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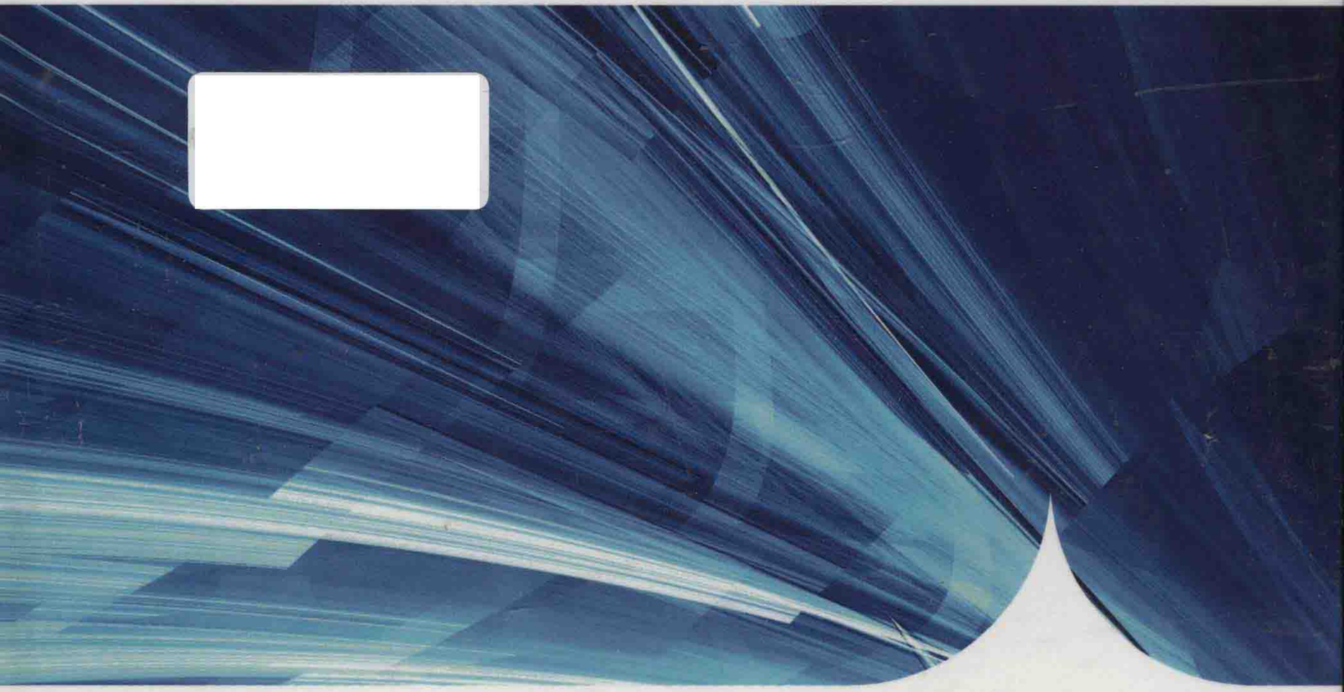

X-Ray Line Profile Analysis in Materials Science

Jenő Gubicza

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X-Ray Line Profile Analysis in Materials Science

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Preface

Crystalline materials are never ideal, perfectly ordered single crystals; rather, they contains lattice defects such as grain boundaries, planar faults, dislocations, disclination, vacancies, and intersticial atoms. The deviation from the perfect single crystalline state is referred to as microstructure of materials. The microstructure is a main factor in determining the physical, chemical, and mechanical properties of materials; therefore, its investigation is unavoidable in materials research. The microstructure of crystalline materials can be investigated by direct methods such as Transmission Electron Microscopy (TEM) or by indirect techniques (e.g. by the analysis of the X-ray diffraction peak shape). Using the microscopy methods, a direct image of the microstructure can be observed; while in the peak shape analysis, only a fingerprint of the microstructure—a diffraction pattern—is obtained, and then the parameters of the defect structure are extracted from the breadth and the shape of the diffraction peak profiles.

