Modeling of diffraction patterns based on microstructural properties
Ph.D. Thesis written by Gábor Ribarik

(Short summary)

Most of the physical properties of crystalline materials are fundamentally determined by the microstructure. Electron microscopy is an important method for the visualization of the microstructure. The most important fundamental microstructural properties are the type, density and distribution of crystal defects and the grain or crystallite structure. X-ray line profile analysis (XLPA) is one of the most important and powerful alternative methods besides electron microscopy for the characterization of microstructures. This method gives information about the following basic microstructural properties: (i) the size and distribution of crystallites, (ii) the shape anisotropy of crystallites, (iii) the density, character and distribution of dislocations and (iv) the density and type of stacking faults and twin boundaries. This dissertation presents the most important theoretical models of the microstructure according to the literature, my results related to the development of these models, the most important methods of XLPA: the classical ones and some of the most recent methods of literature. The most important part of my work is the development and implementation of new XLPA methods, which are based on the modeling of the physical properties of the different crystal defects. By comparing the so obtained model based line profiles with the measured ones the methods give the microstructural parameters of the investigated materials. By applying these methods the microstructural properties of several different materials or groups of materials are determined and these properties are examined as a function of several mechanical or thermal treatments.

The most important new scientific results of my work are: (i) I have determined the size profile [S1] and the size Fourier transform of coherently scattering domains with spherical or ellipsoidal crystallite shape and lognormal size distribution [S4, S5, S6, S8]. (ii) I elaborated the method for applying the strain profile for XLPA based on the Wilkens model of dislocations and the model of average contrast factors [S4, S6, S14]. (iii) I developed two new methods by using the theoretical size and strain profiles for the extraction of microstructural parameters from X-ray measurements: (a) by fitting simultaneously the whole Fourier transforms or intensity profiles, (the method Multiple Whole Profile Fitting: MWP) [S4, S6], and (b) by the convolutional fitting of the whole diffraction spectrum, (the method of Convolutional Multiple Whole Profile Fitting: CMWP) [S14]. (iv) I worked out a software package for these methods described which is publicly available [S4, S6, S14]. (v) By using the MWP method I have shown that: (a) the dislocation density has the average value of $10^{16} m^{-2}$ in severely deformed Ti, which is in accordance with electron microscopy investigations; and in this deformation stage mainly the slip systems <a> and <c+a> are activated [S11, S12, S22], (b) by applying ball milling and heat treatment of PbS (galena) samples, a systematic set of samples has been produced and by evaluating the X-ray line profiles of these samples using the MWP method a map of microstructure has been obtained. By comparing the microstructural parameters of samples from ancient cosmetics fabricated in the Egyptian Kingdom 3500 b.c. with the parameters of the systematically prepared samples it has been shown that only gentle crushing and no heat treatment or heat treatment at a temperature of less than 300 °C was applied [S9] for the fabrication of the archaeological cosmetics. (vi) By using the MWP method I have shown that: (a) in ball milled Al-Mg alloys the dislocation density as well as the average crystallite size shows a saturation after 2h of ball milling and the results obtained by the MWP and CMWP methods have been compared [S13, S16, S14], (b) in ball milled fluorides (MF_2, M=Ca,Ba,Cd,Sr) the presence of an X-ray optical interference effect can be observed which is present primarily if the average crystallite size of a larger portion of crystallites is about 5-10 nm or smaller [S17]. (vii) I have shown that in nanocrystalline Si_3N_4 particles the size distribution determined either by TEM or XLPA are in excellent agreement, cf. [S1]. In a large number of cases very good correlation was found between the microstructural parameters, especially the subgrain size, and size distribution, the dislocation density and dislocation types determined either by TEM or XLPA [S1–S22].

As the results of this work, a coherent set of methods has been developed which is suitable for the characterization of the microstructure of most different crystalline materials.