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A useful new technique for mineralogy: the backscattered-electron/low vacuum mode of SEM operation

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Abstract

Operation of a scanning electron microscope in the backscattered-electron/low-vacuum mode instead of the conventional secondary-electron/high-vacuum mode transforms the SEM into a powerful general-purpose tool for mineralogy and the other earth sciences. The technique has two major advantages over conventional SEM operation: (1) High-quality atomic-number contrast images are produced, providing information that is much more useful than that normally obtained from an SEM. Compositional information dominates topographic information in the images so that most phases are clearly differentiated. (2) The technique is very simple. No conductive coating is required, even at high accelerating voltages. Drill core, hand specimens, mill products, polished sections, or porous wet samples can all be imaged directly as received, and it is possible to display an image and an X-ray spectrum within a minute of receipt of the specimen.

Introduction

The purpose of this paper is to bring to the attention of mineralogists and geologists a very useful development in scanning electron microscopy, namely the backscattered-electron/low-vacuum mode of operation. This technique was developed by Dr. V. N. E. Robinson at the University of New South Wales (Robinson, 1975, 1976) and has been in use in our laboratory since mid-1977. Our experience with the technique has been most encouraging, and we feel it has an assured future in mineralogical research.

Backscattered-electron (BE) images are far more useful for mineralogy than are the secondary electron (SE) images normally used in scanning electron microscopes (SEMs). This is because BE images obtained with a detector placed above the specimen contain a great deal of compositional information (atomic-number contrast) which dominates the topographic information. [See, for example, Blaschke and Heywood (1977), Kiss and Brinkies (1976), Wells (1977).] Thus the primary feature of the BE image is the distribution of phases of different average atomic number, while the topography of the surface is only a superimposed secondary feature. On the other hand, in SE images, compositional information is usually completely masked by the dominant topographic contrast. Figures 1A and 1B illustrate the greatly increased useful image information available from a BE image when compared with a SE image of the same rough area.

Many SEMs now in use produce BE images which are inferior in quality when compared with SE images produced by the same instrument. This is due not to any intrinsic advantage of SEs over BEs, but to the common use of low-efficiency BE detectors (often the BE detector is merely the SE detector with the bias voltage removed). This may explain why the SEM has, to date, found only limited application in mineralogy and has been used chiefly to illustrate mineral morphology.

However, at least two types of BE detectors are now available, capable of producing images of the same high quality and resolution as those produced with SE detectors. Instead of the more common semiconductor BE detector, the detector chosen for this work is of the wide-angle scintillator–photomultiplier type which replaces the normal SE detector in the SEM and requires no additional electronics (Robinson, 1975). This type of detector allows the production of high-quality TV images, which is a major advantage for investigative mineralogy where large areas of sample need to be examined quickly to search for phases of interest. An example of the TV-image quality is shown in Figure 2.

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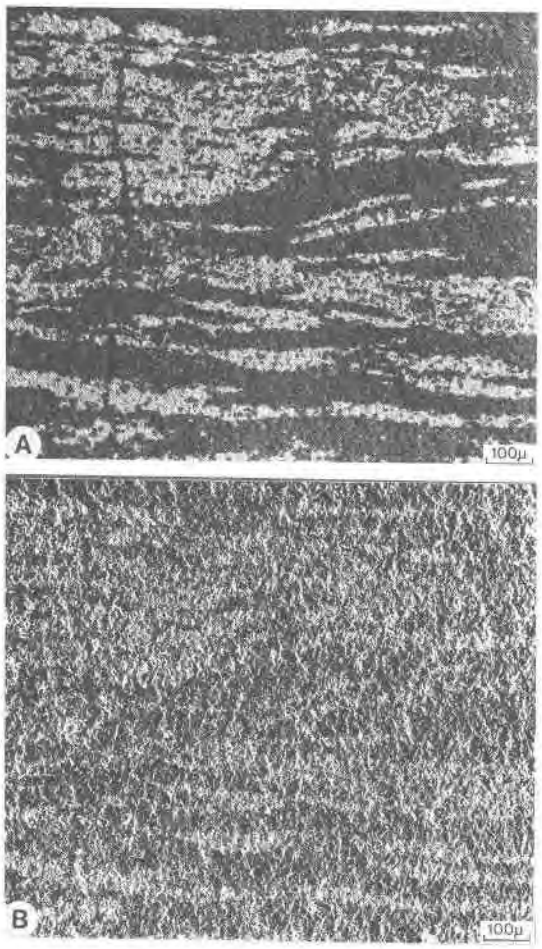

