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Rietveld Refinement: A Technique More than Just Identification

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Abstract

Powder diffraction analysis and the data profile fitting represent the phase identification of a crystalline material which gives information about its unit cell dimensions. In a polycrystalline sample it is inevitable that certain information is lost as a result of the random orientation of the crystallites. So whole Pattern fitting Structure Refinement is now widely accepted to be an exceptionally valuable method for structural analysis of nearly all classes of crystalline materials not available as single crystals. Least squares approach which means manually refining a model to match experimental data can be said as Rietveld analysis which is an extended refinement analysis of a given diffraction data.

Keywords: Diffraction; Unit cell; Refinement; Pattern fitting; Rietveld

Description

Professor Hugo Rietveld first introduced and implemented full profile refinement method in powder diffraction, which became later known as the Rietveld refinement [1,2]. It is a multiparameter curve fitting methodology to refine X-Ray Diffraction results. Its algorithm is that to calculate the entire powder pattern using a variety of refinable parameters and to improve a selection of these parameters by minimizing the weighted sum of the squared differences between the observed and the calculated powder pattern using least squares methods [3,4]. There is much more information hidden in a powder pattern which may be subjected to Rietveld refinement (Figure 1).

Mathematical Expressions

1. The minimization of the weighted sum of the squared differences using least square method is expressed as in eqn. (1) [5]

$$\sum\left(y_i^o - y_i^c\right)^2 \rightarrow Min \quad (1)$$

where

y_i^o →Observed step intensity

y_i^c →Calculated step intensity.

2. Rietveld Refinement: Structure factor is represented as below in eqn. (2)

$$F(hkl) = \sum_i^N f_i \exp\left[2\pi i\left(hx_j + ky_j + lz_j\right)\right] \quad (2)$$

where h, k, l are miller indices [6,7]

3. Rietveld Refinement with Preferred Orientation function: The March Dollase function

$$P_k = \left[G^2\cos^2\alpha_k + \left(1/G\right)\sin^2\alpha_k\right]^{-3/2} \quad (3)$$

where

α_k=angle between (hkl) and preferred orientation vector and [8,9]

$G(\Delta 2\theta_{ik})$: Peak shape function.

Advantages of Rietveld Analysis

1. Quantitative phase analysis,
2. Percentage of crystallinity

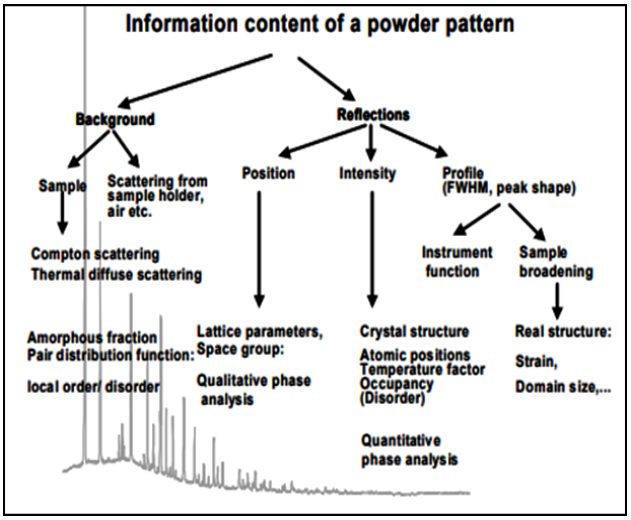

Figure 1: All possible parameters in powder pattern profiling [8].

3. Characterization of Substitutions
4. Unit cell size determination and shape
5. Atomic coordinates/Bond lengths,
6. Micro-strain in crystal lattice-Texture effects [10,11]

Conclusion

In concluding, this analysis is better than mere identification of crystalline materials. It is achieved by incorporating some representative functions that describe certain phenomena in powder diffraction experiments, like errors in 2 theta or broadening of peaks,

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and by fitting other necessary parameters along with varied parameters for processing of full profile analysis using a least-squares procedure, in order to minimize the difference between the calculated and the experimental powder diffraction pattern.

By running Rietveld refinements on diffraction pattern we can get an opportunity to think and learn more about the unknown sample and our model. Though smart guesses/user selected data is required to have a good fit with the data in Rietveld Analysis. So exactly, Rietveld Analysis can be defined as it refines user-selected parameters to minimize the difference between an experimental pattern (Observed data) and a model based on hypothesized crystal structure and instrumental parameters (Calculated Pattern). Small details play an important role in structure analysis using the Rietveld method and attention to these details, though often tedious, is usually rewarded with success.

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