conditions. This proved to be challenging, as the size and shape of the nanoparticles was remarkably similar. Images recorded at 1.5 kV (figure 3a) show distinctly different areas, with darker contrast TiO₂ nanoparticles (for Ti, Z=22) and brighter CeO₂ nanoparticles (for Ce, Z=58). At lower voltages, such as 0.25 kV (figure 3b) the contrast was very similar between the different nanoparticles. Monte Carlo simulations (figure 3c) predict that a contrast inversion would take place at 0.26 kV; however the contrast levels between the two materials remain very similar, not allowing for identification of each nanoparticle type via image contrast alone at very low voltages.

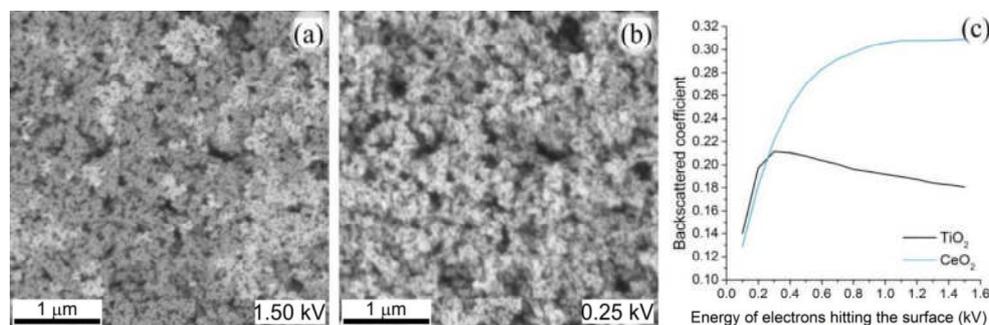


Figure 3. SEM images and analysis of a CeO₂-TiO₂ mixture. As the voltage is reduced the contrast difference observed between CeO₂ and TiO₂ changes. Backscattered electron images recorded at (a) 1.00 kV and (b) 0.25 kV. (c) Plot of the predicted backscatter coefficient of CeO₂ and TiO₂ against voltage; data generated using Monte Carlo modelling.

3.2. Energy filtering and low-loss backscatter electrons

The CNT/TiO₂ sample on an Al stub was examined at 2 kV. Figure 4a is a SE image formed using the upper detector where the low angle BSEs are attracted to the positively charged bias plates (figure 1b) and, while this does cause low energy SE emission, this is pulled back to the plates, and consequently the image is a pure SE signal giving highest resolution and surface sensitivity.

The backscatter top detector with the high-pass filter (figure 1c) permits filtering of the signal; it is possible to image using the entire backscattered signal or only the elastically scattered backscattered electrons, the so called low-loss backscatter signal.

Figure 4b does not use the filter, and here the low Z CNTs show similar contrast to the higher Z Al substrate beneath. Here the elastically and inelastically scattered BSE signal is collected, the escape depth of which is greater than the thickness of the CNT and close to the total interaction volume, such that the contrast consists of a convolution of signals from the Al substrate and the CNT together. The contrast cannot be interpreted in the conventional way expected of a backscatter signal.

Figure 4c does use the top filter such that only low-loss BSE signal is acquired. The signal sampling depth is constrained to approximately the mean free path of the backscatter electrons (since only elastically scattered electrons are being collected and backscatter electrons with energy losses emerging from deeper within the sample are rejected). Thus, the signal becomes sufficiently surface sensitive to represent the correct backscatter contrast of the materials involved and the image can be more directly interpreted. This technique may be used to provide high surface sensitivity compositional imaging without need to reduce the interaction volume or enter the regime of potential contrast inversions.

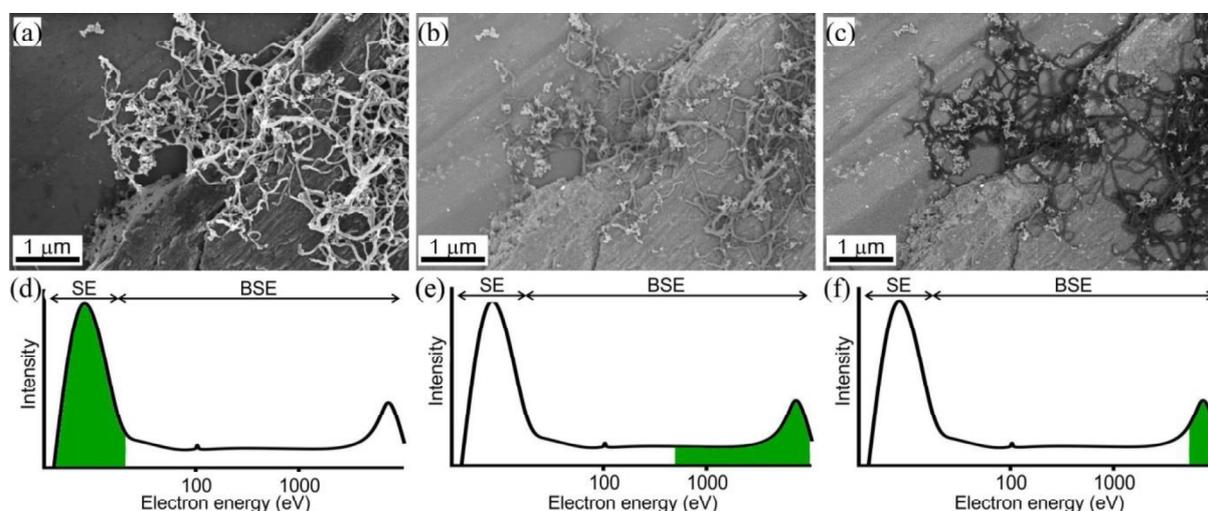


Figure 4. Images of CNT/TiO₂ sample. (a) SE image. (b) BSE image formed using the top detector without the filter. (c) BSE image formed using the top detector with the filter. (d)-(f) Schematic of electron energy spectra associated with images (a)-(c) respectively.

4. Conclusions

These results indicate how low voltage SEM is an effective tool for imaging materials with different average atomic numbers. The use of BSE imaging at low voltages, with and without energy filtering, can provide interesting contrast levels in images, some of which do not follow the expected Z-contrast. The use of energy filtering enables the surface sensitivity of backscatter imaging to be enhanced and can lead to more directly interpretable images. Monte Carlo simulations are a very effective tool for predicting when contrast inversion will occur, and can both explain the contrast levels observed and could be used to determine the most appropriate accelerating voltage to use when examining a mixture of materials.

References

- [1] Müllerová I 2001 *Scanning* **23** 379
- [2] Goodhew P J, Humphreys J and Beanland R 2001 *Electron Microscopy and Analysis, Third Edition* (London: Taylor & Francis)
- [3] Müllerová I and Frank L 2004 *Scanning* **26** 18
- [4] Rodenburg C, Jepson M A E, Inkson B J, Bosch E G T and Humphreys C J 2010 *J. Phys. Conf. Proc.* **241** 012074
- [5] Tsurumi D, Hamada K and Kawasaki Y 2012 *Jpn J Appl Phys* **51** 106503
- [6] Nagoshi M, Aoyama T and Sato K 2013 *Ultramicroscopy* **124** 20