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# The SEM examination of geological samples with a semiconductor backscattered-electron detector: discussion

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## Introduction

Hall and Lloyd (1981) describe the advantages of the semiconductor backscattered-electron (BE) detector used in a scanning electron microscope (SEM) at a normal high vacuum, and they compare that technique with the scintillator backscattered-electron/low-vacuum technique described by us (Robinson and Nickel, 1979). As the former technique is relatively common, we have been able to see it in operation on various SEMs whereas Hall and Lloyd apparently had not had the opportunity to evaluate the low-vacuum technique at first hand before publishing their paper. We would like, briefly, to answer some of the points made by Hall and Lloyd (1981), and especially to disagree with one of their main conclusions, that "carbon-coating is found to be the best method of preventing the specimen from charging" during SEM examination of geological samples.

One of the crucial points in the choice of a method for any geological investigation is its cost-effectiveness. The "low-vacuum" technique has proven remarkably popular with our SEM users because of its ease of use, and because of the rapidity (and hence low cost in terms of investigator time) with which extremely valuable mineralogical information can be obtained (Robinson, B. W., 1980). In this aspect, particularly, we feel it is far more productive than the technique described by Hall and Lloyd (1981).

There is complete agreement between Hall and Lloyd (1981) and ourselves on the value of BE imaging over the more common secondary electron (SE) imaging in SEMs. We feel that the BE signal should be the prime imaging medium for geological use and, in fact, we have not used a SE image since installing a BE detector in 1977. There are a few occasions when SE images have definite advantages over BE images for geological work; these usually involve very high spatial resolution of topographic features, when the edge-enhancement property of SE images is useful. However, in our experience, this aspect is of little interest to most mineralogists.

There are two distinct points under discussion. The first concerns the relative merits of different types of BE detectors, primarily semiconductor, and wide-angle scintillator light-guide photomultiplier types. The latter style of BE detector is often called, for brevity, the Robinson detector after Dr. V. N. E. Robinson, the originator of this type (Robinson, 1975). The second point, a more fundamental issue, is about the advantages and disadvantages of the "low-vacuum" (*i.e.*, no-coating, or environmental-cell) technique.

## BE detector types

The advantages of different BE detector designs are relatively easy to examine, although more difficult to describe in print. The best means of evaluating BE detectors for routine geological use is to put a sample with which one is familiar into a number of SEMs fitted with different BE detectors and to watch the real-time display under normal conditions.

As stated by Hall and Lloyd (1981), for SE detection "practically all SEMs employ the Everhart-Thornley detector . . . . because of its noise-free amplification and good collection efficiency". This is because SEM users and manufacturers alike have found that the Everhart-Thornley scintillator/light-guide/photomultiplier method is the most sensitive and has the lowest noise of available electron detection techniques for the SEM. With the exception of the initial collection stage, the Robinson detector uses the same well-proven method as the Everhart–Thornley detector. The wide-angle collection system makes it possible to collect the majority of backscattered electrons if desired, and at an accelerating voltage above about 20 kV it can surpass a SE detector in output signal (Robinson, 1975, Baumann and Reimer, 1981). It seems to us that the same arguments regarding noise and image quality for scintillator as opposed to semiconductor detector systems apply equally to SE and BE detectors and, until SEM manufacturers change to using semiconductor electron detectors for routine SE detection, scintillator detectors should be generally preferred for SEM electron detection.

There are design constraints that inhibit the construction of large-area semiconductor detectors which can still operate satisfactorily over the full range of SEM scanning speeds (Moll *et al.*, 1978). The low active area of most semiconductor BE detectors requires that in normal operation the sample be relatively close to the detector (to maximize the collection angle, and therefore the signal from the detector). This may increase the difficulty of obtaining X-ray data as it decreases the range of take-off angles available to an X-ray detector. Shorter working distances also increase the care with which samples, especially jagged pieces of rock, must be mounted and transferred to the sample position to avoid hitting the detector. We can vouch for the robustness of the Robinson detector as we know of several, including ours, which bear scars from over-enthusiastic use without ill-effects.

The SEM configuration we use for routine mineralogical investigations consists of the Robinson BE detector, the uncoated sample surface at a nominal 25 mm working distance from the final lens (about 10mm from the detector) and sufficient beam current (about $10^{-9}$ A at 30 kV) to produce high quality true television images and to allow rapid concurrent collection of X-ray data with the energy-dispersive spectrometer. Geologically-oriented users unskilled in SEM techniques have little difficulty in using our SEM with essentially no formal training, as this configuration provides readily understood images and ensures that there is little risk of damage to the SEM or to the BE detector.

