THE CANADIAN MINERALOGIST

Journal of the Mineralogical Association of Canada

Volume 22 MAY 1984 Part 2

Canadian Mineralogist
Vol. 22, pp. 213-218 (1984)

COLOR SEM-IMAGING OF MINERALOGICAL SAMPLES: SULFIDE ORES AND ZEOLITES

ARI ANTONOVSKY
CSIRO Division of Chemical and Wood Technology, P.O. Box 56, Highett, Victoria 3190, Australia

MICHAEL J. SANDY
Geological Survey of Papua New Guinea, P.O. Box 778, Port Moresby, Papua New Guinea

ABSTRACT

The proposed technique for mineralogical scanning-electron-microscopy (SEM) studies utilizes back-scattered-electron and secondary-electron images to form a composite image in color, as shown by such color images of coarsely and finely polished surfaces of sulfide ores and unaltered zeolite minerals. Contrasts between minerals in the sample, both in atomic number and in textural relationships, are visible in these images. The ability to identify phases and artifacts is improved by inclusion of three SEM signals in one image. The use of color enhances both aesthetic quality and detail of technical illustrations.

Keywords: scanning-electron microscopy, composite image in color, sulfides, zeolites.

SOMMAIRE

On combine, en microscopie électronique à balayage (MEB), les images produites par les électrons rétrodispersés et les électrons secondaires pour former une seule image composée, en couleur. On présente de telles images de surfaces grossièrement ou finement polies de minerai de sulfures et de zéolites inaltérées. Les contrastes entre minéraux d'un même échantillon, tant en nombre atomique qu'en relations texturales, sont visibles dans ces images. L'identification des phases (et d'objets insolites) est facilitée par l'utilisation de trois signaux MEB dans une même image. Les illustrations techniques bénéficient de la qualité esthétique et de la résolution améliorée que permet l'utilisation de la couleur.

(Traduit par la Rédaction)

Mots-clés: microscopie électronique à balayage, image composée en couleur, sulfures, zéolites.

INTRODUCTION

The scanning-electron microscope (SEM) is finding wide application in mineralogical and geological studies as relevant instrumentation and techniques are developed (Blaschke 1976). The selection of signal detectors used is crucial in determining the information obtained from a sample, and a variety of detectors may be utilized in the examination of a particular sample (Northrup 1972). A method is described here for utilizing the increased capacity of color media to present information obtained from several detectors, notably the secondary electron (SE) and back-scattered electron (BSE) detectors, in a single image.

In mineralogical work the SE, BSE and X-ray signals are commonly utilized for the imaging of samples (Green *et al.* 1979). A detector of secondary electrons (Everhart & Thornley 1960) produces an image that shows sample morphology, and this is useful for examining the structure of rocks in an unaltered state (Bull 1978). A detector of back-scattered electrons (Robinson 1975, Lin & Becker 1975) reveals differences in average atomic number (Z_{av}) across a sample surface, typically a polished surface of a rock sample (Stanton & Finkelman 1979). The BSE image has proved to be of considerable value in mineral and geological studies where elemental information is needed (Robinson & Nickel 1979, Hall & Lloyd 1981). In many cases, a combination of signals provides much more information than any one signal alone (Ekelund & Werlefors 1976).

The use of color makes it possible to achieve a

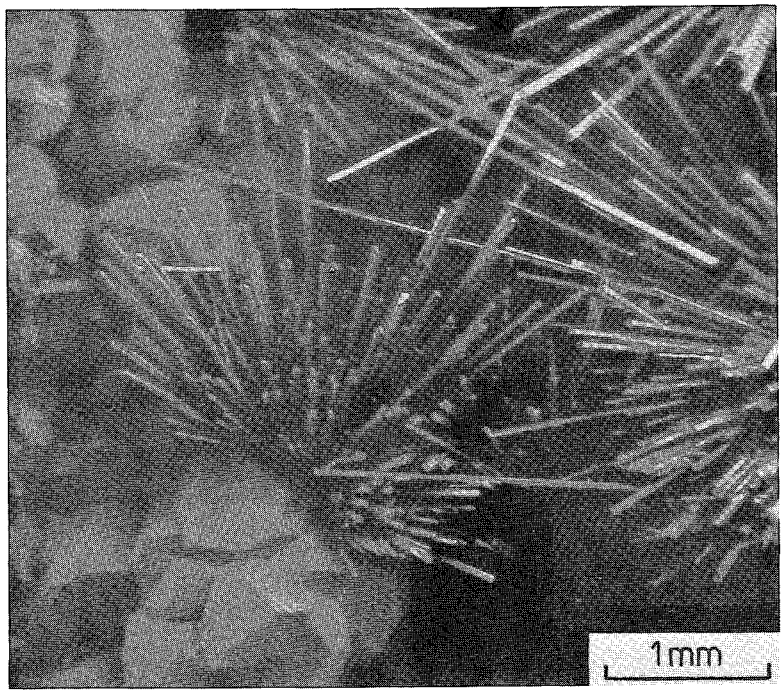

FIG. 1. Zeolites from the coastline near Flinders, Victoria, Australia. Trapezohedra are analcite, radiating fibres are natrolite.

