

The SEM examination of geological samples with a semiconductor back-scattered electron detector

MALCOLM G. HALL AND GEOFFREY E. LLOYD

*Centre for Materials Science, University of Birmingham,
Edgbaston, Birmingham, United Kingdom*

Abstract

This paper describes the use of the back-scattered electron (BSE) mode of scanning electron microscope (SEM) operation for mineralogical and earth sciences applications. Such operation requires an efficient detector system, and of the types presently available, the one described here (an annular silicon diode) gives comparable if not superior results to the commercially available scintillator-photomultiplier systems. Other BSE detectors constructed from photodiodes are also found to give good results and are inexpensive and simple to manufacture. Several aspects of BSE image quantification are considered. It is shown how atomic number contrast (which arises from the dependence of the BSE coefficient on composition) may be calculated, and the BSE coefficients for a wide range of common minerals are presented. Atomic number contrast gives rise to only a small proportion of the total image signal, and in order to remove other sources of contrast, special attention should be paid to specimen preparation. Carbon coating is found to be the best method of preventing the specimen from charging because unlike other methods (*e.g.*, gold coating, environmental cell) it does not interfere with image detail or X-ray analysis. The paper also shows how compositional information and mineral proportions may be obtained from atomic number contrast images using a multichannel analyzer.

Introduction

In a recent article Robinson and Nickel (1979) draw attention to the benefits of using a back-scattered electron (BSE) detector for the examination of geological specimens with the scanning electron microscope (SEM). They suggest, correctly, that the approach is of considerable value to the geologist as it allows mineral phases to be observed with good resolution and the simple variation in image brightness which accompanies variations in composition (usually termed atomic number contrast) makes it particularly suitable for phase and mineral identification. Their contribution describes the use of a scintillator-photomultiplier electron detector and a low vacuum ("environmental cell") specimen chamber to alleviate the specimen charging effects that must be eliminated when non-conducting samples are examined in the SEM.

For several years, we have been able to obtain good BSE images from a wide range of materials (geological, metallurgical and ceramic) with a different detector system and specimen environment. Be-

cause our results are similar, and in some cases superior, to those described by Robinson and Nickel, we wish to present these alternative techniques. In particular, we wish to correct the impression that the results of Robinson and Nickel are unique to their imaging system. We also show how further information may be obtained from the BSE image, especially with regard to mineral compositions and proportions, possibilities not discussed by Robinson and Nickel.

Back-scattered electron detectors

The most common image obtained with an SEM is one showing surface topography. These images, produced by variations in secondary electron emission over the sample surface, are simple to obtain and most appropriate for the examination of rough samples. Practically all SEM's employ the Everhart-Thornley detector (Everhart and Thornley, 1960) for this purpose because of its noise-free amplification and good collection efficiency. As secondary electrons (SE's) are low in energy (<50 eV) the detector can also be placed well away from the sample with a

biasing voltage to attract the electrons. BSE's are more energetic than SE's and are unaffected by weak electrostatic fields. Efficient BSE detectors must therefore subtend as large a solid angle as possible with the specimen so as to intercept as many electrons as possible. The arrangement used in our SEM is shown in Figure 1, which details the relative positions of the electron detector, electron probe forming system, and specimen. Two efficient BSE detector systems have recently emerged which employ either a large area of unbiased scintillator (e.g., Everhart *et al.*, 1959; Wells, 1970; Robinson, 1973, 1975; Schur *et al.*, 1974) or a semiconductor device consisting of a single p-n silicon diode or diode array (e.g., Wolf and Everhart, 1969; Lin and Becker, 1975; Stephen *et al.*, 1975).

