8.4 BACKSCATTERED ELECTRON IMAGING OF POLISHED SECTIONS

8.4.1.2 Polishing

![Image of polished cement sample]

Figure 8.15 Example of a well-polished cement sample hydrated for 7 days. The region shown contains very minor defects. (a) The image by BSE; (b) the same image by SE showing small holes, dust particles and a small scratch.

8.4.1.3 Coating

It is very important to keep the sample in the desiccator to avoid carbonation. [...] carbonate crystals formed on the surface (portlandite was transformed into calcium carbonate) of a sample which was not properly stored. If a previously
polished sample is carbonated, it can be prepared once again for observation by polishing at the lowest grain size (usually 1 μm) for about 30 minutes, dried for 48 hours in a desiccator and carbon coated again.

### 8.4.2 General appearance of backscattered electron images

The uses of BSE images were discussed by Scrivener (2004). A major strength of the technique is the wide range of magnifications which can be studied. The lower end of Figure 8.18 shows a composite image of a concrete with a real size of 2 × 2 cm². This image was made by taking 144 images at a nominal magnification of ×200 and then electronically ‘stitching’ them together to give a composite image. In this image it is also possible to see the very high quality of the polishing by the detail of the interfaces between paste and aggregate. Such images are useful for studying concrete, even from real structures, such as in the study of alkali aggregate reaction detailed later. This technique can also be used to study other degradation processes, which involve the ingress of species from the surrounding environment which will create gradients in microstructure at the surface, as also discussed in detail later.

At the other end of the scale Figure 8.19 shows images at a nominal magnification of up to ×20,000. Early-age samples can show ettringite needles or C-S-H fibrils at high magnification (Figure 8.19a). In Figure 8.19b the resolution of a BSE image is compared to that of a fracture surface showing similar features (C-S-H and ettringite needles). Because the space between grains gets filled with time, such features are less apparent at later ages.

SEM has the distinct advantage of allowing the user to focus on particular regions of a sample, e.g. the aggregates in concrete, the inner product) or the outer product regions of C-S-H at a later age and the ITZ in mortars and concretes (Scrivener et al. 2004). A further advantage of BSE imaging is that the contrast is reproducible as shown in the histograms in Figure 8.20a and b and can be used for quantitative analysis of images, as discussed further later.

However, it is important to bear in mind that we still see only 2D images of a three-dimensional (3D) microstructure. As discussed by Scrivener (2004), one should be aware about some bias in 2D sections of 3D microstructures.
Figure 8.18 Concrete sample in a region containing cement paste and small aggregates (Agg).

Figure 8.19 High-magnification images showing ettringite needles and C-S-H in a similar young cement paste sample. (a) BSE image of a polished paste. (b) High-magnification SE image of fracture surface. (Courtesy of Elise Baradier.)
They include the underestimation of the fraction of small particles and overestimation of the thickness of features such as inner product rims of C-S-H. It is also impossible to know the connectivity of the 3D structure in the volume below the observed surface. Attempts have been made to build up 3D images by serial sectioning, either by mechanical polishing (Scrivener 1988) or at very high resolution by focused ion beam (FIB) techniques (Holzer and Münch 2009; Holzer et al. 2004, 2006, 2007; Trtik et al. 2011; Zingg et al. 2008) as presented at the end of this chapter. Unfortunately, even with modern automated FIB techniques, this process is too tedious, expensive and time consuming to be used routinely.

8.4.2.1 Accelerating voltage and resolution

Cementitious materials contain mainly light elements. Consequently, the interaction volume is larger than for heavier materials, such as metals commonly studied in the SEM. Many centrally located SEMs in materials science
departments commonly operate at accelerating voltages of 20–40 kV. At such high accelerating voltages the resolution in BSE images of cementitious materials is far from optimal. In the 1980s one of the authors (Scrivener) found that 15 kV gave a good compromise between reducing the size of the interaction volume while still giving a high enough intensity of electrons. Improvements in microscope performance mean that good BSE contrast can now be obtained at even lower accelerating voltages. However, very low volt-ages will mean that electrons only come from the very near surface, which will usually be damaged from the polishing process, and if BSE imaging is to be combined with study by characteristic X-Ray (Section 8.5), a minimum voltage of about 12 kV is needed to excite iron, which is typically the heaviest element of interest. For general use 15 kV remains a good accelerating voltage.

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