higher content of information in SEM images (Hayes *et al.* 1969); color has been used to enhance SEM techniques in mineralogical studies, including X-ray mapping (Pawley & Fisher 1977) and cathodoluminescence (Obyden *et al.* 1980). Actual colors in a specimen cannot be reproduced by the SEM. Therefore, the use of color can be directed toward the presentation of compositional and morphological differences.

MATERIALS

In the present study, SEM images were produced from three samples; one has morphological interest and two have compositional interest. The first is a zeolite sample (Fig. 1) collected on a beach near Flinders, Victoria, Australia (Vince 1980, Coulsell 1980). The specimen consists of weathered, highly vesicular green basalt. The outer rim of the vesicle containing the zeolites consists of magnesian chlorite. Two zeolites are present: analcite $NaAlSi_2O_6 \cdot H_2O$ occurs as regular trapezohedra, and natrolite $Na_2Al_2Si_3O_{10} \cdot 1.5\ H_2O$ appears as radiating, fibrous clusters (Saha 1959).

The second, an ore sample (Fig. 2), is finely polished to enhance differences in composition between minerals present. It is a piece of drill core from a copper–zinc prospect of copper-rich massive sulfides near Daly River, Northern Territory, Australia. The country rock consists mainly of metamorphosed green schistose rocks. The minerals observed are, in order of decreasing average atomic number: galena, sphalerite, chalcopyrite, pyrrhotite $Fe_{n-1}S_n$ (n from 5 to 16) and actinolite, which is the gangue mineral.

The third sample consists of ore (Fig. 3) from a massive sulfide deposit at the Woodlawn mine, 75 km northeast of Canberra, ACT, Australia (Malone *et al.* 1975). It is finely banded and has been polished only coarsely to allow for some topographic variation. Present are pyrite, sphalerite, galena and chalcopyrite, in order of decreasing abundance. Talc, chlorite and quartz are present in the gangue.

METHOD

The samples of zeolite and sulfide ore were cut, and the ore samples polished. After coating with carbon, they were examined in a Cambridge S4-10 Stereoscan microscope. The SE signal was detected with a positively biased Everhart–Thornley detector, whereas the low-collector voltage SE signal was obtained with the same detector operating at a reduced-bias voltage. The BSE signal was detected by using four semiconductor crystals (available from LeMont Scientific Inc., State College, Pennsylvania, USA) arranged around the ceiling of the specimen

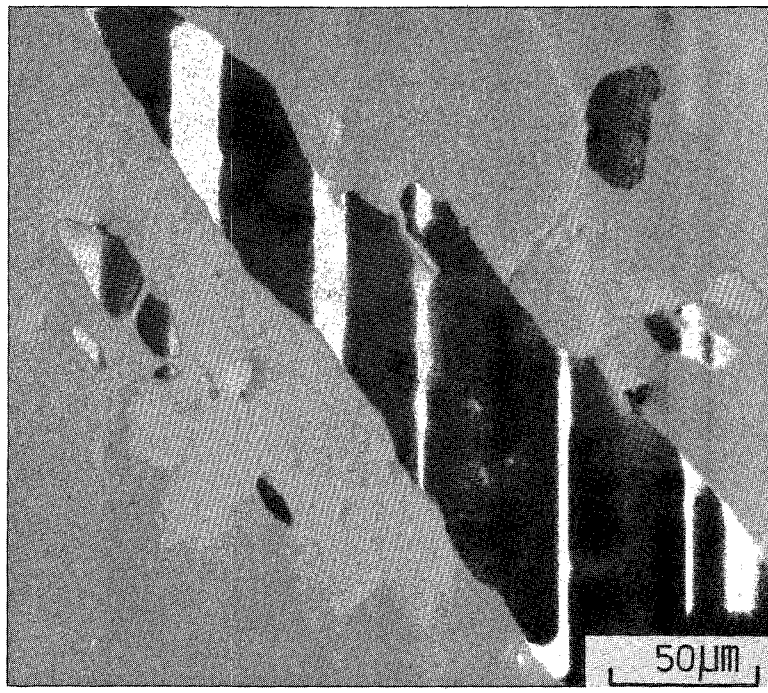

FIG. 2. Sulfide minerals from prospect near Daly River, Northern Territory, Australia. Color varies from dark green for actinolite to bright yellow for galena. Polished section.

chamber. Other signals accessible from the SEM, *e.g.*, X-ray or cathodoluminescence, could be utilized in forming the color image.