X-ray diffraction peak profiles broaden mainly due to the (1) small crystallite size, (2) dislocations, (3) planar defects (e.g. stacking faults and twin boundaries), (4) chemical heterogeneities, and/or (5) surface relaxation in the case of nanoparticles. Some of these effects also cause peak displacement from its ideal position. The procedure of the evaluation of the peak profiles for the microstructure is referred to as X-Ray Line (or Peak) Profile Analysis (XLPA). The term "line" refers to the lines detected on films in powder cameras in the early years of XLPA (in 1920s). These lines are segments of the Debye-Scherrer rings diffracted from powder samples. The scattered intensity distribution in the "line profile" was obtained by scanning the film with microphotometer (Langford, 2004). The phrases "peak profile," "diffraction profile," and "line profile" are used equivalently in the relevant literature.

The history of the method of XLPA started in 1918 when Paul Scherrer related the line breadth to the size of noble metal colloidal particles (Langford, 2004). The equation of Scherrer expresses the inverse proportionality between the crystallite size and the breadth of X-ray diffraction peak profiles (Scherrer, 1918). It should be emphasized that this first application of XLPA occurred only six years after the pioneering experiment of X-ray diffraction performed by Friedrich and Knipping.

The latter researchers detected the interference intensity spots scattered from a copper-sulphate single crystal placed in the path of X-rays. Their experiment was based on the calculation of Max von Laue who determined the conditions necessary for diffraction by X-rays.

In the 1920s, XLPA was mainly restricted to the determination of the size and shape of diffracting crystallites. However, some observed experimental phenomena in XLPA could not be interpreted in terms of the crystallite size and shape; therefore, the method was further developed by incorporating lattice defects into the evaluation. First, the effects of stacking faults in metals (e.g. in Co) and structural defects in ordered alloys (e.g. in Cu_3Au) on line profiles were considered in 1930s (Langford, 2004). The pioneers of this field include, but are not limited to, B. E. Warren, A. J. C. Wilson, and A. R. Stokes (Stokes & Wilson, 1944; Warren, 1959). In the 1940s, the evaluation of X-ray line profiles was improved by E. F. Bertaut, enabling the determination of the crystallite size distribution from the Fourier-transform of the diffraction peak intensity (Bertaut, 1950). Later, the works of J. I. Langford and D. Louer had imperishable merit in the a deeper understanding of size-broadening of peak profiles (Louer, Auffredic, Langford, Ciosmak, & Niepce, 1983; Langford & Louer, 1996).

Between the 1960s and 1990s, several researchers (e.g. M. A. Krivoglaz, K. P. Ryaboshapka, M. Wilkens, T. Ungár, P. Klimanek, R. Kuzel, G. Groma, and A. Borbély) contributed to the development of XLPA in order to determine the type, density, and arrangement of dislocations (Krivoglaz & Ryaboshapka, 1963; Krivoglaz, 1969; Wilkens, 1970; Groma, Ungár, & Wilkens, 1988; Kuzel & Klimanek, 1988; Ungár & Borbély, 1996). Dislocations are important lattice defects in crystalline materials as they play a unique role in plastic deformation, especially in metals and alloys. However, besides dislocations, other lattice defects, such as planar faults, also yield characteristic broadening of X-ray line profiles. Although the basic relationships between the line breadth and the planar fault probability were established in 1950s (Warren, 1959), sophisticated methods for the evaluation of stacking fault and twin boundary probabilities from peak profiles were developed in the 2000s by E. Estevez-Rams, E. Velterop, L. Balogh, and their coworkers (Velterop, Delhez, de Keijser, Mittemeijer, & Reefman, 2000; Estevez-Rams, Leoni, Scardi, Aragon-Fernandez, & Fuess, 2003; Balogh, Ribárik, & Ungár, 2006; Balogh, Tichy, & Ungár, 2009). In addition to lattice defects, the variation of alloying element concentration in a solid solution or an intermetallic compound phase also results in microstrain-like broadening of diffraction peaks. The theory of the effect of chemical heterogeneities on line profiles was elaborated by A. Leineweber, E. Mittemeijer, and R. Delhez, and they have also proposed some procedures for the evaluation of composition distribution from diffraction peaks (Mittemeijer & Delhez, 1980; Leineweber & Mittemeijer, 2004; Leineweber, 2009). The microstrains caused by surface relaxation

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in nanoparticles were introduced into the line profile evaluation by M. Leoni and P. Scardi (Leoni & Scardi, 2004).