Fig. 1. (A) Backscattered-electron, and (B) secondary-electron images of the same area of a rough section of banded iron formation. The hematite (bright) is clearly distinguished from the quartz (dark) on the BE image. On the SE image, however, the compositional information is almost completely masked by topographic information of no mineralogical interest. Carbon coated, 20kV. ETEC Autoscan semiconductor BE detector.

In addition to providing a more useful image than SE detectors, BE detectors allow the use of an "environmental cell" or low-vacuum modification to the specimen chamber (Robinson, 1976). This is simply the creation of a pressure differential between the specimen chamber and the column so that, while the electron gun runs at a normal "high" vacuum, the specimen chamber is maintained at a "low" vacuum of about 0.1 torr. The pressure differential is maintained by the constricting effect of the small final aperture in the electron column. The relatively high gas pressure in the specimen chamber allows the dissipation of charge from the specimen surface and prevents the occurrence of charging artefacts in BE images. Thus no conductive coating is required even

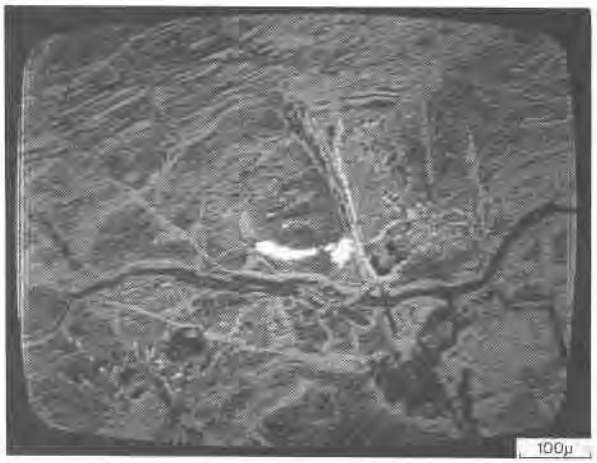

Fig. 2. Photograph of TV screen showing high-quality BE image at TV scanning rates. Polished section of gossan clearly shows a gold grain (bright); uncoated, 30kV.

for insulating specimens at high accelerating voltages. This essentially eliminates any specimen preparation and greatly increases sample throughput. Wet, dirty, and porous samples may be quickly examined without fear of contamination of the electron gun by gases or volatile compounds.

An SEM used in the BE/low-vacuum mode is a most useful tool for many mineralogical tasks, especially when it is equipped with an energy-dispersive X-ray (EDX) detector, and the technique is as easy to use as optical microscopy. As no conductive coating is required, subsequent use of other techniques on the same specimen is not hampered by the presence

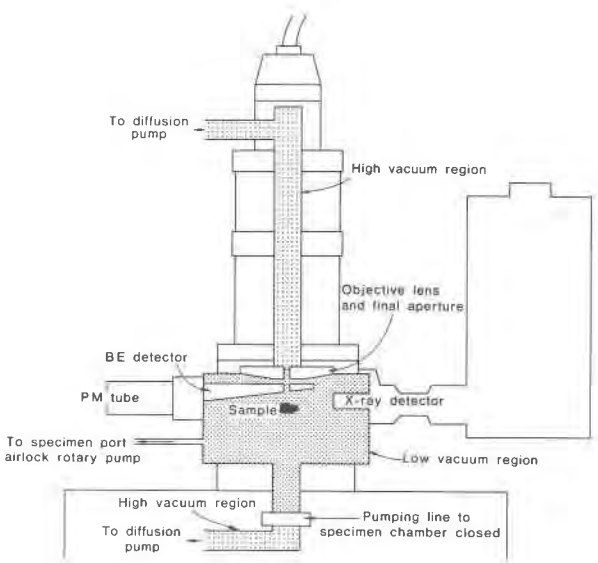

Fig. 3. Diagram of JSM-2 SEM in BE/low-vacuum mode.