In spite of the statements by Hall and Lloyd (1981), we can attest to the facts that Robinson detectors do not degrade significantly with use and that they do not require frequent attention to the coatings. Our current BE detector is uncoated and shows no sign of degradation after almost four years of heavy use. Both these misconceptions probably arise from extrapolation from the properties of the Everhart-Thornley SE detector which focuses the electrons onto a small active area and thus receives a high density electron bombardment. Our Robinson detector, for example, has an active area of more than 1600 $mm^2$ and so should have a much longer lifetime than a SE detector of the same material used under the same beam conditions. The detector is made of a simple plastic scintillator and can be durably aluminum-coated for use in high-vacuum, if required.

Another recent BE detector design which should have applications in the geological use of SEMs is the converted BE detector (Moll *et al.*, 1978, Boyd and Cowham, 1980). It requires a high vacuum, but it does use the proven scintillator-photomultiplier method and may have advantages over the Robinson detector when the specimen chamber is very crowded and where low-vacuum capability is not needed. Electron microprobe analysers and SEMs configured with many accessories are examples where the converted BE detector may be appropriate.

## Low-vacuum *vs.* carbon-coating

We concede that there are times when SEM examination of geological specimens is best done in normal high-vacuum mode. This is, in our view, most likely when SEs need to be used, when quantitative X-ray analysis is required, or when specific X-ray information is sought which would be complicated or invalidated by the potential stray X-radiation generated in low-vacuum mode (Robinson and Nickel, 1979). For example, we would not attempt to run an electron microprobe analyser in low-vacuum mode because of this problem.

Our users have found very little difficulty in making allowance for the stray X-radiation problem. Should it be of concern, most low-vacuum configurations allow for a return to high-vacuum operation within a few minutes, when required. As BE images are far less susceptible than SE images to charging artifacts, it is quite possible that valuable X-ray information can be obtained from uncoated nonconducting samples after switching to high-vacuum mode. Otherwise, the sample can be removed, carbon-coated in the normal way, and returned to the SEM.

For the majority of geological samples we feel the benefits of the low-vacuum mode far outweigh this single disadvantage. The advantages include: (1) the speed and ease of use of the SEM is greatly increased, giving far greater sample throughput; (2) other techniques, such as optical microscopy, can be used after SEM examination of a sample without the need to remove a carbon-coating. Removal of a carbon-coating from a rough specimen and from museum specimens may be impossible or at least very hazardous for the specimen; (3) charging artefacts in the image are completely eliminated; (4) filament life is considerably increased, at least when used with an air-lock sample change; (5) damp, oily or porous samples can be handled without hindrance. Gossan samples, for example, usually outgas for long periods in the SEM vacuum, but do not inconvenience our SEM operation (Nickel, 1981); and (6) there is increased tolerance to operator error and to specimen-chamber leaks.

Our SEM is a 1969 model and so does not provide a useful means of showing the highest resolution attainable in low-vacuum on geological samples. The spatial resolution achieved in the low-vacuum mode with an uncoated sample should be almost the same as that which would be achieved in high vacuum in the same SEM with the sample carbon-coated using the same detector and operating conditions. Moncrieff *et al.* (1979) report data on electron scattering in the SEM relevant to this point. Robinson and Nickel (1979) pointed out that for high spatial resolution of topographic features, better performance is obtained in low-vacuum mode with gold-coated specimens than with uncoated specimens. This is analogous to the improvement shown in high-vacuum mode when changing from carbon-coated to gold-coated specimens.

## Specimen preparation

One of the advantages of the BE/low-vacuum technique is the ease with which valuable mineralogical information can be obtained from normal laboratory specimens, be they rough hand specimens or polished sections. There are a number of benefits of being able to examine rough specimens in the SEM. For instance, preliminary studies can be done quickly to see if further work, perhaps involving the time-consuming preparation of polished surfaces, is warranted. There are also instances where friable fine-grained material is very hard to polish without selectively plucking out some phases which may be of significance. For such samples, the study of a fracture surface ensures that there is much less chance of the phase of interest being removed prior to examination.

Certainly we agree with Hall and Lloyd (1981) that for best atomic-number contrast a polished surface is important. This is because any topographic information present (from surface roughness) will be superimposed in the BE image upon the atomic-number information (from compositional differences). Reducing the topographic contrast by polishing allows much smaller atomic-number differences to be distinguished. For detailed studies of the common rock-forming minerals, the use of polished sections is highly desirable, but for studies of ore minerals and accessory minerals, where the relative atomic-number differences are usually higher, polishing is advantageous but by no means a necessity. The choice of whether to use polished sections, sawn surfaces or fracture surfaces we leave to be decided by the inclinations and aims of the users. It is relevant that the quality of polish needed for SEM work is not usually as high as that needed for reflected-light optical microscopy, because the SEM can image and identify (from the X-ray spectrum) phases within chipped, scratched and unpolished areas. Of course, normal thin sections with coverslips cannot be examined in the SEM.

## Conclusion

In summary, we feel that any SEM intended for geological use should provide the option of use in the BE/low-vacuum mode and that this opinion is best evaluated by seeing the technique in use, then comparing the results and the time taken to achieve them with alternative techniques such as that described by Hall and Lloyd (1981).

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