In the 2000s, a revolution in XLPA started by elaborating sophisticated diffraction-pattern-fitting methods in which microstructure-based theoretical functions were fitted to the whole experimental diffractogram. An incomplete list of researchers who contributed to this field includes M. Leoni, G. Ribárik, and P. Scardi (Ribárik, Ungár, & Gubicza, 2001; Scardi & Leoni, 2002; Ribárik, 2009). This progress in XLPA was enabled by the increase of the power of computers in terms of memory capacity and calculation speed. Concurrently with the development of the methodology, the instruments (e.g. X-ray sources and detectors) used for the measurement of line profiles were also improved (Levine, Larson, Yang, Kassner, Tischler, Delos-Reyes, Fields, & Liu, 2006). Currently, the parameters of the microstructure in XLPA are usually determined by fitting theoretical functions on the measured peak profiles. These functions are calculated on the basis of a model of the microstructure. For the construction and control of realistic models, simultaneous direct investigations of the microstructure by Transmission Electron Microscopy (TEM) are suggested.

XLPA and TEM may be either alternative or complementary procedures in materials science. Comparing the two methods, the most important advantage of TEM is that the microstructure can be investigated directly, while a model of the microstructure is necessary to evaluate X-ray line profiles. Furthermore, the instrumental broadening in XLPA yields upper and lower detection limits for the crystallite size and the dislocation density, respectively. These limits are 200-800 nm for the crystallite size and 10^{12}-10^{13} m^{-2} for the dislocation density, depending on the instrumental effects. At the same time, XLPA gives the parameters of the defect structure with a better statistics since X-ray diffraction usually examines 10^3-10^5 larger sample volume than that studied by TEM. Additionally, XLPA is a non-destructive method with a much easier sample preparation process, as compared to TEM. Moreover, for materials containing very high densities of lattice defects (e.g. nanostructured materials), XLPA has no competitive methods in the determination of dislocation density in a reliable manner. High-Resolution TEM (HRTEM) can provide the local dislocation density even for its very large values, but with a much worse statistics compared to XLPA. Furthermore, the local dislocation density may strongly deviate from the value averaged for a larger volume. However, in most cases, the methods of XLPA and TEM are suggested to be used to as complementary procedures in the investigation of the microstructure of materials, as they provide different aspects of the microstructure. For instance, in severely deformed metals and alloys, the crystallite size obtained by XLPA corresponds rather to the subgrain size; therefore, its value is 2-10 times smaller than the grain size determined by TEM.

In the last decades, there is a growing demand for the application of XLPA in materials science. This can be explained by the recognition of the effectiveness of this method in studying the defect structure in advanced materials. In the last decade of the second millennium, nanomaterials have become a focal point of materials science due to their unique physical, chemical, and mechanical properties that destine these materials to novel and promising applications. The small dimension of the grains or particles in nanomaterials and their specific processing methods affect their defect structure (vacancies, dislocations, planar defects, and grain boundaries) that has a significant influence on the properties of these materials. The knowledge of the relationships between the production methods, the lattice defects, and the physical properties of nanomaterials is very important not only in order to understand the specific phenomena occurring when the grain size is very small but also from the point of view of practical applications of these materials. XLPA can be used effectively for the study of defect structure in nanomaterials since the high defect density and the small crystallite size in these materials yield large peak broadening, thereby increasing the reliability of the results obtained by this non-destructive method. The large interest in this topic is indicated by the two very successful international training courses on the theory and application of XLPA, which were organized in Eötvös Loránd University, Hungary.

Although, instruments for measuring X-ray line profiles are available in the majority of research laboratories in either the industrial or the academic sector, the "know-how" for determining the microstructure parameters from X-ray diffraction line profiles is often missing. Therefore, this book aims to synthesize the knowledge of the theory, the methodology, and the application of XLPA. The book is written with a very strong theoretical background; at the same time, it presents numerous examples for practical applications. Therefore, the book serves as a basic reference for materials scientists and also a practical guide for engineers. In the first section, the theory of diffraction line broadening caused by the finite crystallite size, the different lattice defects (e.g. dislocations and planar faults), and the chemical heterogeneities is presented. Then, the evaluation methods of line profiles are introduced on the basis of theoretical considerations. Several suggestions for the practical applications of the methods and instruments are also given. The capability and the limits of XLPA in comparison with other methods are presented. The information in this book is organized and presented in the form that is hopefully beneficial for a wide audience: materials scientists, engineers, as well as lecturers and students at universities.

