Mineral identification using a scanning electron microscope

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Abstract
Quick, inexpensive and accurate mineral-phase identification is of utmost importance to applied mineralogists. The precise relationship between the intensity of measured backscattered electrons in a scanning electron microscope and the average atomic number of the specimen is still argued. Nevertheless, much can be gained by using backscattered electron images to identify mineral phases in a specimen. An application was developed to facilitate this method and makes it a practical, inexpensive and valuable option in many mineralogical investigations.

Key words: Mineral identification, Scanning electron microscope, Backscattered electron detectors

Introduction
Scanning electron microscopes (SEM) and backscattered electron (BSE) detectors have been used for several decades. It is known that the intensity of the BSE signal is a strong function of the average atomic number under the electron beam and that this fact can be exploited to develop a useful mineral identification method. Still, there has not been an agreement on the relationship between the BSE coefficient and the atomic number or the appropriate way to average the atomic numbers in compounds. Models were proposed by Müller (1954) and Salick and Allen (1954) in the 1950s; by Everhart (1960) in the 1960s; and by Büchner (1973) in the 1970s. These models have been restudied, modified and supported several times since their inception. Yet, none of these models has been universally accepted. In fact, in 1998 Howell et al. (1998) found that none of the proposed relationships accurately predicted the BSE peaks when applied to biomaterials. Because of the disagreement over the exact relationships, the practical value of mineral identification using the backscattered electron imaging is being overlooked. If a relationship could be shown to have acceptable results in mineralogical specimens, it could be applied in many practical situations.

Quantitative image analysis (IA) is a valuable tool for practical mineralogical investigations in the mining industry. In addition to mineralogical composition, quantitative IA can be used to determine the associations of the phases of interest, the liberation of the valuable minerals from the gangue minerals for a given particle size distribution and the important mineralogical textural properties that are relevant for processing. In general, the minerals that are of significance in any mined ore are well known in advance. For example, in a common copper-porphry ore, one would expect to find chalcopyrite, pyrite and bornite as abundant sulfides and gangue composed of many silicates and carbonates. In this type of ore, it is of the utmost importance to distinguish between the gangue minerals and the sulfide minerals, and this is straightforward with backscattered electron images. However, it is not very easy to accurately distinguish chalcopyrite, bornite and pyrite. Existing EDS (energy dispersive spectroscopy) aided systems, such as the QEM®SEM, are now being used to overcome this problem. However, this class of systems is both slow and very expensive (Petruk, 2000). An alternative solution based solely on backscattered electron images would offer significant cost savings. An additional advantage of this method stems from the insensitivity of EDS to light atoms. This makes it difficult for EDS to distinguish certain phases such as iron oxides. Backscattered electron analysis does not have this limitation.

This paper describes the development of a convenient mineral identification method based on backscattered electron images generated in a scanning electron microscope.

Theoretical background
An image of a specimen can be made with a scanning electron microscope (SEM). In a SEM, a narrow stream of electrons is accelerated towards a specimen. Electrons interact and scatter when they impact the sample. Images are made by scanning the specimen and mapping the intensity of corresponding scattered electrons and other energy signals.

Inelastic collisions produce secondary electrons (SE) that are used to map the topography of a sample. Elastic collisions reflect the primary electrons, called backscattered electrons. Of all the electrons fired from the electron gun, only a fraction of them are backscattered. This fraction (η) is called the BSE coefficient. The coefficient is related to the atomic number of the phase interacting with the electron beam, but as mentioned
above the relationship is still argued. Many empirical equations have been put forward on the basis of the natural log, such as proposed by Müller (1954) and further developed by Lloyd (1987), such as the following equations:

\[ \eta = \frac{\ln(Z)}{6} + \frac{1}{4} \]  

(1)

where

Z is the atomic number of the elements in the sample.

