McCusker (1988), Cernik *et al.* (1991), Morris, Harrison, Nicol, Wilkinson & Cheetham (1992), and others.

Structures have also been solved using a two-stage method in which the integrated intensities are determined by profile fitting the individual reflections and used in a powder least-squares refinement method (*POWLS*) (Will, Bellotto, Parrish & Hart, 1988). The method was tested with silicon, which gave R(Bragg) 0.7%, and quartz, which gave 1.6%, which is a good test of the high quality of the experimental data and the profile-fitting procedure. Fig. 2.3.2.6 shows Fourier maps of orthorhombic Mg_2GeO_4 calculated using Fourier coefficients taken directly from the profile-fitting intensities.

Other types of powder studies have been carried out successfully. For example, these have been used in anomalous-scattering studies (Will, Masciocchi, Hart & Parrish, 1987; Will, Masciocchi, Parrish & Hart, 1987), Warren–Averbach profile-broadening analysis (Huang, Hart, Parrish & Masciocchi, 1987), studies of texture in thin films (Hart, Parrish & Masciocchi, 1987), and precision lattice-parameter determination (Hart, Cernik, Parrish & Toraya, 1990).

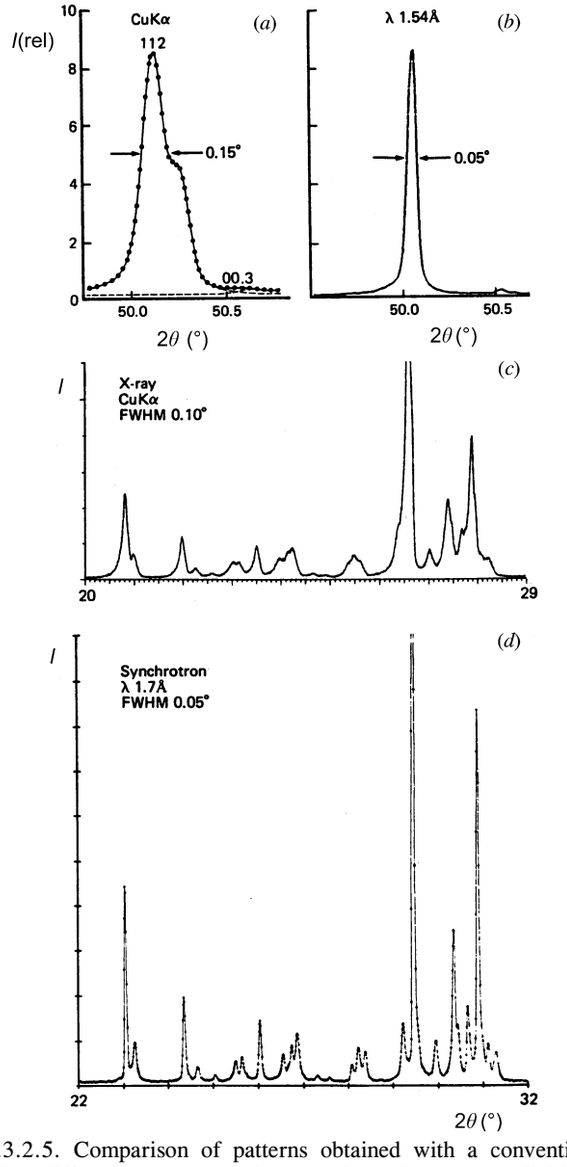

Fig. 2.3.2.5. Comparison of patterns obtained with a conventional focusing diffractometer (*a*) and (*c*), and synchrotron parallel-beam method (*b*) and (*d*). (*a*) and (*b*) quartz powder profiles; (*c*) and (*d*) mixture of equal amounts of quartz, orthoclase, and feldspar.

2.3.2.2. *Cylindrical specimen, 2θ scan*

The flat specimen can be replaced by a thin cylindrical [Fig. 2.3.2.4(*c*)] specimen as used in powder cameras. The powder can be coated on a thin fibre or reactive materials can be forced into a capillary to avoid contact with air. The intensity is lower than for flat specimens because of the smaller beam, and less powder is required. Thompson, Cox & Hastings (1987) used the method to determine the structure of Al_2O_3 by Rietveld refinement. They used a two-crystal incident-beam Si(111) monochromator; the first crystal was flat and the second a cylindrically bent triangular plate for sagittal focusing to form a 4×2 mm beam with spectral bandwidth $\Delta\lambda/\lambda \simeq 10^{-3}$.

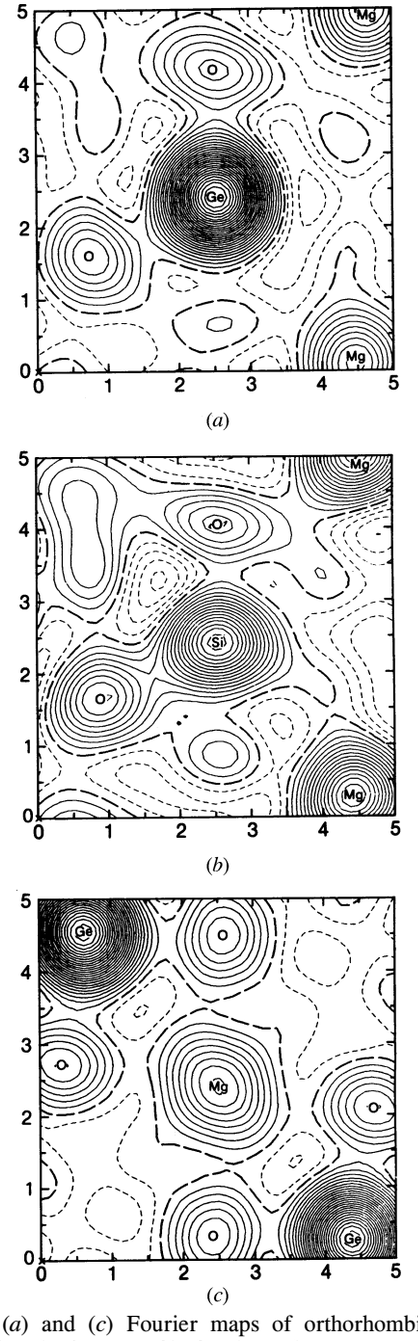

Fig. 2.3.2.6. (*a*) and (*c*) Fourier maps of orthorhombic Mg_2GeO_4 calculated directly from profile-fitted synchrotron powder data. (*b*) Fourier section of isostructural Mg_2SiO_4 calculated from single-crystal data for comparison with (*a*).

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