The book gives a comprehensive introduction to XLPA and a coherent overview from the basics to the applications that is missing from the literature of this field. Using this book, the mechanical engineers and the materials scientists can learn how to apply the most sophisticated methods of XLPA in the study of microstructures of materials. Therefore, this book may contribute to the development of novel materials and the better understanding of the correlation between their microstructures and physical/chemical properties. This publication may improve the engineering and materials sciences, as well as all fields connected to materials (e.g. medicine). Additionally, this book is also useful for scholars and their students at universities; therefore, it may promote the education of the next generation of engineers and materials scientists.

ORGANIZATION OF THE BOOK

The book is organized into nine chapters. In the following, a brief description of each chapter is given.

Chapter one reviews the basic concepts and equations of the kinematical X-ray scattering in order to better understand the theory of line profile analysis. The correlation between the crystal structure and the diffracted intensity distribution is shown. The concept of the reciprocal lattice is introduced, and the correspondence between the reciprocal lattice vectors and the lattice planes is also presented. The angles between the directions of the diffracted maxima and the incident beam are calculated from the wavelength of X-rays and the interplanar spacings using the Bragg's law. The Ewald construction is presented for the graphical representation of the condition for the diffraction intensity maxima.

In chapter two, the X-ray peak profile broadening caused by the finite size of scattering crystallites is studied in detail. It is shown that the Fourier transform of the line profile with indices *hkl* at a variable value *L* can be obtained as the volume of the common of the scattering crystallite and its "ghost" shifted by *L* perpendicular to planes (*hkl*). Additionally, it is revealed that the *hkl* line profile is the same as the sum of the intensities scattered incoherently from the columns building up the crystallites normal to the (*hkl*) reflecting planes (Bertaut's theorem). 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, Poisson, 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 (e.g. octahedron, cube, and tetrahedron).

In chapter three, the line shape caused by lattice distortions in a crystal is reviewed. It is revealed that the broadening of a diffraction peak with indices *hkl* is related to the mean-square-strain perpendicular to the reflecting (*hkl*) lattice planes. The strain broadening of line profiles depends on the order of diffraction. The line profiles for a crystal in which the lattice distortions are caused by dislocations are described in detail. It is revealed that due to the anisotropic strain field of dislocations and the elastic anisotropy of crystals, the peak broadening of dislocations strongly depends on the indices of reflections. This effect, referred to as "strain anisotropy," is taken into account by the dislocation contrast factors, which can be determined from the diffraction vector, the Burgers and line vectors of dislocations, and the anisotropic elastic constants of the crystal. It is shown that the stronger the screening of the strain fields of dislocations, the longer the tails in the diffraction profiles. For polarized dislocation walls, the diffraction peak is asymmetric and the antisymmetric component of the profile is determined by the dislocation polarization. It is shown that in the case of nanoparticles with the size of a few nanometers, the lattice distortions caused by surface relaxation give considerable contribution to strain broadening of line profiles.

In chapter four, the shift and the broadening of the diffraction profiles caused by stacking faults and twin boundaries in face centered cubic (fcc) and hexagonal close packed (hcp) crystals are overviewed. It is shown that the intensity distribution around the reciprocal lattice points is broadened only perpendicular to the crystal planes of the faults, forming streaks in the reciprocal lattice. The Bragg reflections consist of subreflections that can be categorized by specific selection rules for the *hkl* indices. The profile shape of all subreflections is of Lorentzian-type. However, the breadth and the position of the subreflections relative to the exact Bragg angle depend on their indices. For instance, if the sum of indices of a subreflection is a multiple of three, neither the position nor the breadth of this peak is influenced by planar faults. Other subreflections are broadened and shifted simultaneously due to intrinsic and extrinsic stacking faults. In the case of twin boundaries in both fcc and hcp crystals, each broadened subreflection is a sum of symmetric and antisymmetric Lorentzian functions. The latter profile component is caused by the interference between the radiations scattered from the parent and twinned lamellae in the crystal. The antisymmetric Lorentzian function yields a shift of the subprofile center. For fcc materials, this displacement of peak position is marginal since twin boundaries are formed on close packed {111} planes; however in hcp crystals, where twinning usually occurs on pyramidal planes, this effect should be taken into account in the line profile evaluation. The full width at half maximum and the position of subreflections can be parameterized as a function of the density and type of planar faults. Although the line profile width strongly depends on *hkl*, the broadening does not increase with diffraction order, suggesting that the effect of faulting is of size character. Intrinsic and extrinsic faults cause opposite shifts of the subreflections with the same *hkl*. For a given type of stacking fault, the peaks with subsequent harmonics of *hkl* indices are shifted in opposite directions. The effect of anti-phase boundaries on line profiles of superstructure reflections for Cu_3Au is also discussed.