This well describes the response of heavy samples (high atomic numbers). But it poorly describes samples with low atomic numbers and should not be used with atomic numbers below ten. A relationship proposed by Reuter (1972) gives a better fit at low Z:

\[ \eta = -0.0254 + 0.016Z \pm 1.86 \times 10^{-4} Z^2 + 8.3 \times 10^{-7} Z^3 \]  

(2)

A new equation is proposed here, namely

\[ \begin{align*}
\eta &= C_{1}Z + B_{1} & Z &\leq Z' \\
\eta &= C_{2}\ln(Z) + B_{2} & Z &> Z'
\end{align*} \]  

(3)

At the point Z', the BSE coefficient and its slope are the same using either half of the compound function. The BSE coefficient can linearly be related to S, as shown in Eq. (4):

\[ \eta = C'S + B' \]  

(4)

where

\[ S = \begin{align*}
\frac{Z}{Z'} + \ln(Z') & Z \leq Z' \\
\ln(Z) & Z > Z'
\end{align*} \]

The values of C' and B' have been empirically found to be 0.1987 and -0.3623, respectively. The value of Z' is close to 14.46. Figure 1 compares the three mentioned relationships against measured data.

Because in practice signals from the BSE detectors are sent through an amplifier, the intensity (I), measured in gray level units, recorded in the image is related to the amount of BSE striking the detector (n) and internal amplifier coefficients for contrast (C") and brightness, (B")

\[ I = nC'' + B'' \]  

(5)

The number of BSE striking the detector (n) is related to the probe current (\(P\)) multiplied by \(\eta\) and the fraction (\(f\)) of all BSE produced that strike the detector. This gives a new equation for intensity

\[ I = (Pnf)C'' + B'' \]  

(6)

By substituting in the equation for \(\eta\) (Eq. (4)), the following equation is produced

\[ I = PfC''C' + PfC''B' + B'' \]  

(7)

In the process of taking an image, the variables \(P, f, C''\) and \(B''\) are kept constant. Thus, the differing intensities in an image are due only to a change in \(S\) and, hence, atomic number. The other terms can be combined into two constants

\[ I = CS + B \]  

(8)

where

\[ C = PfC''C' \]

\[ B = PfC''B' + B'' \]

For practical convenience, C and B will be called contrast and brightness, respectively.

This results in a very simple and practical equation that relates the intensity, measured in gray level units, to the atomic number of the phase under the beam. Each image of a properly prepared sample will have associated brightness and contrast levels. If these levels can be determined, the atomic number can be estimated from the gray level intensity in the image. Figure 2 shows how some measured data fit with linear regression by the three relationships described above.

So far, only pure elements have been discussed. There has not been any consensus as to the best way to deal with compounds. In a study of the various methods, Howell et al. (1998) found that Castings method of mass weighting the corresponding BSE coefficients gave the best results. This approach can be modified so that it can be used with S, because \(\eta\) is linear with \(S\).

\[ S = \sum \frac{N_{i}A_{i}S_{i}}{N_{i}A_{i}} \]  

(9)

The \(S\) can be converted back to a \(\bar{Z}\) or an average atomic number.

**Method**

To begin the mineral identification process, the brightness (B) and contrast (C) levels must be determined for an image. As seen above, these coefficients are functions of the probe...
current, the fraction of BSE that strike the detector and the contrast and brightness coefficients used in the amplifier. In practice, it is difficult to calculate $B$ and $C$ using these variables. Many of the obstacles in doing so are discussed in a paper by Ball and McCartney (1981).

The most practical way to determine $B$ and $C$ is by calibrating them using phases of known $S$. Only two known phases are necessary to calibrate the brightness and contrast levels, but when using more than two phases, these levels can be determined with greater confidence and accuracy through linear regression. Once the $C$ and $B$ values have been calibrated using the known phases, the $S$ value of unidentified phases can be calculated.

In cases where there are not enough phases that are readily identifiable in a specimen, standard materials embedded in the sample holder can be used to calibrate the brightness and contrast of an image near the edge of the sample. The brightness and contrast levels of the image can be determined and the $S$ values of the phases can be estimated. Subsequently, these newly identified phases can be used to determine the brightness and contrast levels of images taken in other areas of the sample.

