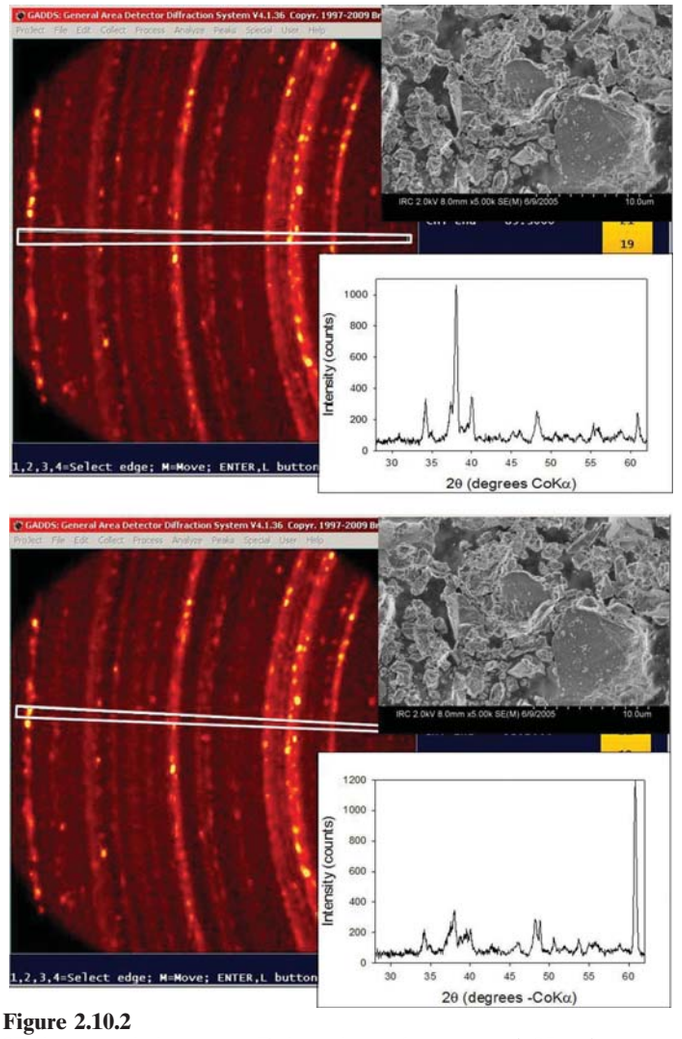


**Figure 2.10.2**
2D images of the spotty Debye rings of a coarse (~35 µm) cement powder using a Co *Kα* radiation 1 mm point source. Overlaid are SEMs of the sample material and integrated patterns from the thin slices indicated in the 2D patterns to illustrate what a point or 1D detector would see. Note: in these 2D data sets the low 2$\theta$ rings are on the right-hand side.

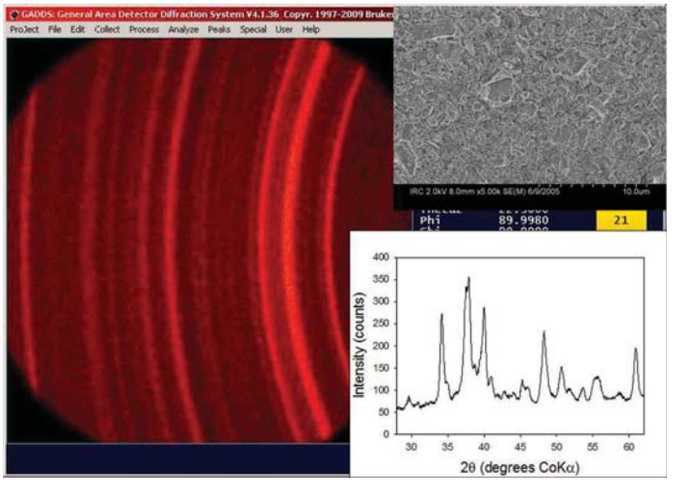


**Figure 2.10.3**
2D image from the same sample after reducing the crystallites down to a few µm, together with the properly averaged integrated data.

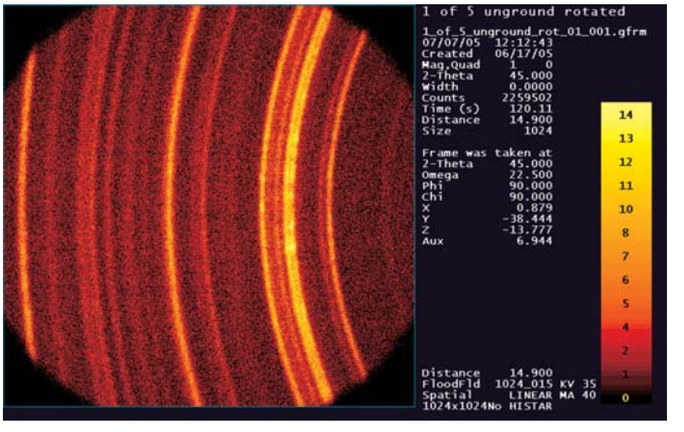


**Figure 2.10.4**
2D image showing the Debye rings when the unmilled sample from Fig. 2.10.2 is rotated. The slight spottiness shows that the quality is not as good as the milled sample, even when not rotated, as shown in Fig. 2.10.3.

typically very small beam divergence, and the tunable wavelength can be very helpful in circumventing some problems.

It will become apparent that many problems relating to specimen preparation and data quality are directly and indirectly the result of samples being too coarse to produce a random powder. The word 'powder' forms part of the name of the technique, but what makes a powder a powder?

### 2.10.1.1. *Powders and particle statistics (granularity)*

The question of when a powder is a 'true' powder is not new. It was dealt with in Klug & Alexander (1954) and Alexander & Klug (1948), and more recently by Smith (Smith, 2001; Buhrke *et al.*, 1998). The short answer is that at least 50 000 crystallites in the illuminated volume are necessary to obtain a random powder pattern.

The classic Debye rings of powder diffraction are formed by the random orientation of a large number of single crystallites, which are either physically separate or part of larger agglomerates. These rings used to be a common sight when film cameras were the norm, but can still be seen where two-dimensional (2D) or area detectors are used, most often on microdiffraction systems or synchrotron beamlines. Where there are sufficient crystallites diffracting, the spots from the crystallites merge into smooth rings. Problems with insufficient crystallites are often indicated by the presence of high-intensity spots in the Debye rings. When using 2D data sets, part or all of the intensity in the Debye rings may be integrated to produce an average 1D powder pattern.

More serious problems can arise in cases where 0D or 1D detectors are used. Most modern laboratory powder diffractometers use some form of 0D point detector (*e.g.* a scintillation counter) or 1D position-sensitive detector (PSD). When collecting data, these detectors pass through the Debye rings along a radius vector. Should the Debye ring be spotty, it is purely down to chance whether the detector will intersect with a spot of higher intensity or low intensity within the ring. An example of how spotty Debye rings can have an adverse effect on the integrated pattern can be seen in Fig. 2.10.2. Unfortunately there is usually no indication of the problem in the resulting integrated 1D pattern. The uncertainty with regard to the intensity of the Bragg reflections is something that must be minimized should accurate relative intensities be required for an analysis. This reproducibility is the concern when the term 'particle statistics' is used in relation to powder diffraction. The desirable smooth Debye rings shown in Fig. 2.10.3 were produced after reducing