2005; Leoni & Scardi, 2004; Leineweber & Mittemeijer, 2004; van Berkum, 1994; Cheary & Coelho, 1992).

The approach is strictly valid when the broadening sources can be considered as diluted and independent (*i.e.* uncorrelated defects). If this does not apply, then cross-terms should be considered and the whole approach revised. In fact, here we assume that the structure factor can be factored and the lattice is fully periodic in three dimensions: under these conditions, structure (peak intensity) and microstructure (peak shape) can be decoupled as the peak positions can be determined in a straightforward way. Extended defects (*e.g.* faults) cause the appearance of diffuse effects and the displacement of the Bragg peaks: in order to calculate the diffraction pattern, the structure and the microstructure must be simultaneously known (see, for example, Drits & Tchoubar, 1990).

3.6.2.6. *Broadening components*

A brief account is given of the main sources of broadening that can be encountered in practice. An accent will be placed on X-rays, but extension to electrons and neutrons is in most cases straightforward. Concerning electron diffraction, precession data can be used in a straightforward way, whereas for traditional data, containing dynamical effects, further calculations, for example of the intensity, are in principle needed.

3.6.2.6.1. *Instrument*

Each of the components of the diffraction instrument (*i.e.* source, optics, specimen stage, measurement geometry and detector) can have a dramatic impact both on the position and the broadening of the peaks. Axial divergence, for instance, introduces both an asymmetric broadening and an apparent shift of the low-angle peaks. When microstructure (*i.e.* specimen-related effects) is the focus of the analysis, the primary recommendation is to try to limit the instrumental influence. Alternatively, it is preferred to have an instrumental profile (no matter how complex) that can be well described and properly simulated: for instance the profile of an instrument with a $K\alpha_1$ primary monochromator (apparently advantageous) might be hard to model if the $K\alpha_2$ removal is not perfect. This becomes more and more important when the instrumental effects are of the same order of magnitude as the specimen-related broadening.

Two possible paths can be followed when dealing with the instrumental contribution: modelling using the fundamental parameters approach (see, for example, Cheary & Coelho, 1992; Kern & Coelho, 1998) or parameterization of the pattern of an ideal specimen. In the fundamental parameters approach, the geometry of the instrument and the effects of each optical component on the peak profile are described mathematically in 2θ. Most of the formulae for the various optical elements can be found, for example, in the work of Wilson (1963), Klug & Alexander (1974) and Cheary & Coelho (1992, 1994, 1998*a*,*b*). The aberration profiles are folded into the (X-ray) source emission profile (Hölzer *et al.*, 1997; Deutsch *et al.*, 2004) to generate a combined instrumental profile.

When no information on the instrument is available, it is possible to predict the instrumental profile just by using the nominal data for the optical components. It is however advised, whenever possible, to tune the instrumental parameters using the pattern of a line-profile standard [*e.g.* NIST LaB_6 SRM 660(x) series; Cline *et al.*, 2010] showing negligible specimen effects. These instrument-only parameters must then be kept fixed for any subsequent microstructure refinement. It is of paramount importance that all instrumental features are well reproduced when dealing with microstructure effects. Provided that this condition is met, we can therefore employ any arbitrary function to describe the instrumental profile. Thus, as an alternative to FPA, we can either 'learn' the instrumental profile from a standard (Bergmann & Kleeberg, 2001) or use a Voigtian to model it. The Voigtian is particularly convenient as it can be defined directly in L space and thus directly enter the Fourier product of equation (3.6.13).

3.6.2.6.2. *Source emission profile*

For X-rays, the source emission profile at an energy E_l can be well described by a Lorentzian of energy width Γ_l (Hölzer *et al.*, 1997; Deutsch *et al.*, 2004),

$$I_l(E) = \frac{2}{\Gamma_l \pi}\left[1 + 4\left(\frac{E - E_l}{\Gamma_l}\right)^2\right]^{-1}. \qquad (3.6.14)$$

As $\mathrm{d}E/E = \mathrm{d}\lambda/\lambda = \mathrm{d}s/s$, the function can also be represented as a function of s:

$$I^{\mathrm{IP}}_{hkl,l}(s, d^*_{hkl}) = \frac{2}{\pi}\frac{E_l}{d^*_{hkl}\Gamma_l}\left[1 + 4\left(\frac{s_{hkl}}{d^*_{hkl}\Gamma_l/E_l}\right)^2\right]^{-1}. \qquad (3.6.15)$$

For a laboratory tube emitting simultaneously a set of N_λ wavelengths, we have

$$I^{\mathrm{IP}}_{hkl}(s, d^*_{hkl}) = \sum_{l=1}^{N_\lambda} w_l I^{\mathrm{IP}}_{hkl,l}(s, d^*_{hkl}), \qquad (3.6.16)$$

where w_l is the relative intensity of the lth wavelength component (referred, for example, to $w_1 = 1$). The corresponding Fourier transform entering (3.6.13) can be written as

$$\begin{aligned} T^{\mathrm{IP}}(L) &= \sum_{l=1}^{N_\lambda} \exp\left[2\pi i d^*_{hkl}\left(1 - \frac{\Gamma_l}{E_l}\right)L\right]\exp\left(-2\pi s_{hkl}\frac{\Gamma_l}{E_l}L\right) \\ &= \sum_{l=1}^{N_\lambda}\left\{\cos\left[2\pi d^*_{hkl}\left(1 - \frac{\Gamma_l}{E_l}\right)L\right] + i\sin\left[2\pi d^*_{hkl}\left(1 - \frac{\Gamma_l}{E_l}\right)L\right]\right\} \\ &\quad \times \exp\left(-2\pi s_{hkl}\frac{\Gamma_l}{E_l}L\right). \end{aligned} \qquad (3.6.17)$$

The complex term in (3.6.17) accounts for the shift of each emission component with respect to the reference one. For more flexibility (for example to consider the non-ideal behaviour of the instrument), we can use a pseudo-Voigt (pV) in place of the Lorentzian in equation (3.6.14).

3.6.2.6.3. *Optical elements*

The equation of Caglioti *et al.* (1958), modified by Rietveld (1969) and originally developed for constant-wavelength neutron diffraction, is frequently employed for parameterization of the instrumental profile. The FWHM and the pV mixing parameter η (replacing the Lorentzian and Gaussian widths of the Voigt) are then parameterized according to functions in $\tan(\theta)$ and θ, respectively (Caglioti *et al.*, 1958; Leoni *et al.*, 1998; Scardi & Leoni, 1999),

$$\mathrm{FWHM}^2 = U\tan^2\theta + V\tan\theta + W, \qquad (3.6.18)$$

$$\eta = a + b\theta + c\theta^2. \qquad (3.6.19)$$

The parameters of the Fourier transform of a Voigt or pseudo-Voigt are then constrained to those of equations (3.6.18) and