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Powder Diffraction
Theory and Practice

Edited by

Robert E. Dinnebier
Max-Planck Institute for Solid State Research, Stuttgart, Germany

Simon J. L. Billinge
Department of Physics and Astronomy, Michigan State University, USA

RSC Publishing

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CHAPTER 9

Rietveld Refinement

R. B. VON DREELE

IPNS/APS Argonne National Laboratory, Argonne, IL, USA

9.1 INTRODUCTION

A polycrystalline powder can be represented in reciprocal space as a set of nested spherical shells positioned with their centers at the origin[1] (Figure 9.1). These shells arise from the reciprocal lattice points from the myriad (*e.g.* $\approx 10^9\,mm^{-3}$ for 1 μm crystallites) of small crystals, ideally with random orientation, in the sample (Chapter 1). Their magnitude is related to the crystalline structure factors as well as the symmetry driven overlaps (*i.e.* reflection multiplicities) and are affected by systematic effects (*e.g.* Lorentz and polarization, absorption, extinction and preferred orientation). The structure factors and their systematic effects are discussed elsewhere in this volume (Chapter 3). These shells have some thickness or broadening from instrumental effects and the characteristics of the crystalline grains themselves; details are given in Chapters 5, 6 and 13. An experimentally measured powder diffraction pattern is a scan through this suite of shells, which by its nature, is a smooth curve consisting of a sequence of peaks resting upon a slowly varying background.

Techniques for obtaining these data are discussed in Chapter 2.

Early data analysis attempted to extract values of the individual structure factors from peak envelopes and then apply standard single crystal methods to obtain structural information. This approach was severely limited because the relatively broad peaks in a powder pattern resulted in substantial reflection overlap and the number of usable structure factors that could be obtained in this way was very small. Consequently, only very simple crystal structures could be examined by this method. For example, the neutron diffraction study of defects in CaF_2-YF_3 fluorite solid solutions[2] used 20 reflection intensities to determine values for eight structural parameters. To overcome this limitation, H. M. Rietveld[3,4] realized that a neutron powder diffraction pattern is a smooth curve that consists of Gaussian peaks on top of a smooth background

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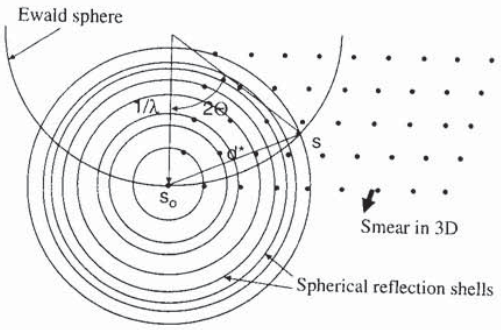

Figure 9.1 Reciprocal space construction for a powder diffraction experiment. The myriad reciprocal lattice points for the crystallites combine to form nested spherical shells centered at the reciprocal space origin.

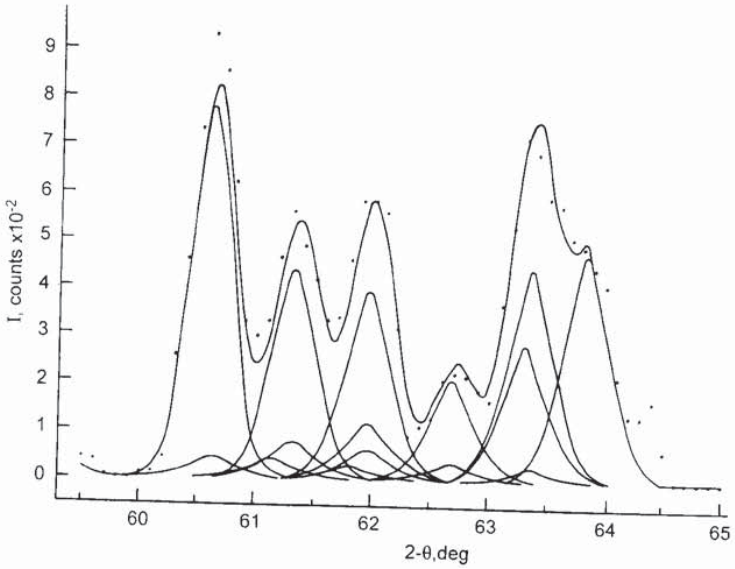

Figure 9.2 Portion of a powder diffraction pattern showing the contributions to the calculated pattern from 15 reflections above a small flat background.

and that the best way of extracting the maximum information from it was to write a mathematical expression to represent the observed intensity at every step in this pattern:

$$Y_c = Y_b + \sum Y_{\mathbf{h}} \tag{1}$$

This expression has both a contribution from the background (Y_b) and each of the Bragg reflections ($Y_{\mathbf{h}}$; $\mathbf{h} = hkl$) that are near the powder pattern step (Figure 9.2).

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