American Mineralogist, Volume 68, pages 843–844, 1983

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

M. G. Hall and G. E. Lloyd

Center for Materials Science, The University of Birmingham
Elms Road, North Campus, P. O. Box 363, Birmingham B15 2TT, Great Britain

The aim of our paper (Hall and Lloyd, 1981) was to indicate to potential users of SEM back-scattered electron (BSE) microscopy that the more standard techniques of operation involving a high vacuum specimen chamber, a semi-conductor BSE detector and carbon coated samples still remain of considerable value and should not be neglected despite the alternative techniques proposed by Robinson and Nickel (1979). The results presented compare favorably with those of Robinson and Nickel (1979) and it was also shown how BSE methods can be easily extended to provide quantitative information (see Hall and Skinner, 1981); a technique termed "modal analysis" by Barrett *et al.* (1982). However, a critical comparison with the methods of Robinson and Nickel (1979) was not attempted.

In reply to the many discussion points raised by Robinson and Nickel (1983) this note is similarly divided into three sections.

BSE detectors

The early semi-conductor BSE detectors were undoubtedly noisy and had poor bandwidths and signal collection efficiencies. However, recent improvements have resulted in devices with large active areas and low-noise characteristics, some of which may even be capable of operating at TV scan rates (*e.g.*, Gedcke et al., 1978). Although the detector used by Hall and Lloyd (1981) was manufactured some years ago (Stephen *et al.*, 1975) its characteristics are comparable to the recent detectors, having a large active area, low noise level[1], and good frequency response.

In many circumstances, however, SEM image noise does not originate from the detector but is caused by the statistical nature of the signal development and collection process (Gedcke *et al.*, 1978). The semi-conductor used by Hall and Lloyd (1981) has an inherent noise level which is much less than is observed on the image in most practical situations. Moreover, since similar noise levels are also observed for a scintillator-photomultiplier detector (Etp-SEMRA Pty. Ltd. Model No. RBSE-4) using the same range of samples it is concluded that there is very little difference between the noise characteristics of the two detector systems.

There is no doubt that the most sensitive electron collection system for *secondary electrons* is the Everhart–Thornly detector which is a scintillator–photomultiplier device. However, the inference (Robinson and Nickel, 1983) that a scintillator-photomultiplier detector should necessarily be more suitable for back-scattered electrons is largely unfounded and probably based on a natural prejudice. To be efficient, BSE detectors should subtend a large solid angle at the specimen and need to be positioned directly over or close to the specimen. Unfortunately, since scintillator-photomultiplier BSE detectors remove the signal optically they are limited in size by the geometrical requirement to fit the photomultiplier and consequently tend to be rather large and clumsy. SEM operation with short (<10 mm) working distances is therefore precluded and although this may not be significant for atomic number contrast examination, it assumes prime importance for selected area electron channelling patterns (Joy, 1974). Semi-conductor detectors, however, can fit neatly onto the final lens of the microscope and hence do not preclude short working distance operation. X-ray microanalysis may even be performed with the detector in place, thus allowing simultaneous microstructural and compositional analysis.

Low vacuum *vs.* carbon coating

Although BSE images are less susceptible to specimer charging artifacts than secondary electron images, most geological specimens (except perhaps some ore samples such as metal sulphides) charge readily under the influence of the electron beam unless preventative measures are taken. Robinson and Nickel (1979, 1983) use the low vacuum technique and there may be certain circumstances where this approach will be very useful (*e.g.*, for gassy and/or friable samples). However, stray X-ray generation prevents the use of the technique for detailed microanalytical studies particularly where quantitative measurements are required. In such circumstances, and

[1] Equivalent to an incident beam current of a few pa at 30 kV, for a copper test sample at normal working distances. Hall and Skinner (1981) indicate how the X-ray multi-channel analyser may be used to measure both the detector-amplifier noise and SEM image noise.

0003–004X/83/0708–0843$02.00

probably also for electron channelling contrast, a thin, controlled, layer of a conducting medium (*e.g.*, carbon) will be required.

Specimen preparation

Apart from the variation in mean atomic number in the sample, a major influence on the BSE signal is derived from surface topography. Thus as the signal due to atomic number contrast may be confused or overwhelmed by surface topographic effects, only large differences in atomic number can be detected in rough or sawn samples. Robinson and Nickel (1983) argue that careful specimen preparation, which removes surface topography is not necessary, and again this is probably true for ore specimens which show large differences in mean atomic number (*e.g.*, Figure 5 of Robinson and Nickel (1979) exhibits a mean atomic number difference of ~68). However, in general atomic number differences will be much smaller (often <1) and some attention to specimen preparation is therefore required. Usually, the amount of preparation (polishing) needed is no greater than that for optical microscopy but for some samples (*e.g.*, slates) finer polishing may be necessary.

It is also worth mentioning that the other BSE contrast effect due to electron channelling contrast, which is at least as useful as atomic number contrast, is especially susceptible to interference from surface topography and surface damage. Normal mechanical polishing using diamond pastes is no longer suitable and final polishing with a colloidal slurry is usually necessary (Lloyd *et al.*, 1981).

Conclusion

There is no doubt that BSE contrast effects are of considerable value to the geologist and the difference between the approaches of Robinson and Nickel (1979, 1983) and Hall and Lloyd (1981) is mainly one of emphasis. For Robinson and Nickel the desire for speed and the need to examine unprepared, gassy samples dictates their microscopic procedures. We, on the other hand, need to observe the true structure of samples using weak contrast effects and have to consider in more detail the consequences of the specimen preparation and imaging conditions. Both approaches, no doubt, will have their proponents.

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Manuscript received, June 9, 1982;
accepted for publication, December 8, 1982.