Fig. 4. BE image of bromian chlorargyrite crystal [Ag(Cl,Br)] in goethite; broken surface; uncoated, 30kV. Heavy trace elements are easily found, even on a fracture surface, if concentrated in such specific phases.

of a carbon or gold coating and, furthermore, valuable museum specimens and gemstones may be examined and analyzed without alteration. Sample-change time is reduced by the low vacuum requirement, and it is quite possible to show an image and an X-ray spectrum of an unprepared mineral sample within a minute of its receipt in the SEM laboratory.

A basic SEM with a high-quality BE detector and a simple EDX system is available new for around $40,000–$60,000. This makes it closer in price, as well as in ease of use and maintenance costs, to a high-quality optical microscope than to an electron probe microanalyzer; the SEM/EDX system described here is a most useful complement to both techniques. The decreasing price and improving performance of simple SEMs should further improve the cost-effectiveness of this technique.

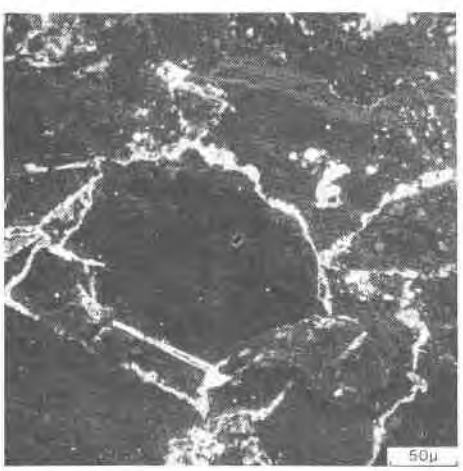

Fig. 5. Uraninite (bright) rimming sandstone grains; unpolished sawn drill-core; uncoated, 20kV.

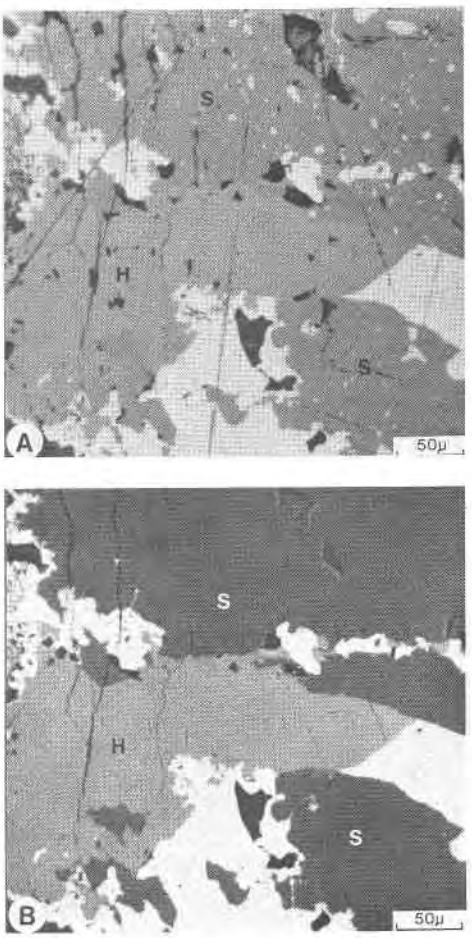

Fig. 6. (A) Optical photomicrograph of polished section, showing an ore-mineral assemblage consisting chiefly of hawleyite (H), sphalerite (S) (both gray), and galena (white). The sphalerite contains abundant chalcopyrite inclusions (also white; indistinguishable from galena). (B) BE micrograph of the same area. The hawleyite and sphalerite are now clearly differentiated, whereas there is now very little contrast between sphalerite and chalcopyrite. Uncoated, 30kV.