The steps followed in producing the image are illustrated in Figure 4. The BSE, SE and low-collector voltage SE image were photographed sequentially through red, green and blue filters, respectively, onto a single color film. Kodak Ektachrome transparency film was used to facilitate processing (E-6 process) and viewing. To compensate for the phosphor of the record CRT, the strength of red exposures was increased and that of blue exposures was decreased. Contrast was reduced from that of a monochrome image for red and green exposures and increased for blue exposures. The original monochrome images can be extracted by rephotographing the transparency onto black-and-white film using the appropriate filter.

RESULTS AND DISCUSSION

A variety of color images was obtained owing to the differing nature of the samples in this study. In the case of the zeolite specimen, very little compositional variation was expected [Z_{av} (analcite) = 20.22, Z_{av} (natrolite) = 20.25]; therefore, color differences within the image can be related to topography. This can be seen in Figure 1, in which fibrous natrolite produced a strong SE (green) image, whereas the polyhedral analcite produced a strong BSE (red) image. This occurred because the SE signal tends to be relatively intense at edges and at high take-off angles (Newbury 1975). However, the yield of BSE is greatest in the direction of optical reflections (Niedrig 1978), so that flat areas of the sample return the strongest signal to the BSE detector positioned at the top of the specimen chamber. The two minerals present are clearly differentiated in color on the basis of topography alone. Whereas they would remain distinguishable by morphology in a monochrome image, the use of three different SEM signals adds visual information. (Subtle structural differences that are not clearly illustrated by a single monochromatic image may be noticeable in a color micrograph.)

The core sample from Daly River represents the opposite case. Polishing removes most topographical features, leaving differences in composition to provide most of the image contrast. (Such information is derived from the BSE signal but, through color imaging, it is possible to retain SE information as well.) As shown in Figure 2, the gangue mineral (green-black) is easily distinguished from the sulfide minerals because of a weak red image resulting from