The relative merits of the two BSE detector systems are as follows. Scintillator-photomultipliers initially require less instrumental modifications as they are interchangeable with the Everhart-Thornley system and may be operated at TV scanning rates. However, they degrade with use and probably require frequent attention to the coatings. They also tend to be rather bulky. Semiconductor detectors, on the other hand, are extremely robust (we have had ours for about 5 years and have used it for many purposes, including *in-situ* heating experiments) and can be limited to a few millimeters in thickness. The semiconductor device, however, needs an extra (simple) amplifier, and although it will not normally

operate at TV rates, it's frequency response is adequate for visual examinations with frame times of 1–2 sec. Most of the results presented here were obtained with the diode system developed by Stephen *et al.* (1975), and although this is difficult to obtain commercially, we have had good results from an alternative constructed from low noise photodiodes. Such diodes are easily obtainable, for example from OCLI (15251 East Don Julian Rd, City of Industry, California 91746) and four diodes, enough to manufacture a detector, cost about \$15.

A measure of the performance of a BSE detector is the minimum difference in composition that is detectable as atomic number contrast. For the two systems considered here this would appear to be very similar. Robinson and Nickel (1979, Fig. 13) are able to distinguish between Fe_7S_8 and Fe_9S_{10} , whereas we are able to obtain clear contrast between the α and β phases in a Cu–Zn alloy, an almost identical difference in atomic number of ~ 0.1 .

Methods of preventing specimen charging

Non-conducting materials, including most geological specimens, examined in the SEM will charge unless preventive measures are taken. The charging artifacts which are produced are undesirable for good image detail and clarity. Robinson and Nickel (1979) argue that the low vacuum "environmental cell" (Robinson, 1975) is most appropriate for geological samples. They do, however, note that this method has a detrimental effect on X-ray resolution. The alternative method considered, coating with gold, is equally unsuitable as it strongly suppresses atomic number contrast, results in extra X-ray peaks and absorbs the lines of the lighter elements, especially Na and Mg.

A solution to all of these problems can be found by using a thin conducting layer of carbon; despite its use in many laboratories, this method is not discussed by Robinson and Nickel. Thin conducting layers of carbon, 50–100 Å in thickness, can be deposited in controlled amounts by vacuum evaporation and have little effect on either atomic number contrast or X-ray production. It therefore seems superfluous to consider the use of the environmental cell.

Atomic number contrast

Atomic number contrast is one of the easiest SEM contrast mechanisms to obtain. In this section we give a brief account of the relevant theory, examine

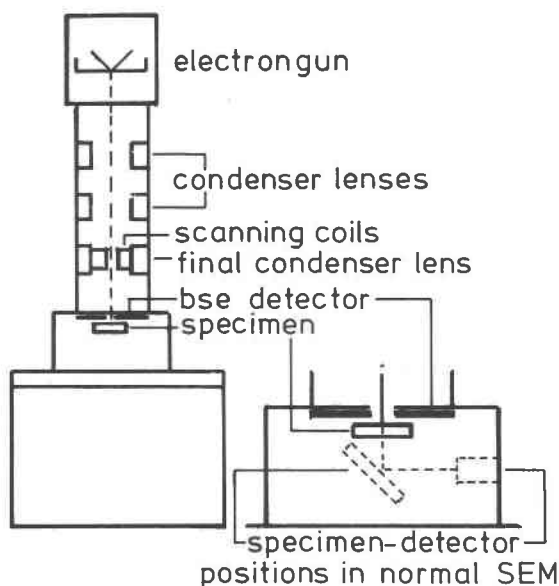


Fig. 1. Schematic diagram of SEM system showing positions of electron detectors and specimens for BSE and SE modes.