Modifications for BE/low-vacuum use

Many existing SEMs may be readily and cheaply adapted for BE/low-vacuum use. In the case of the JSM-2 SEM in our laboratory, a wide-angle scintillator BE detector (manufactured by ETP SEMRA, 60 Atchison Street, St. Leonards, N.S.W., Australia 2065) was installed directly above the specimen position and connected to the existing secondary-electron detector electronics. The vacuum system was modified by blanking off the normal pumping line to the specimen chamber, leaving the electron gun still

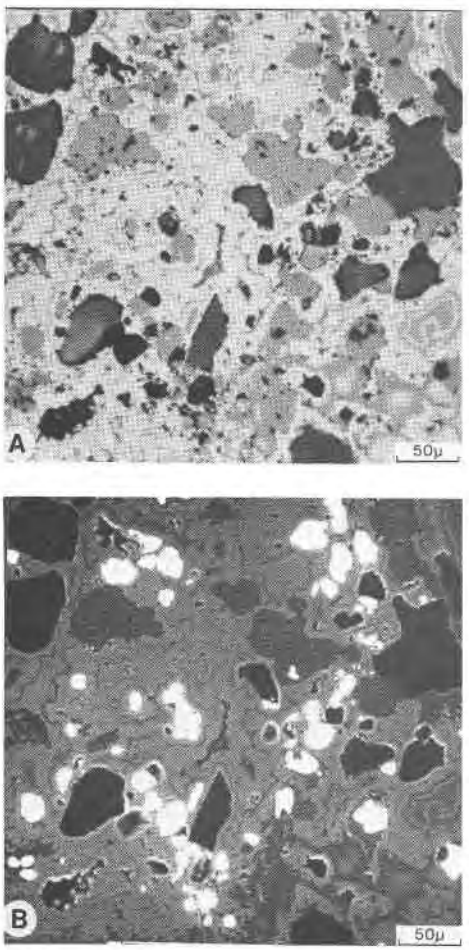

Fig. 7. Contrasting images of gossan sample containing goethite, quartz, and cassiterite, (A) optical microscope, and (B) SEM in BE mode (polished section, uncoated, 30kV). In the optical photomicrograph, quartz and cassiterite (both medium gray) are indistinguishable; the black areas are holes filled with mounting resin, the remainder is goethite (white). In the BE image the cassiterite is white and dramatically differentiated from the quartz (gray).

pumped normally, but now isolated from the specimen chamber except for the standard final beam aperture in the objective lens. This enables a large pressure differential to be maintained between the specimen chamber (at 0.5 to 0.05 torr) and the electron gun chamber (at 10^{-4} to 10^{-5} torr). The pressure in the specimen chamber is controlled by leakage up the column and by regulating with the specimen-port airlock valve if required. Details of these modifications are shown in Figure 3.

Applications

The BE/low-vacuum SEM has a large number of applications in the earth sciences, particularly in mineralogy. The applications rely on the useful high atomic-number contrast images that are obtained, and the ease with which this information can be acquired (Robinson and Robinson, 1978).

The most striking of the many applications to mineralogical problems is in the location of heavy-element minerals in a light-element matrix. Figure 4 shows that even on broken surfaces, the high backscatter-coefficient of the heavy minerals makes the location of such minerals extremely easy. Very small amounts of heavy-element minerals can be found in this way, especially when the TV display is used to image the specimen while it is being moved under the electron beam. The bright images of heavy minerals mean that the user does not have to rely on the recognition of particular mineral shapes, as he would with an SEM operated in the conventional mode.