In samples that contain particles suspended in an epoxy, the epoxy can be used as a calibration point. The $S$ value will not change across the sample if the epoxy is well mixed. Different kinds of epoxies will have different $S$ values. Nevertheless, once the $S$ value is first determined using other known phases, it can be used to calibrate $C$ and $B$ in other images.

![Figure 2](image2) — The three mentioned methods of determining the backscattered electron coefficient converted to Gray Level, compared to data gathered by Schneider (2001).

**Implementation**

It becomes quite apparent that an application could greatly facilitate the process described above. Such an application was developed called MinID. MinID is a Tcl/Tk/C/C++ application that combines a histogram display (Fig. 3) with a mineral database (Fig. 4). The brightness and contrast levels of an image are determined by selecting a gray level peak on the histogram, corresponding to a known phase, and assigning a mineral to the peak. The minerals can be looked up in a large database containing common minerals. Other minerals can be entered and saved in user-maintained databases. Once two or more minerals are associated to peaks and identified as known

![Figure 3](image3) — The main histogram window in MinID. The peaks correspond to phases in the sample. The vertical lines show where a mineral has been assigned to a phase.

![Figure 4](image4) — The interface to the MinID database.
phases, the brightness and contrast levels of the image are automatically calculated by linear regression. The $S$ value for every gray level is now known. When three or more peaks are identified as belonging to known phases, the 95% confidence intervals are also calculated. By clicking on peaks in the histogram, a window is brought up with a list of minerals in the databases whose $\bar{Z}$ values are close to what is calculated for that point in the histogram. Many other features assist in the identification and evaluation processes. A graph can be displayed plotting the minerals on a gray level vs. $\bar{Z}$ (Fig. 5) or gray level vs. $\bar{S}$ (Fig. 6).

**Experiment**

A ground copper porphyry sample from Indonesia was mounted in epoxy under vacuum conditions. The sample surface was then ground and polished. The samples were coated with a uniform carbon film to facilitate the exit of accumulating electrons from the beam. The sample was placed in a specially prepared sample holder. Three standards — platinum, aluminum and cobalt — are embedded in the rim of the sample holder. The rim of the sample holder was also polished with a 1-µm slurry.

Six images of the copper porphyry sample were made in a Topcon SM-300 scanning electron microscope. Three of the images included the sample holder with the embedded standards and the sample; the others imaged only the sample. Three of the images were taken at different contrast and brightness settings on the amplifier.

**Results**

Using MinID software, histograms of the images were made. The peaks corresponding to the standard phases were located and the atomic numbers, looked up in the database, were associated with those peaks. Automated linear regression was used to derive the contrast and brightness coefficients for the images (Fig. 6).

Histograms of individual particles were made (Figs. 3 and 7). Pyrite and chalcopyrite particles were identified by the estimated average atomic number. All phases were found to fit the relationships (Fig. 5).

The same particles of pyrite and chalcopyrite were used in another image (Fig. 8) to calibrate the brightness and contrast. For most commercial SEMs, it can be assumed that the brightness and contrast of groups of images acquired together will have the same brightness and contrast, as long as the sample is prepared properly and the settings have not been altered. Therefore, it is not requisite that the same particles are found again.
Conclusions
A practical method was developed to identify mineral phases rapidly using an integrated computer package with backscattered electron images generated in a scanning electron microscope. The method can easily be calibrated using any known phases in the image, including phases not in the specimen. The method will be of considerable interest to applied mineralogists in the mining industry, particularly those dealing with problems associated with the efficient liberation of economically important mineral. This method can be especially useful when coupled with other computer automated image analysis tools such as the Metallurgy and Materials Image Analysis (MMIA) that was developed at the University of Utah (King and Schneider, 1993).

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References

Figure 8 — Another image of the same sample. The stage has been moved, the image zoomed and the brightness and contrast adjusted. Individual particle histograms have been made of the same particles.

Schneider, C.L., 2001, private communication.