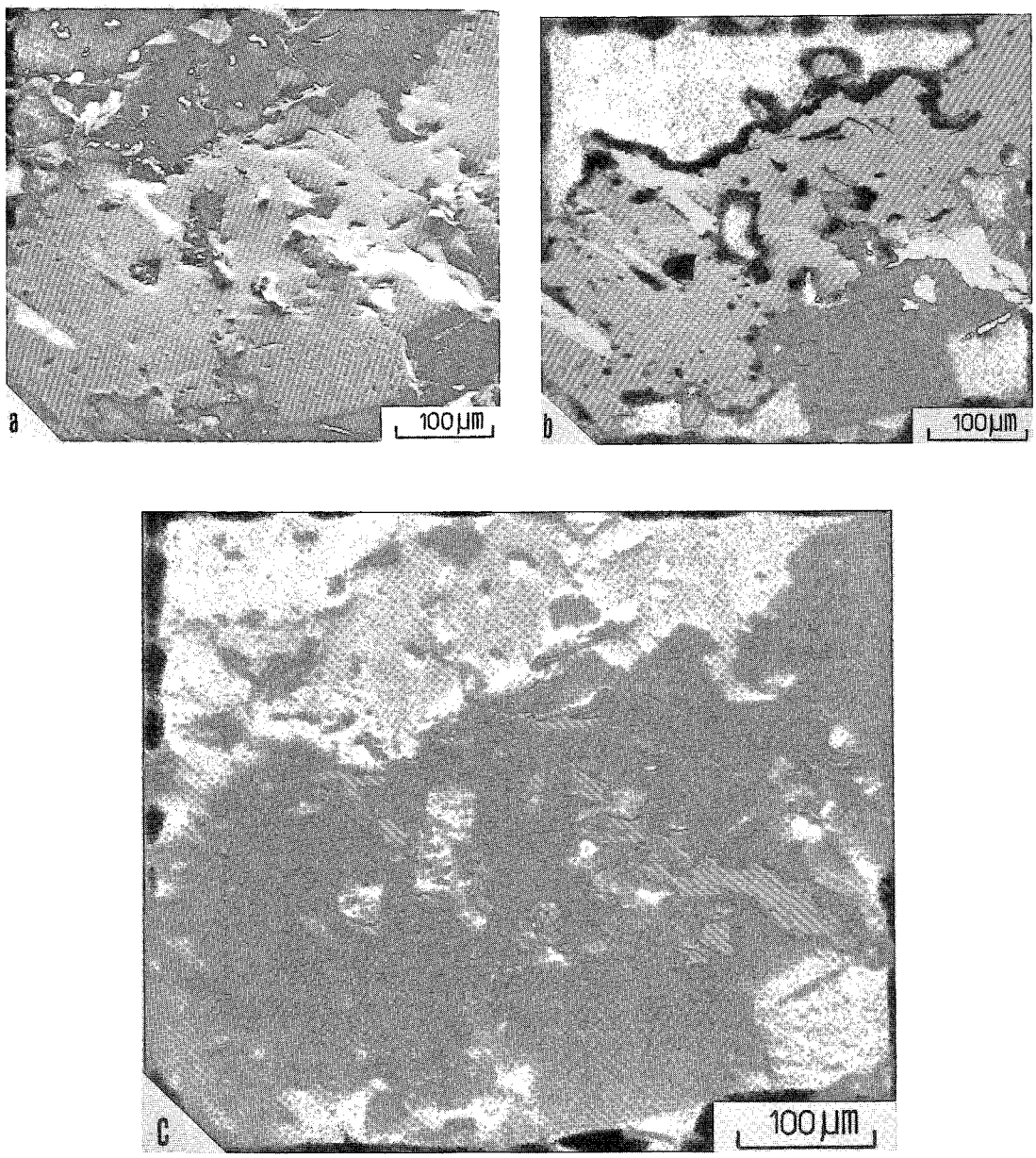

FIG. 3. Sulfide deposit from Woodland mine near Canberra, ACT. Coarsely polished section. a) SE image showing topographic features resulting from differences in hardness between minerals present. b) Image showing differences in atomic number between minerals present. c) Color image derived from BSE and SE images. The dust particles are clearly distinguishable.

its low Z_{av}. As Z_{av} increases from gangue minerals through to galena, the BSE yield increases, and so the red image component is enhanced. At the same time the SE yield is relatively constant for a polished sample, and so the intensity of the green image remains relatively even. The combination of red (BSE) and green (SE) forms areas of the image ranging from light green for pyrrhotite to orange for sphalerite and yellow for galena.

Some topographic contrast between minerals could

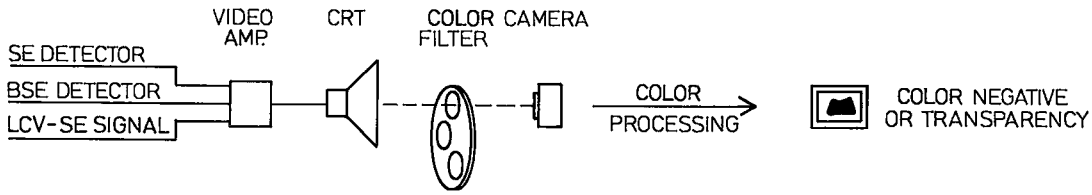

FIG. 4. Arrangement for producing a colored image from three distinct SEM images.

be observed, particularly between galena, which is relatively soft, and pyrrhotite which is relatively hard. The relationship between the minerals (*e.g.*, the position of physical boundaries relative to composition gradients and boundaries) is more easily determined when physical information is included in the micrograph concurrently with compositional information.

Topographic contrast was also observed in the ore sample from the Woodlawn mine (Fig. 3). Incomplete polishing during preparation introduced a topography based on relative hardness of the minerals present. The dark regions in the secondary-electron image (Fig. 3a) consist of three distinct phases distinguishable by topography: a soft gangue mineral (chlorite) that had been eroded during polishing, a hard gangue mineral (quartz) that had not been eroded, and foreign particles collected on the surface. Localized charging was observed where the conductive coating was insufficient (the bright green objects indicate charging of material not part of the sample at the time of coating, *e.g.*, dust particles.) The BSE image (Fig. 3b) distinguished the sulfide minerals from the gangue and from each other by their relative brightness, which is dependent on atomic number. These details were more easily perceived in the composite image in color (Fig. 3c), in which the six minerals present, dust and charging could all be observed.

