Experiment Brief

Cipher system

Title

Quantified backscattered electron analysis in the scanning electron microscope (SEM)

Gatan Instrument Used

Cipher™ system, including the OnPoint™ backscattered electron detector and DigitalMicrograph® software.

Background

In the scanning electron microscope (SEM), backscattered electron (BSE) imaging is used routinely to reveal the compositional variation within a specimen. The contrast in a BSE image results from the difference in average atomic number between analysis points. Typically, BSE imaging is performed qualitatively; however, quantitative BSE (qBSE) imaging may be performed under appropriate conditions to determine the mean atomic number $(\bar{Z})$. Recently, interest in qBSE analysis has surged after researchers demonstrated the ability to reveal single-digit weight percentages of lithium in a sample quantitatively using qBSE and quantitative energy dispersive x-ray spectroscopy (EDS) analysis [1, 2]. In this article, we examine the use of the OnPoint BSE detector and DigitalMicrograph software (components of the Cipher system) for qBSE applications.

Materials and Methods

qBSE analysis was performed on 55 high purity standards ($4 < \bar{Z} < 83$). The samples were mechanically polished and coated with a 2.0 nm thick carbon layer using a PECS™ II system. Figure 1 shows the qBSE signal measured as a function of $\bar{Z}$ (calculated using the modified electron approach after [3]). For compounds with $\bar{Z} < 40$, an excellent fit of the experimental data to the trendline was observed with few—if any—outliers.

A second sample was analyzed to investigate the accuracy of qBSE measurements in randomly orientated crystals. LiF particles, approximately cuboidal in shape and 1 – 15 µm in size, were embedded in epoxy resin. The sample was milled to reveal the particle interior and coated with a 2.0 nm thick carbon layer using a PECS II system. qBSE analysis of the particles was performed, Figure 2. The $\bar{Z}$ determined experimentally was 7.15 ± 0.19, comparing favorably with the calculated value of 7.10. Analysis of the lithium content by Cipher reveals the mean lithium content to be 48.8 ± 0.04 at.% and demonstrates that channeling contrast is not a significant factor in determining the lithium content under these conditions.

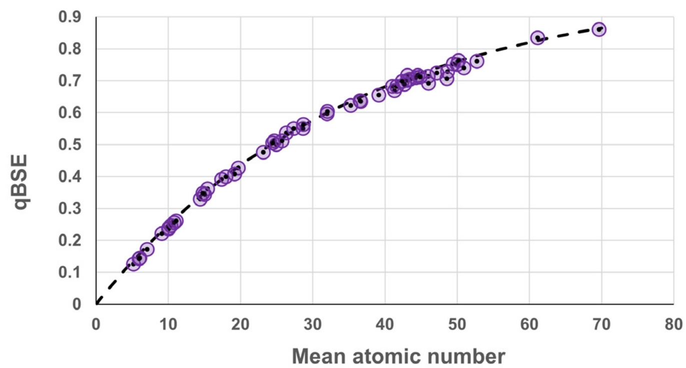

Figure 1. The plot of normalized backscattered electron grey levels against mean atomic number was calculated using the modified electron approach (after [3]). The circles are experimental data collected at an accelerating voltage of 15 kV, and the dotted line is the exponential fit function.

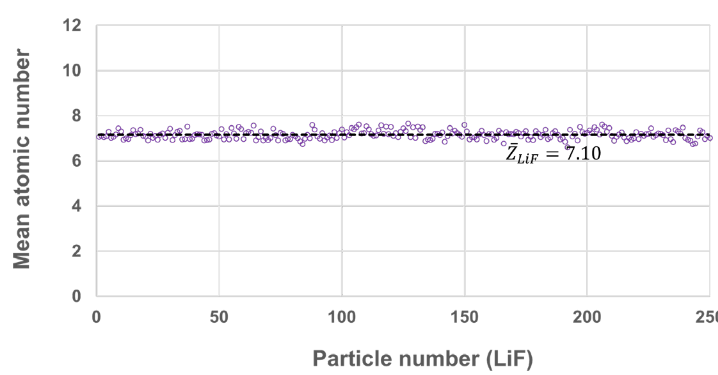

Figure 2. Mean atomic number of 250 LiF particles as determined by qBSE analysis using the Cipher system. Collected at an accelerating voltage of 15 kV.

Summary

Precise determination of a sample's mean atomic number $(\bar{Z})$ by quantitative backscattered electron analysis was demonstrated for a wide range of materials of $\bar{Z} < 40$. Crystal orientation was found to have minimal effect on qBSE results enabling the lithium content of LiF particles to be determined with an accuracy of 1.2 at.% (<1 wt.%) by the Cipher system. These results promise exciting characterization possibilities of lithiated materials.

Gatan, Inc. is the world's leading manufacturer of instrumentation and software used to enhance and extend electron microscopes—from specimen preparation and manipulation to imaging and analysis.

Credit(s)

[1] J.A. Österreicher et al., Scripta Materialia 194 (2021), 113664
[2] J. Lee et al., Microsc. Microanal. 28 (2022), p548-550
[3] J. Donovan et al., Microsc. Microanal. 9 (2003), p202

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