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CONVOLUTION BASED PROFILE FITTING IN X-RAY DIFFRACTION: INSTRUMENTAL EFFECTS AND THEIR DESCRIPTION

by

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Due to the use of new X-ray geometries and large scale powder diffractometers such as synchrotron beam lines for the development of industrial materials and quality control, evaluation of XRD data needs to be adopted to these recent developments.
It is the aim not only to obtain structural informations such as the lattice parameters or the crystal structure of a crystalline substance but also to obtain informations about the physical properties of the crystalline materials by measuring the size of the crystallites and the microstrain within the sample. In order to determine the size of the crystallites and the microstrain (real structure) of a crystalline sample a close description of the obtained peak shapes and the instrumental contribution of the observed pattern is necessary.

This work introduces the experimentally-based convolution fitting method, which can be modeled by software modules, based on the Fundamental Parameter Approach (FPA). This FPA is performed with software modules such as TOPAS, developed by Bruker-AXS, which allow to describe the contribution of the instrumental function of physically complicated X-ray paths, such as modern parallel beam optics used in X-ray diffractometers.
The basis of this experimentally-based convolution fitting method is the strict separation of the instrumental contribution, the contribution due to the X-ray source and the sample contribution. This procedure is performed in three steps: (1) The basis of this procedure is a set of well defined aberrational functions which are used to model the instrumental contributions. (.) These aberrational functions are then convoluted over the profile of a well known sample specimen, such as CeO_2, LaB_6 and Y_2O_3, to derive the unique instrumental function. In addition to the sample characteristics, the obtained function also strongly depends on the x-ray optics and wavelength characteristics used. (3.) In order to verify the quality of the data set presented here, the last step involves the comparison of the results to the ongoing round robin test by D. BALZAR [1].
The major advantage of this convolution based approach is (.) the capability of describing a wide range and variety of powder diffraction peak shapes with very high accuracy and (2) its applicability to XRD data which have been derived from both, conventional and synchrotron x-ray sources.

Reference

[1] BALZAR, D. (2000): "Size/ Strain Round Robin"; CPD-Project, http://www.boulder.nist.gov/div853/balzar/s-s_rr.htm.