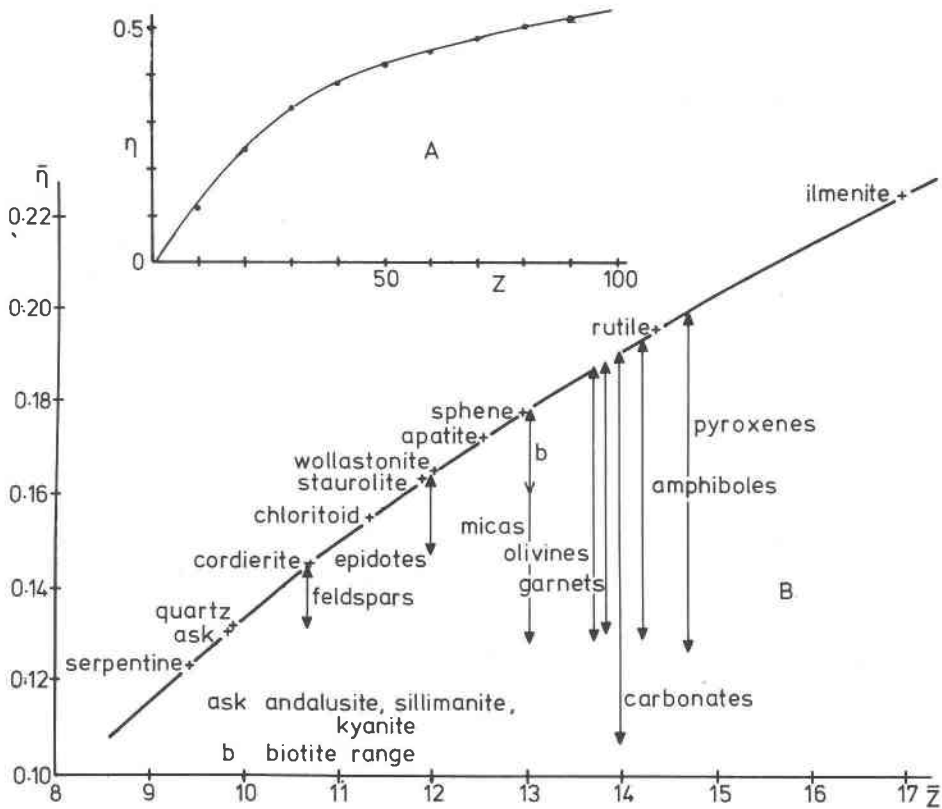


Fig. 2. (a) relationship between back-scattered electron coefficient (η) and atomic number (Z) for pure elements (after Oatley, 1972); (b) relationship between mean back-scattered electron coefficient ($\bar{\eta}$) and weighted mean atomic number (\bar{Z}) for common rock-forming minerals plotted on a curve of η against Z .

it's implications and consider some possible applications of BSE imaging to geology. Atomic number contrast is produced when the composition of the specimen varies over the field of view and arises from the dependence of the back-scattering coefficient (η) of the primary electron beam on target composition. The definition of η is the ratio of the total BSE current to that of the incident beam and for the pure elements the variation with atomic number (Z) is a simple function (Oatley, 1972) and is reproduced in Figure 2a. For compounds (*i.e.*, most geological samples) the weighted mean BSE coefficient ($\bar{\eta}$) is usually employed (Reed, 1975),

$$\bar{\eta} = \sum_{i=1}^n C(i)\eta(i) \quad (1)$$

where $C(i)$ is the concentration by weight of each element in the compound, $\eta(i)$ is the elemental BSE coefficient (see tabulation by Reed, 1975) and n the number of elements. In Figure 2b, we have superimposed the values of $\bar{\eta}$ for common rock-forming minerals on the η vs. Z curve for pure elements. In all

cases the simplest chemical formula (given by Deer *et al.*, 1966) is used. We also show $\bar{\eta}$ for a number of minerals belonging to solid solution series, here the range can be quite considerable.

If image detail is to be obtained, then some contrast, δ , must be apparent, and for two phases A and B this is given by the ratio of difference in BSE coefficients to the total BSE signal,

$$\delta = [\bar{\eta}(A) - \bar{\eta}(B)] / 0.5[\bar{\eta}(A) + \bar{\eta}(B)] \quad (2)$$

From the $\bar{\eta}$ values presented in Figure 2b, it is clear that in most geological situations the amount of contrast will be small, typically <15% of the total signal. Consequently the imaging system employs a large amount of "black level" (DC suppression) in order to reveal any detail. Specimen contrast from other sources, such as surface topography, must also be suppressed. For this reason it is important to use carefully polished specimens in which surface topography has been reduced to a minimum (uncovered thin sections are eminently suitable). The ability of any BSE system to examine unpolished or rough

