International Tables for Crystallography (2018). Vol. H, Figure 3.6.1, p. 298.

of a real-space approach with the possibility of working directly with the measured data as typical in reciprocal-space methods.

3.6.3. Examples of WPPM analysis

To illustrate the power of the WPPM approach, a few examples are provided based both on simulated and on real data. The analyses were performed using the *PM2K* software (Leoni *et al.*, 2006). Similar results are obtained using the WPPM implementation in the commercial program *TOPAS*.

3.6.3.1. *Nanocrystalline ceria*

The first example concerns a nanocrystalline ceria powder obtained by the calcination of a cerium isopropoxide gel (Leoni, Di Maggio *et al.*, 2004; Leoni & Scardi, 2004; Scardi *et al.*, 2004). A large amount of XRD and transmisson electron microscopy (TEM) data have been collected on the same system (and specimen), starting from the xerogel and following calcination (Scardi *et al.*, 2010). Fig. 3.6.1(*a*) shows the X-ray powder diffraction pattern of the gel calcined for 1 h at 673 K measured with Cu radiation (40 kV, 45 mA) on a Rigaku PMG/VH diffractometer.

The data were collected over the 2θ range 18–154° (with a step of 0.05°) with a counting time of 60 s per step: a wide angular range and a high signal-to-noise ratio (SNR) are prerequisites for a proper line-profile analysis. The large span in reciprocal space is important for the complete characterization of any anisotropy in the broadening (a large set of independent directions in reciprocal space needs to be sampled), whereas the high SNR guarantees the collection of data at peak tails where the differences between similar microstructure models manifest themselves. The log scale employed in Fig. 3.6.1(*a*) highlights the low level of noise present in the pattern.

The diffractometer had 0.5° divergence and 2° Soller slits mounted on the primary arm and 0.15 mm antiscatter, 0.5° receiving and 2° Soller slits and a curved graphite analyzer crystal mounted on the secondary arm. This setup provided a narrow and symmetrical instrumental profile that could be described by a pseudo-Voigt curve and was thus ideal for line-profile analysis studies. The Caglioti *et al.* (1958) parameterization of the instrumental profile [*cf.* equations (3.6.18) and (3.6.19)] performed on the profiles of the NIST SRM 660a standard (LaB_6) is shown in Fig. 3.6.2.

Analysis of the pattern using traditional methods (see Scardi *et al.*, 2004) required 59 parameters, 53 of which were actually refined:

(i) one unit-cell parameter (a_0),
(ii) six (fixed) parameters defining the instrumental contribution [five parameters for the Caglioti parameterization (U, V, W, a and b) and one for the $K\alpha_2$ intensity ratio],
(iii) three parameters for the background,
(iv) one parameter for the specimen displacement,
(v) 48 parameters for the peaks (intensity, FWHM and shape for 16 peaks).

An analysis using traditional analysis methods resulted in an 'average domain size' of 3.65 (10) nm using the (modified) Warren–Averbach method and in the range from 4.95 (10) to 5.3 (1) nm using a (modified) Williamson–Hall approach. A discussion of the meaning and accuracy of the results can be found in Scardi *et al.* (2004).

The WPPM result, shown in Fig. 3.6.1(*b*), matches the experiment quite well: this is remarkable considering that the whole pattern (1800 data points) is modelled using just 32 parameters (26 free parameters):

(i) one unit-cell parameter (a_0),
(ii) six (fixed) parameters defining the instrumental contribution [five parameters for the Caglioti parameterization (U, V, W, a and b) and one for the $K\alpha_2$ intensity ratio],

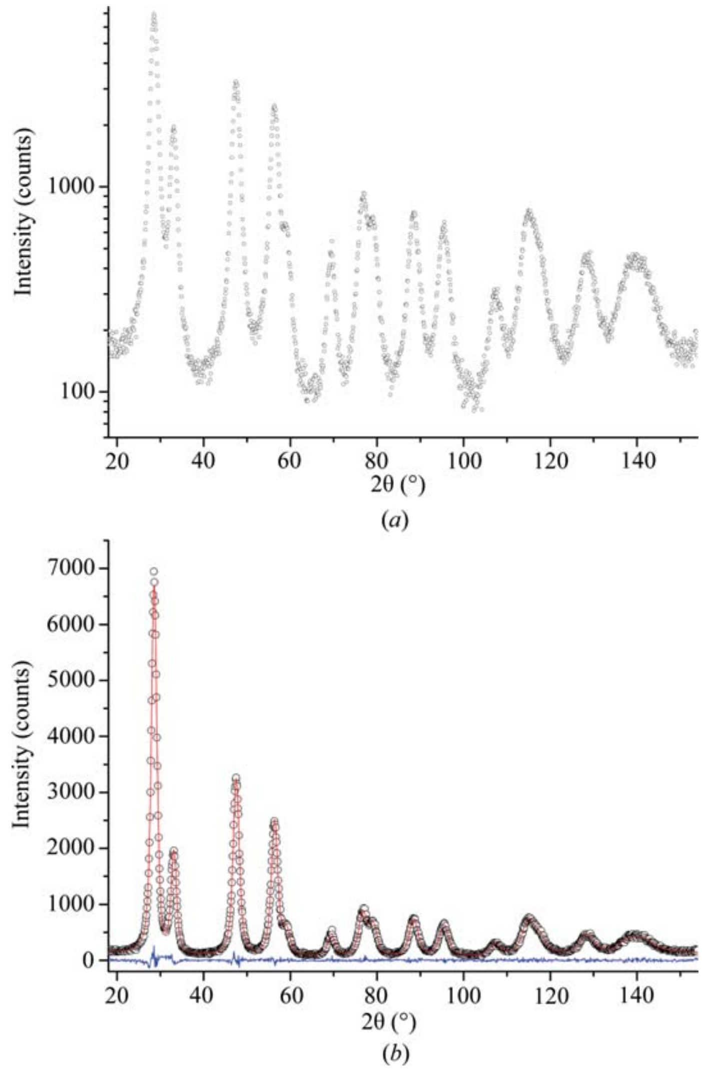

Figure 3.6.1
X-ray powder diffraction pattern of nanocrystalline ceria calcined at 673 K. In (*a*) the pattern is shown on a log scale to highlight the weak features in the data. In (*b*) the results of WPPM are shown: raw data (dots), model (line) and difference (lower line).

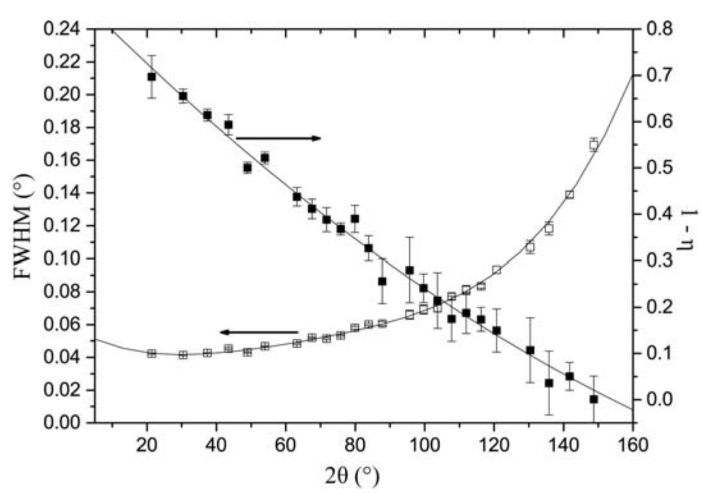

Figure 3.6.2
Parameterization of the instrumental resolution function using a pseudo-Voigt and the relationship of Caglioti *et al.* (1958).

  references