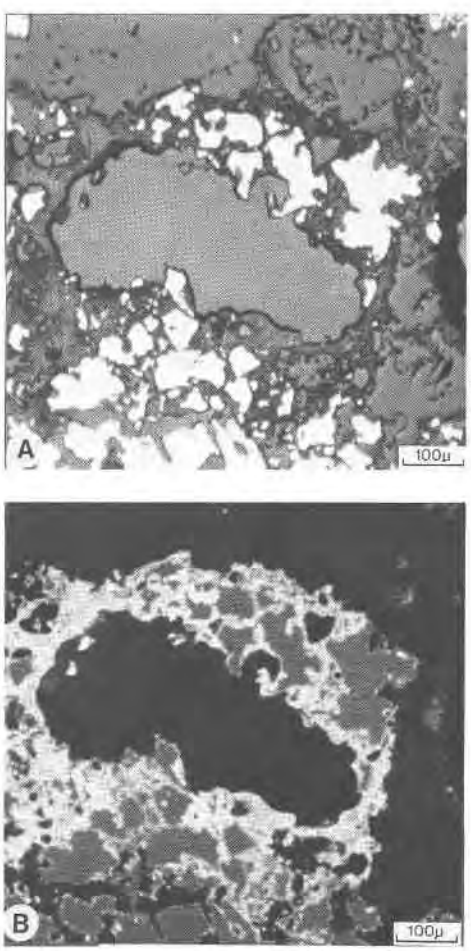

Fig. 8. Contrasting images of gossan sample containing anglesite, quartz, and goethite, as seen through (A) optical microscope, and (B) SEM in BE mode (polished section, uncoated, 30kV). In the optical micrograph, anglesite and quartz are gray, goethite is white; in the BE image, anglesite is white, goethite is gray, and quartz is black.

To find and identify small particles of heavy-element minerals present in low concentrations, or to obtain accurate information on the distribution of the mineral of interest, it is preferable to prepare a flat surface so that significant features (Fig. 5) are not obscured by major surface irregularities. Polished sections are ideal for this purpose. Examples of the dramatic highlighting of heavy minerals by the SEM, in contrast to their appearance in ore-microscopic images, are given by Figures 6–9.

Compositional variations that are difficult or impossible to detect under the optical microscope are often clearly discerned by means of the SEM. In Figure 10, the substitution of iron by manganese in siderite is shown. Figure 11 shows a zoned intergrowth of K-feldspar with a (Na,K) feldspar. In Figure 12, the effect of Sb–Bi substitution is demonstrated (Hudson *et al.,* 1978). Compositional differences as small as those between monoclinic (Fe_7S_8) and hexagonal (Fe_9S_{10}) pyrrhotite can even be detected (Fig. 13).

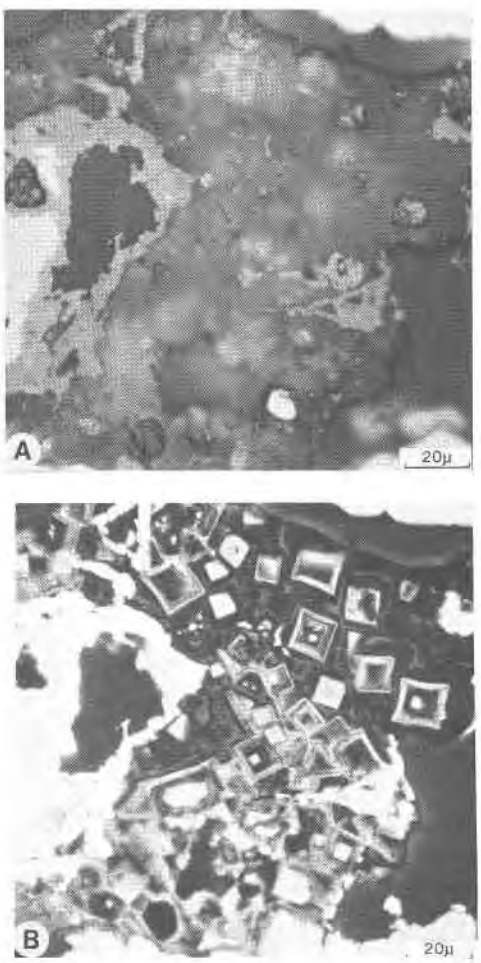

Fig. 9. Contrasting images of supergene assemblage, as seen through (A) optical microscope, and (B) SEM in BE mode (polished section, uncoated, 30kV), showing area of cubic plumbogummite pseudomorphs after galena in a matrix of alunite. The plumbogummite and alunite are virtually indistinguishable in the optical photomicrograph, while strongly differentiated in the SEM micrograph.