CONCLUSIONS

Considerable information for mineralogical studies is provided by the SEM through different imaging signals. A color film can effectively store three distinct images of a single sample, which can be individually retrieved by photographic means. A composite of BSE and SE images in one color micrograph illustrates both compositional and textural relationships between the minerals within a sample. In rocks exhibiting large differences in atomic number between constituents, such as sulfide ores, material and topographic variations (either naturally occurring or induced by preparation methods) shown within a single micrograph serve to distinguish one mineral from another. These variations can be perceived more easily through a color image. In other cases, such as that of the zeolites, in which morphology is a primary concern, a color image tends to be a more aesthetically pleasing medium for illustrative material for technical presentation. Particular features of interest are easily highlighted, though other features of less interest, *e.g.*, pits, dust particles and specimen charging, may also become more apparent.

The colors in the SEM image cannot be made to correspond with natural coloration. However, with a greater capacity for display of information and detail, the colored picture is a means of extending the versatility of the scanning-electron microscope in mineralogical research.

REFERENCES

BLASCHKE, R. (1976): Signals excited by the scanning beam. *In* Electron Microscopy in Mineralogy (H.R. Wenk, ed.). Springer-Verlag, Berlin.

BULL, P.A. (1978): Observations on small sedimentary quartz particles analysed by SEM. *Scanning Electron Micros.* **1978**, 821-826.

COULSELL, R. (1980): Notes on the zeolite-collecting area of Flinders, Victoria, Australia. *Aust. Mineral.* **33**, 163-167.

EKELUND, S. & WERLEFORS, T. (1976): A system for the quantitative characterization of microstructures by combined image analysis and X-ray discrimination in the SEM. *Scanning Electron Micros.* **1976**, 417-424.

EVERHART, T.E. & THORNLEY, R.F.M. (1960): Wide band detector for micro-microampere low-energy electron currents. *J. Sci. Instrum.* **37**, 246-248.

GREEN, D.A., DENEE, P.B. & FREDERICKSON, R.G. (1979): The application of heavy metal staining (OsO_4) and BSEI for detection of organic material in gas and oil shales. *Scanning Electron Micros.* **1979**, 495-500.

HALL, M.G. & LLOYD, G.E. (1981): The SEM examination of geological samples with a semiconductor back-scattered electron detector. *Amer. Mineral.* **66**, 362-368.

HAYES, T.L., GLAESER, P.M. & PAWLEY, J.B. (1969): Information content of EM images: color and depth information as vehicles for subjective information transfer. *Proc. 27th Ann. Meet., Electron Microscopy Soc. Amer.*, 410-411.

LIN, P.S.D. & BECKER, R.P. (1975): Detection of BSE with high resolution. *Scanning Electron Micros.* **1975**, 61-70.

MALONE, E.J., OLGERS, F., CUCSHI, F.G., NICHOLAS, T. & MCKAY, W.J. (1975): Woodlawn copper-lead-zinc deposit. *In* Economic Geology of Australia and Papua New Guinea. 1. Metals (C.L. Knight, ed.). *Austral. Inst. Mining Metall., Mon.* **5**, 701-710.

NEWBURY, D.E. (1975): Image formation in the SEM. *In* Practical Scanning Electron Microscopy (J.I. Goldstein & H. Yakowitz, eds.). Plenum, New York.

NIEDRIG, H. (1978): Physical background of electron backscattering. *Scanning* **1**, 17-34.

NORTHRUP, D.C. (1972): Interaction of electrons with solids. *In* The Use of the Scanning Electron Microscope (J.W.S. Hearle, J.T. Sparrow & P.M. Cross, eds.). Pergamon, Oxford.

OBYDEN, S.K., SAPARIN, G.V. & SPIVAK, G.V. (1980): Observation of long persistence luminescent materials using colour TV SEM. *Scanning* **3**, 181-192.

PAWLEY, J.B. & FISHER, G.L. (1977): Using simultaneous three-colour X-ray mapping and digital-scan-stop for rapid elemental characterization of coal combustion by-products. *J. Micros.* **110**, 87-101.

ROBINSON, B.W. & NICKEL, E.H. (1979): A useful new technique for mineralogy: the backscattered-electron/low-vacuum mode of SEM operation. *Amer. Mineral.* **64**, 1322-1328.

ROBINSON, V.N.E. (1975): Backscattered electron imaging. *Scanning Electron Micros.* **1975**, 51-60.

SAHA, P. (1959): Geochemical and X-ray investigation of natural and synthetic analcites. *Amer. Mineral.* **44**, 300-313.

STANTON, R.W. & FINKELMAN, R.B. (1979): Petrographic analysis of bituminous coal: optical and SEM identification of constituents. *Scanning Electron Micros.* **1979**, 465-470.

VINCE, D.G. (1980): Zeolites and other minerals in vesicles in the Newer Basalt of the Melbourne area, Victoria. *Aust. Mineral.* **32**, 155-161.

Received November 8, 1982, revised manuscript accepted May 18, 1983.