

Outline

1. Summary of the simplified method (double-Voigt approximation) for analyzing microstructure of materials
2. Isotropic cases
3. Anisotropic cases

Live presentation: (Cont. isotropic) Complete creation of an IRF file using the case of spinel MgAl_2O_4

Live presentation: (Anisotropic) Examples of anisotropic cases

Summary: IRF for microstructure analysis

1. Collect a diffraction pattern with a well crystalized sample free of defects (Al_2O_3 , Garnet, LaB_6 , $\text{Na}_2\text{Ca}_3\text{Al}_2\text{F}_{14}$, etc.) for each used conditions of the diffractometer
2. Refine the collected patterns to obtain either the Gaussian and Lorentzian components of each peak (Peak fitting, Res=4, 8) or the instrumental U_{ins} , V_{ins} , W_{ins} , X_{ins} , Y_{ins} (Le Bail fits or Rietveld, Res=1)
3. Use EdPCR to copy the refined parameters in the dialog: Template > Instrumental Resolution File. Save the IRF file.
4. Refine a diffraction pattern of a normal sample using the IRF file putting, for starting the refinement, all the UVWXY parameters equal to zero. Select a microstructural model and refine it.

Observed, instrumental and intrinsic profiles

$$h(x) = g(x) \otimes f(x)$$

GAUSSIAN U, V, W, I_G *LORENTZIAN* X, Y

The parameters U, V, W, I_G, X, Y appearing in the PCR file make reference to the observed profile $h(x)$ **if no IRF file is used** ($U_h, V_h, W_h, I_{hG}, X_h, Y_h$).

The same parameters refer to the intrinsic profile $f(x)$ **if an IRF file is used** (U_f, I_{fG}, X_f, Y_f). Normally $V_f=0, W_f=0$

The IRF file stores the characteristics (U_g, V_g, W_g, X_g, Y_g) of the instrumental profile $g(x)$

Simplified methods for treating the intrinsic profile as implemented in FullProf

Parameters controlling the Full-Width at half maximum (isotropic case)

Isotropic Gaussian Strain

Isotropic Gaussian Size

$$H_{hG}^2 = (U_g + U_f) \tan^2 \theta + V_g \tan \theta + W_g + \frac{I_{fG}}{\cos^2 \theta}$$

Isotropic Lorentzian Strain

Isotropic Lorentzian Size

$$H_{hL} = (X_g + X_f) \tan \theta + \frac{Y_g + Y_f}{\cos \theta}$$

Simplified methods for treating the intrinsic profile as implemented in FullProf

Parameters controlling the Full-Width at half maximum (anisotropic case)

Isotropic Strain Anisotropic Strain Isotropic Size Instrumental

$$H_{hG}^2 = (U_f + (1 - \xi_f)^2 D_{fST}^2(\alpha_D)) \tan^2 \theta + \frac{I_{fG}}{\cos^2 \theta} + H_{gG}^2$$

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Isotropic Strain Anisotropic Strain Isotropic Size Anisotropic Size Instrumental

$$H_{hL} = (X_f + \xi_f D_{fST}(\alpha_D)) \tan \theta + \frac{[Y_f + F_f(\alpha_s)]}{\cos \theta} + H_{gL}$$

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Anisotropic strains

Position of peaks determined by:

$$\{\alpha_i\} = \{A, B, C, D, E, F\}$$

$$M_{hkl} = \frac{1}{d_{hkl}^2} = M(\alpha_i; hkl)$$

$$\frac{1}{d_{hkl}^2} = Ah^2 + Bk^2 + Cl^2 + Dkl + Ehl + Fhk$$

The mean and the variance of the function M_{hkl} are given by :

Average cell parameters

$$\langle M_{hkl} \rangle = M(\langle \alpha_i \rangle; hkl)$$

Spread of peaks (variance) :

$$D_{fST}^2(\alpha_D) \sim$$

$$\sigma^2(M_{hkl}) = \sum_{i,j} C_{ij} \frac{\partial M}{\partial \alpha_i} \frac{\partial M}{\partial \alpha_j}$$

$$\sigma^2(M_{hkl}) = \sum_{\substack{HKL \\ \{H+K+L=4\}}} S_{HKL} h^H k^K l^L$$

$$\alpha_D$$

Anisotropic strain parameters

Anisotropic size

$$H_{sizeL} = k \beta_{size} = \frac{[Y_f + F_f(\mathbf{a}_s)]}{\cos \theta} = \frac{k \lambda}{D_h \cos \theta}$$

Integral breadth (β) and FWHM (H) are related by the constant k

Ellipsoidal shapes of crystallites .

$$\frac{\lambda}{D_h} = Y_f + F_f(\mathbf{a}_s) = k_s d_h^2 (\alpha_1 h^2 + \alpha_2 k^2 + \alpha_3 l^2 + 2\alpha_4 hk + 2\alpha_5 hl + 2\alpha_6 kl)$$

Arbitrary shapes of crystallites can be simulated using spherical harmonics.

$$\frac{\lambda}{D_h} = Y_f + F_f(\mathbf{a}_s) = \sum_{lmp} a_{lmp} P_{lm}(\cos \Theta_h) \begin{cases} \cos m\Phi_h \\ \sin m\Phi_h \end{cases}; \quad p = + / -$$

Volume averaged shape of crystallites is obtained from the refined parameters

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