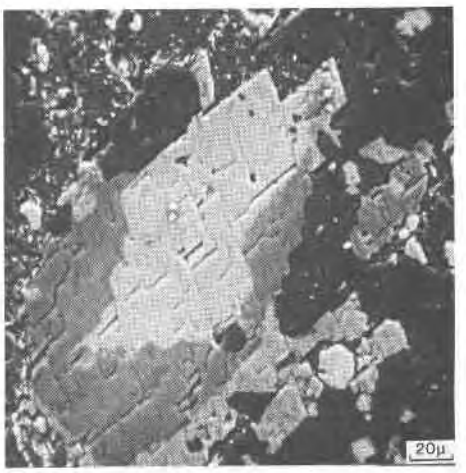

Fig. 10. Siderite (light gray) rimmed by manganoan siderite (dark gray); polished thin section, uncoated, 30kV.

Limitations of the instrument

Although the SEM, as modified, has many advantages over the instrument as it has been used in the past, there are two minor disadvantages that should be mentioned. The first is that the maximum useful magnification is somewhat lower when the specimen is uncoated than when it is gold-coated. This is be-

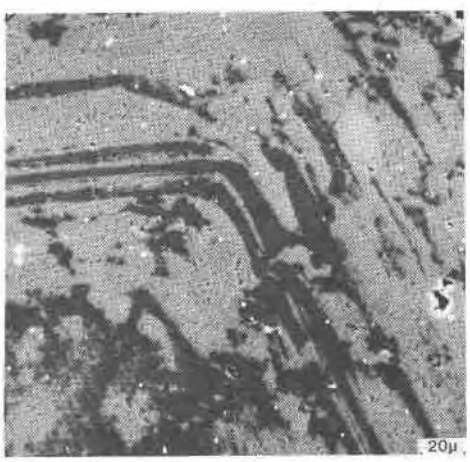

Fig. 11. BE micrograph of roughly polished slab of porphyritic syenite, showing zoned intergrowth of two feldspars; the lighter phase is K-feldspar, the darker, (Na,K)-feldspar. The white particles are inclusions of a variety of minerals, including barite, rutile, and hematite. Uncoated, 30kV.

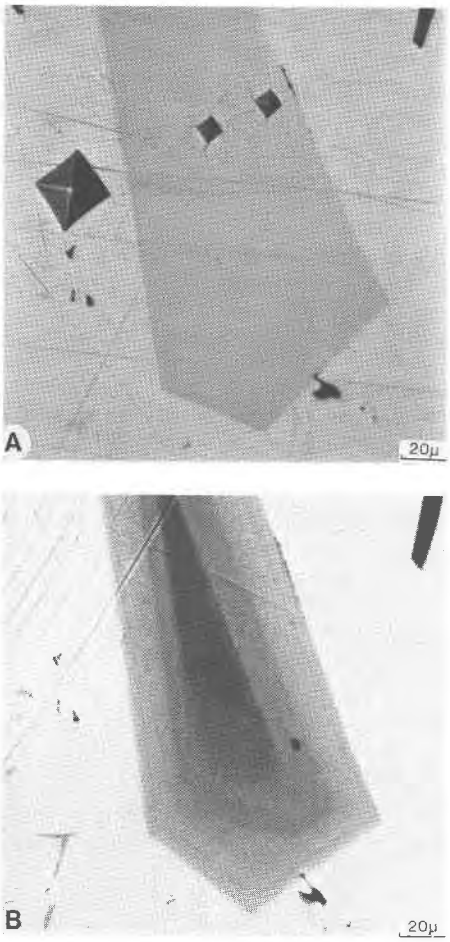

Fig. 12. (A) Optical photomicrograph of michenerite–testibiopalladite grain (medium gray) in altaite (light gray), marked with pyramidal hardness indentations. (B) BE micrograph of same grain, showing pronounced compositional zoning discovered using the SEM. The lighter zones are close to michenerite in composition, the darker to testibiopalladite. Uncoated, 20kV. See Hudson *et al.* (1978).