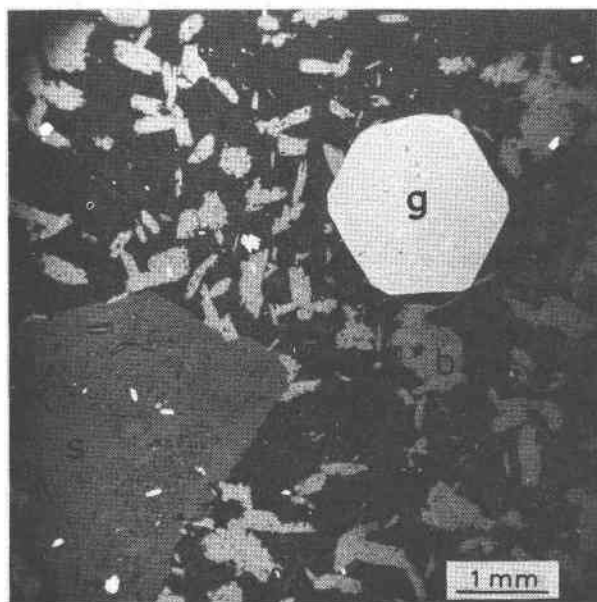


Fig. 3. Atomic number contrast image of a hornfels metagreywacke showing six common rock-forming minerals: g, garnet; b, biotite; s, staurolite; i, ilmenite; matrix consists of quartz and muscovite and is shown more clearly in Fig. 7a. Carbon coated, 30 kV accelerating voltage.

samples will be hampered by topographic contrast. The examples of rough specimens shown by Robinson and Nickel (1979, Figs. 4 & 5) represent extremes of atomic number difference, for the most part they

show images of polished specimens. Indeed, it is doubtful if BSE imaging will gain wide acceptance unless it is understood that care must be taken in specimen preparation. With careful specimen preparation most of the minerals represented in Figure 2b are capable of being distinguished.

The maximum useful magnification obtainable with the system described by Robinson and Nickel (1979) is $\sim 3,000\times$. With our system we have obtained images at $10,000\times$ and regularly use magnifications of $5000\times$ with a useful amount of image detail. Examples of BSE images showing atomic number contrast obtained with our system are presented in Figs. 3 to 6. We have concentrated on common rocks rather than those containing heavy minerals.

It should be mentioned that atomic number contrast is not the only contrast mechanism which is observed with the BSE detector and which is of use to the geologist. Other effects, such as electron channelling (e.g., Coates, 1967; Booker *et al.*, 1967; Schulson, 1977) and magnetic contrast (Holt *et al.*, 1974) are potentially relevant and frequently used in metallurgy (see Hall and Hutchinson, 1980, for a review). We have recently obtained electron channelling patterns and electron channelling contrast from geological specimens (Lloyd and Hall, in press; Hall *et al.*, in prep.) in which specimen preparation plays an even more important role.

