Chapter 2
Crystallite Size Broadening of Diffraction Line Profiles

ABSTRACT

In this chapter, the X-ray peak profile broadening caused by the finite size of scattering crystallites is studied in detail. According to Bertaut's theorem, the line profile with the indices hkl is determined by the length distribution of columns building up the scattering crystallites normal to the hkl reflecting planes. The column length distribution determined from line profiles can be converted into crystallite size distribution. The effect of median and variance of crystallite size distribution on the shape of line profiles is also discussed. The line shapes for different crystallite size distribution functions (e.g. lognormal and York distributions) are given. It is shown that for spherical crystallites the peak broadening does not depend on the indices of reflections. The dependence of line profiles on the indices hkl is presented for various anisotropic shapes of crystallites.

INTRODUCTION

The diffraction peaks are broadened if the perfect order of atoms in a crystalline material is destroyed by lattice distortion (e.g. due to the strain field of crystal lattice defects) and/or by fragmentation of the material into domains with different crystallographic orientations. These diffracting domains are usually referred to as

DOI: 10.4018/978-1-4666-5852-3.ch002

crystallites and the increase of the peak width caused by their small sizes is called "size broadening." The smaller the diameter of the crystallite perpendicular to the reflecting planes, the broader the profile of the reflection. The inverse proportionality between peak width and size of crystallites was explored for small powder particles about a century ago (Scherrer, 1918). Stokes and Wilson (1942) and Bertaut (1950) have imperishable merit in the deep understanding of size broadening of line profiles. X-ray diffraction line broadening is frequently used for the determination of the size of the scattering crystallites (Armstrong & Kalceff, 1999). The popularity of this methodology is based on the very easy sample preparation for X-ray diffraction experiments and the availability of simple formulas for the evaluation of diffraction peaks. Frequently, only the width of a profile is determined and used for the calculation of the average crystallite size. However, the breadth and shape of line profiles depend not only on the mean size, but also on the size distribution and the shape of crystallites (Bertaut, 1950; Louer, Auffredic, Langford, Ciosmak, & Niepce, 1983; Rao & Houska, 1986; Langford, Louer, & Scardi, 2000). Additionally, the separation of the contributions of other effects (e.g. instrumental effect or lattice distortions) to peak breadth is usually very uncertain (Scardi, Leoni, & Delhez, 2004). Therefore, the evaluation of the whole line profile is suggested in order to obtain the crystallite size distribution. Usually, the Fourier transform of the line profile for any source of broadening has a simplier form than the line profile itself. Moreover, the different intensity contributions should be convoluted in order to obtain experimental line profile, while its Fourier transform can be calculated as a simple product of the Fourier transforms of the different component line profiles. Therefore, this chapter provides the derivation of both the intensity profile function and its Fourier transform related to small crystallite size. Special attention is paid to the effect of size distribution and shape of crystallites on the line profile and its Fourier transform. It is noted that the instrumental broadening limits the crystallite size measurable by X-ray line profile analysis. Even for the experimental setups with small instrumental broadening, the detection limit of crystallite size is below 1 µm. It should also be noticed that in many cases the crystallite size determined from peak width differs from the grain or particle size determined by electron microscopy mainly due to the subdivision of grains or particles into subgrains separated by low angle grain boundaries or dislocation walls. In this case, line profile analysis gives the size of subgrains. This effect is discussed in details in the chapter "Practical applications of X-ray line profile analysis" of this book.

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