cause the spatial resolution is a function of specimen density and of average atomic number which is, of course, greater in the gold-coated sample (Robinson, 1975). The maximum magnification at which useful images are obtained with our SEM on average uncoated mineralogical subjects is about 3000×, but with gold-coated samples, similar image quality can be obtained at magnifications almost an order of magnitude greater, as shown in Figure 14. Gold-coating is used only for high-magnification examination of mineral morphology (for clay minerals and asbestos fibers, for example). The gold-coating partially or completely obscures atomic-number differences in the sample and interferes with X-ray analysis, especially for light elements such as sodium and

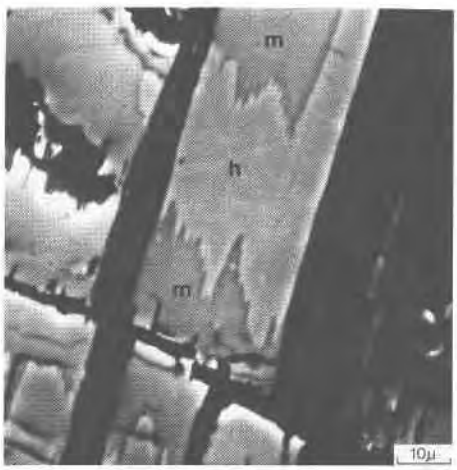

Fig. 13. Intergrowth of monoclinic (m) and hexagonal (h) pyrrhotite; polished section, uncoated, 30kV.

magnesium, so the vast majority of our specimens are examined uncoated.

The second limitation is an increase in the level of X-radiation generated outside the point of interest on the specimen. The relatively high pressure in the specimen chamber increases the scattering of the primary electron beam, and some electrons hit the sample a considerable distance (at least several hundred microns) from the position of the primary beam. These stray electrons generate X-radiation at their point of impact, and this radiation is detected along with radiation from the point of interest. This means, for example, that the X-ray spectrum from a point on a quartz grain in hematite will contain some iron radiation which has, in fact, originated from outside

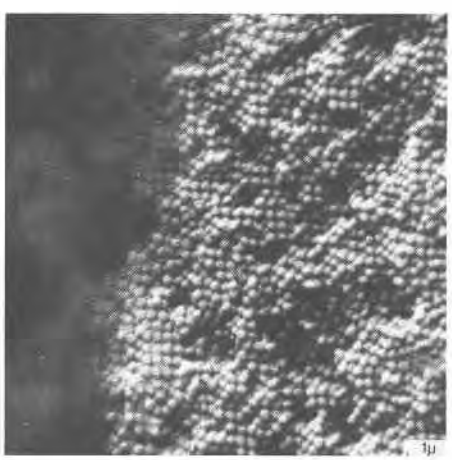

Fig. 14. BE image of an etched opal sample, partially gold-coated. The uncoated section (on the left) shows greatly inferior resolution to that visible in the gold-coated section. 30kV.

the quartz grain. This effect may be minimized, if necessary, by lowering the pressure in the specimen chamber, by using a shorter working distance, and by improving the collimation of the X-ray detector. However, we find that it is quite easy to make allowances for this effect when interpreting the X-ray spectra.

Conclusions

The modified scanning electron microscope has a number of important applications in many aspects of mineralogical investigation, and promises to become an important mineralogical tool. Apart from mineralogical studies, the instrument has also been used in our laboratory for other investigations in metallurgy, archaeology, gemmology, forensic science, and the study of particulate pollutants. Doubtless many more applications will come to light as the SEM in this modified form is used in other laboratories.

Acknowledgments

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