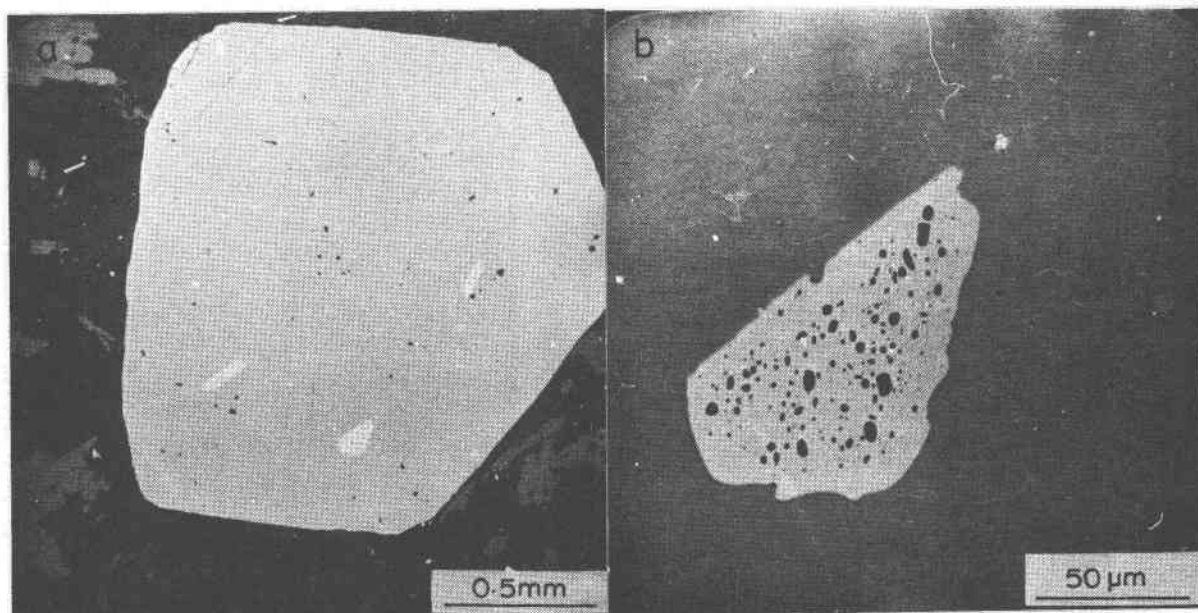


Fig. 4. Detail of garnet porphyroblasts shown in Fig. 3; (a) inclusions of quartz (black) and ilmenite (white). (b) quartz inclusions within the ilmenite. Carbon coated, 30kV.

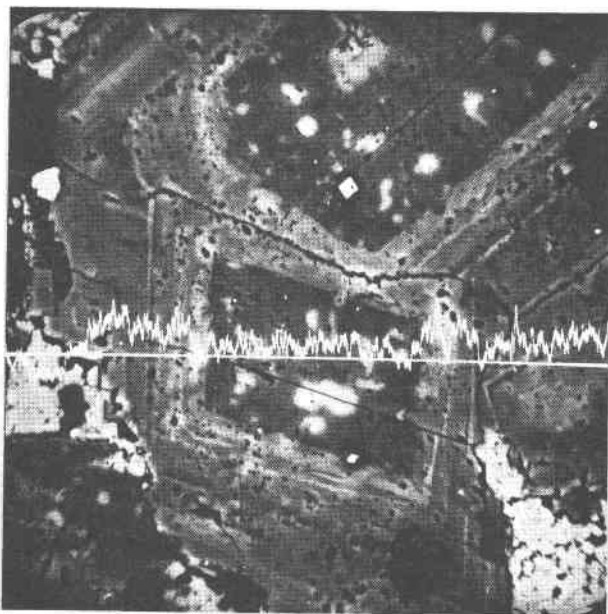


Fig. 5. Compositional zoning in carbonate. The zoning is due to variations in Fe as shown by the X-ray line scan; the brighter zones contain more Fe than the darker zones. Carbon coated, 30 kV, Si(Li) X-ray detector.

Quantitative analysis of BSE images

Since images of atomic number contrast consist of discrete levels of brightness it is often possible to use the BSE detector system to assist with mineral identification and analysis and to measure the proportions of each mineral in the image. At the same time as it was being recorded on film, the image signal of Figure 7a was processed by a multi-channel pulse height analyzer (MCA). A simple interface unit to enable this to be achieved is described in Hall and Skinner (in prep.), while the MCA is a standard feature of non-dispersive X-ray analysis systems. The "grey level" spectrum obtained (Fig. 7b) shows peaks at positions corresponding to the brightness (and therefore $\bar{\eta}$) of each phase and the display permits the accurate measurement of each brightness level. If the compositions of two of the minerals are known (or one and the zero intensity position) then the horizontal axis can be calibrated with respect to $\bar{\eta}$.

