

Chapter 9

Practical Applications of X-Ray Line Profile Analysis

ABSTRACT

In the previous chapters, the theory and the main methods of diffraction peak profile analysis were presented. Additionally, the specialties in the measurement and the evaluation of line profiles in the cases of thin films and single crystals were discussed. In this chapter, some practical considerations are given in order to facilitate the evaluation of peak profiles and the interpretation of the results obtained by this method. For instance, the procedures for instrumental correction are overviewed. Additionally, how the prevailing dislocation slip systems and twin boundary types in hexagonal polycrystals can be determined from line profiles is shown. Besides the dislocation density, the vacancy concentration can also be obtained by the combination of electrical resistivity, calorimetric, and line profile measurements. The crystallite size and the twin boundary frequency determined by X-ray peak profile analysis are compared with the values obtained by the direct method of transmission electron microscopy. Furthermore, the limits of line profile analysis in the determination of crystallite size and defect densities are given. Finally, short overviews on the results obtained by peak profile analysis for metals, ceramics, and polymers are presented.

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INTRODUCTION

The experimental settings of the X-ray diffractometer used in the measurement of line profiles and the preparation of the data before evaluation (e.g. background subtraction or truncation of peak angular range) influence the results obtained in peak profile analysis (Langford, 1968). For instance, the peak broadening increases with increasing the width of divergence and receiving slits used in the diffraction instrument (Wilson, 1963; Cheary & Cline, 1994). The contribution of instrumental effects to line broadening should be removed from the measured data in order to obtain the peak profiles caused purely by the microstructure. Both a wrong instrumental correction and a subsequent incorrect evaluation of diffraction peaks may result in systematic errors in the obtained parameters of the microstructure. For instance, when the range of the scattering angle, 2θ , for reflection 111 of cold-worked Ni was smaller than five times the full width at half maximum (FWHM), the mean crystallite size determined from the Fourier transform was significantly smaller than the true value (Langford, 1968). The wrong background subtraction also alters the breadth and shape of peaks used in the evaluation procedures and therefore yields deviation from the true values of the crystallite size or the defect densities. Thus, besides the theory of X-ray line profile analysis some practical skills should also be known in order to obtain reliable results in peak profile analysis.

X-ray line profile analysis is an indirect method for the determination of the microstructure, therefore the evaluation of peak profiles and the interpretation of the results should be performed carefully. Usually, a reliable model of the microstructure is necessary for a correct evaluation of the profile shape (Ungár, Gubicza, Ribárik, & Borbély, 2001), which can be constructed with the help of complementary methods, such as transmission electron microscopy (TEM). However, the parameters of the microstructure determined from line profiles often differ from those obtained by other methods (e.g. by TEM), as the various experimental procedures study the microstructure from different aspects (Gubicza & Ungár, 2007; Samadi Khoshkhoo, Scudino, Thomas, Gemming, Wendrock, & Eckert, 2013). Thus, the viewpoint of line profile analysis in investigation of microstructure and the limits of this method in practice should be known. In this chapter useful practical considerations are presented in order to facilitate the evaluation of peak profiles and the interpretation of the obtained results. The microstructure parameters determined by line profile analysis are compared to the values obtained by microscopic methods.

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