In Figure 7b quartz and ilmenite are used as calibration standards with which to determine the value of $\bar{\eta}$ for the biotite (0.1648). From Figure 2b the possible values of $\bar{\eta}$ for all biotites range from 0.160 to 0.180. The fact that this particular value falls towards the lower end of this range suggests a deficiency in a heavy element (e.g., Fe). The *mean* composition of

the biotite determined by electron-microprobe is $K_{1.64}Al_{0.98}Ti_{0.17}Fe_{2.57}Mg_{2.09}Si_{5.43}Al_{2.57}O_{20}(OH)_4$ and values of $\bar{\eta}$ calculated from the analyses range from 0.1635 to 0.1653, in excellent agreement with that determined from the BSE coefficient. However, the technique is nonspecific and except for simple systems does not allow the proportions of the components to be determined. The usefulness of the approach is therefore likely to diminish as the number of components increases. It is nevertheless a useful ancillary technique for phase identification and for performing preliminary measurements, especially when used in conjunction with X-ray analysis (e.g., the X-ray spectrum for the biotite is shown in Fig. 7c).

The grey-level spectrum obtained from the MCA can also be used to determine the proportion of each mineral phase in the image provided each brightness level is displayed as a clearly defined peak. The area under the peak is proportional to the amount of that brightness level in the image and therefore to the concentration of the mineral responsible for the peak. The integration necessary to calculate the area can usually be performed directly by the MCA and in some cases printed automatically after each image has been examined.

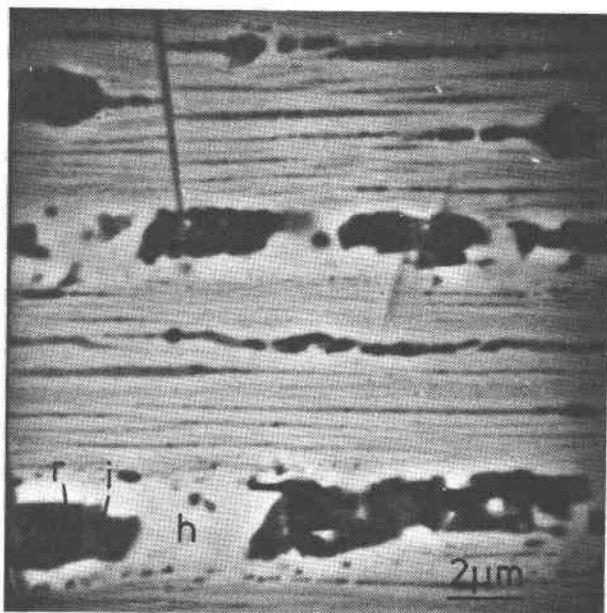


Fig. 6. High magnification ($\sim 5000\times$) atomic number contrast image of exsolution features in rutile-ilmenite hematite; the exsolved minerals are: r, rutile; i, ilmenite; and h, hematite. Carbon coated, 30 kV.

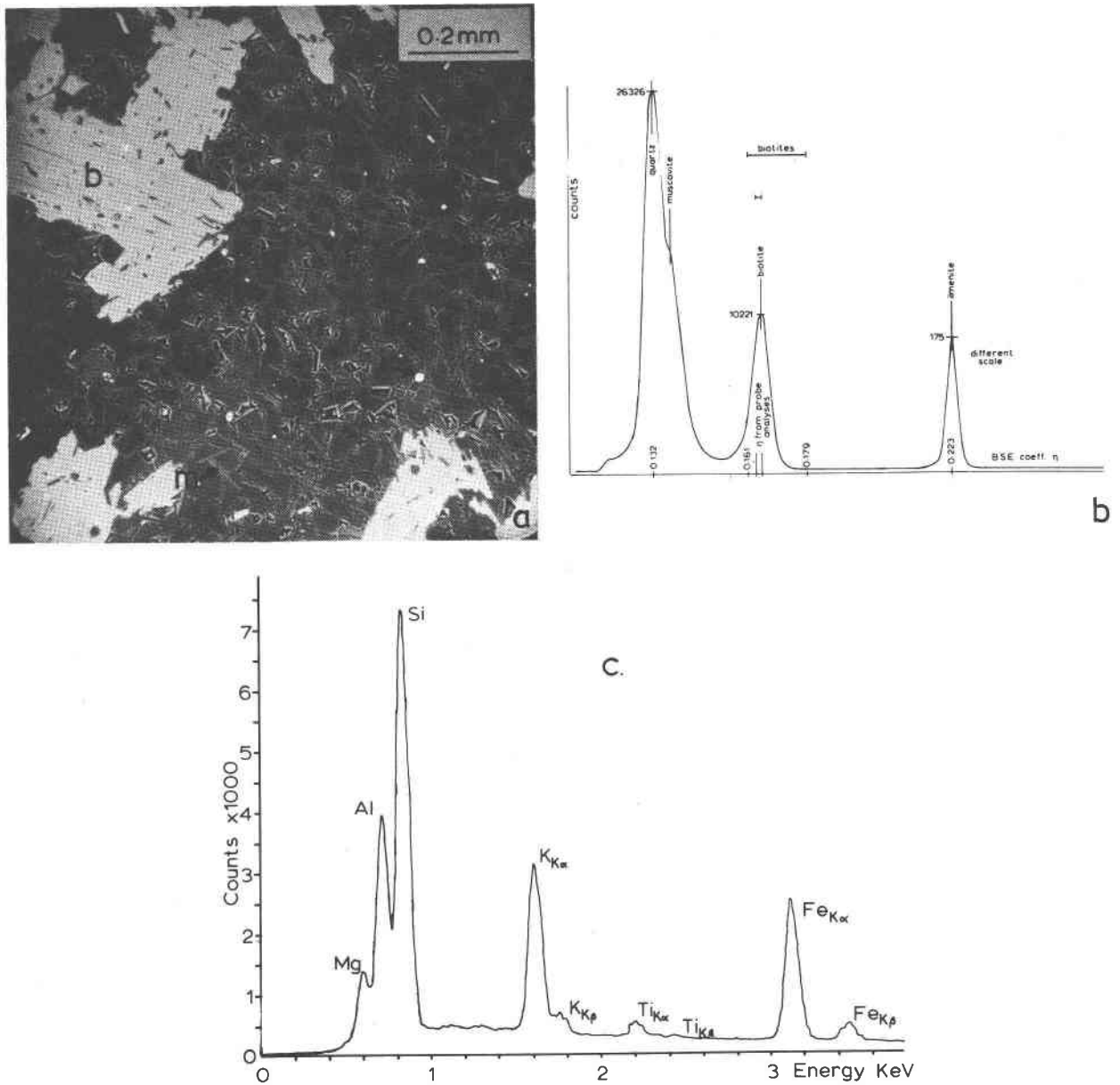


Fig. 7. (a) matrix of rock shown in Fig. 3 and used in the MCA analysis of the BSE coefficients, minerals are: q, quartz; b, biotite; m, muscovite; and i, ilmenite. Carbon coated, 30 kV; (b) grey-level spectrum obtained from (a) using the MCA; note ilmenite peak is on a different scale; (c) X-ray spectrum from biotite using the MCA; 60 sec count time, 20 kV, Si(Li) detector.

Conclusions

Back-scattered electron imaging with an SEM is a valuable technique for the study of minerals and rocks. The images invariably have a better resolution than those obtained by optical microscopy. For most investigations, especially those involving the common rock-forming minerals, specimen preparation is

important. Specimens should be carefully polished to remove surface topography and coated, preferably with carbon, to prevent charging. In certain cases atomic number contrast images can be used to determine mineral proportions and to give an indication of composition. Combined with X-ray analysis the technique shows great promise for geological studies,

especially as recent results suggest that BSE's can also be used to determine crystallographic information from rock minerals.

Acknowledgments

We thank Dr. D. W. Jones for the provision of laboratory facilities and Drs. P. K. Harvey, B. P. Atkin and J. A. D. Dickson for the specimens used. Electron microprobe analyses were performed at the Department of Mineralogy and Petrology, University of Cambridge, with the help of Dr. P. Treloar.

References

- Booker, G. R., Shaw, A. M. B., Whelan, M. J. and Hirsch, P. B. (1967) Some comments on the interpretation of the "Kikuchi-like reflection patterns" observed by scanning electron microscopy. *Philosophical Magazine*, 16, 1185-1191.
- Coates, D. G. (1967) Kikuchi-like reflection patterns obtained with the scanning electron microscope. *Philosophical Magazine*, 16, 1179-1184.
- Deer, W. A., Howie, R. A. and Zussman, J. (1966) An introduction to the rock-forming minerals. Longman, 528 p.
- Everhart, T. E. and Thornley, R. F. M. (1960) Wide-band detector for microampere low-energy electron currents. *Journal of Scientific Instruments*, 37, 246-248.
- Everhart, T. E., Wells, O. C. and Oatley, C. W. (1959) Factors affecting contrast and resolution in the scanning electron microscope. *Journal of Electron Control*, 7, 97-111.
- Hall, M. G. and Hutchinson, W. B. (1980) Smooth surface metallography using the scanning electron microscope. *The Metallurgist and Materials Technologist*, 12, 371-376.
- Holt, D. B., Muir, M. D., Grant, P. R. and Boswarva, I. M. (1974) Quantitative scanning electron microscopy. Academic Press, 570 p.
- Lin, P. S. D. and Becker, R. P. (1975) Detection of back-scattered electrons with high resolution. In O. Johari and I. Corvin, Eds., *Scanning electron microscopy/1975*, p. 61-70, IITRI, Chicago.
- Lloyd, G. E. and Hall, M. G. Applications of scanning electron microscopy to the study of deformed rocks. Presented at "Effect of Deformation on Rocks", Goettingen, W. Germany, April 1980; Proceedings to be published in *Tectonophysics*.
- Oatley, C. W. (1972) *The scanning electron microscope*. Cambridge University Press.
- Reed, S. B. J. (1975) *Electron microprobe analysis*. Cambridge University Press.
- Robinson, B. W. and Nickel, E. H. (1979) A useful new technique for mineralogy: the backscattered electron/low vacuum mode of SEM operation. *American Mineralogist*, 64, 1322-1328.
- Robinson, V. N. E. (1973) A reappraisal of the complete electron emission spectrum in SEM. *Journal of Physics*, D6, L105-L107.
- Robinson, V. N. E. (1975) Back-scattered electron imaging. In O. Johari and I. Corvin, Eds., *Scanning electron microscopy/1975*, p. 51-60, IITRI, Chicago.
- Schulson, E. M. (1977) Electron channelling patterns in scanning electron microscopy. *Journal of Materials Science*, 12, 1071-1087.
- Schur, K., Blashke, R. and Pfeifferkorn, G. (1974) Improved conditions for back-scattered electron SEM micrographs on polished sections using a modified scintillator detector. In O. Johari, Ed., *Scanning electron microscopy/1974*, p. 1003-1010, IITRI, Chicago.
- Stephen, J., Smith, B. J., Marshall, D. C. and Witham, E. M. (1975) Applications of semiconductor back-scattered electron detector in a scanning electron microscope. *Journal of Physics*, E: Scientific Instruments, 8, 607-618.
- Wells, O. C. (1970) New contrast mechanism for scanning electron microscope. *Applied Physics Letters*, 16, 151-153.
- Wolf, E. D. and Everhart, T. E. (1969) Annular diode detector for high angular resolution pseudo-Kikuchi patterns. In O. Johari, Ed., *Scanning electron microscopy/1969*, p. 43-44, IITRI, Chicago.

*Manuscript received, August 28, 1980;
accepted for publication, December 1, 1980.*