Chapter five presents a detailed description of the line broadening caused by chemical heterogeneities. The composition fluctuation in a material results in a variation of the lattice parameter, which yields a diffraction peak broadening. In the calculation of line broadening due to chemical heterogeneities, it is assumed that the composition varies from crystallite to crystallite in a polycrystalline material, but it is uniform inside each crystallite. It is shown that the nature of peak broadening caused by composition heterogeneities resembles a microstrain broadening as in a given direction of the reciprocal lattice the peak width increases proportionally with the length of the diffraction vector. At the same time, the anisotropic properties (i.e. the dependence of the broadening on the direction of the diffraction vector) are different from the strain-broadening caused by dislocations. The anisotropic line-broadening caused by composition fluctuation is parameterized for different crystal systems and incorporated into the evaluation procedures of peak profiles. It is shown that the composition probability distribution function can be determined from the moments of the experimental line profiles using the Edgeworth series. It is also revealed that the concentration fluctuations in decomposed solid solutions can also be determined from the intensity distribution in the splitted diffraction peaks.

In chapter six, the evaluation procedures of X-ray line profiles are overviewed. The following groups of methods are presented in detail: (1) the most simple breadth methods that evaluate only the widths of diffraction peaks (e.g. Scherrer method, classical and modified Williamson-Hall procedures), (2) the methods using the Fourier-transforms of line profiles for the determination of the parameters of microstructures (e.g. classical and modified Warren-Averbach methods), (3) the variance methods evaluating the restricted moments of peaks, and (4) the procedures fitting the whole diffraction pattern (e.g. Whole Powder Pattern Modeling or Convolutional Multiple Whole Profile fitting methods). It is shown that the crystallite size distribution and the densities of lattice defects cannot be determined from the peak width alone as the rule of summation of breadths of size, strain, planar faults, and instrumental profiles depends on their shape. However, the breadth methods can be used for a qualitative assessment of the main origins of line broadening (size, dislocations, planar faults) (e.g. for checking the model of microstructure used in whole powder pattern fitting procedures). The application of Fourier and variance methods is limited if the diffraction peaks are overlapping. In the case of pattern-fitting procedures, usually a microstructure model is needed for the calculation of the theoretical fitting functions. The reliability of these methods increases with increasing the number of fitted peaks.

Chapter seven analyses the special phenomena in XLPA occurring in thin films. It is revealed that for textured nanocrystalline thin films, the crystallite size-broadening of the diffraction peaks increases with the length of the diffraction vector. This effect is explained by the interference of X-rays scattered coherently from adjacent crystallites with close orientations. The partial coherence of adjacent nanocrystallites is caused by the overlapping of their reciprocal lattice points. The smaller the size and the stronger the orientation preference of crystallites, the improved the coherence.

It is shown that this interference effect yields narrowing line profiles at small diffraction angles, while it has no influence on line-broadening at large angles, since the corresponding reciprocal lattice points do not overlap. Therefore, the traditional line profile evaluation methods give much larger crystallite size than the real value and may detect a false microstrain broadening. Some ways for the correction of the interference effect are proposed. If there is no texture in the lateral directions of the thin foil, XLPA can be carried out without taking into account the interference effect by performing the measurements in transmission geometry. Detailed case studies are given for the determination of the defect structure in Cu thin films and Cu-Nb multilayers in the as-processed state and after plastic deformation by rolling.

In chapter eight, the measuring and the evaluation procedures of X-ray line profiles applied on single crystalline materials are overviewed, and their effectiveness is illustrated by representative examples. These procedures are usually different from the methods used for polycrystalline materials. The evaluation of the intensity distribution around the reciprocal lattice points for single crystals provide detailed characterization of the dislocation structure. The broadening along the *hkl* reciprocal lattice vector corresponds to the variation of the lattice spacing for planes (*hkl*) due to the strain fields of dislocations. The intensity distribution perpendicular to the *hkl* reciprocal lattice vector represents a variation of the orientations of lattice planes (*hkl*). From the detected intensity distribution the density, the slip systems and the arrangement of dislocations as well as the lattice misorientation can be determined. It is shown that the average misorientation obtained from rocking curve measurement can be related to the density of geometrically necessary dislocations. It is also revealed that the inhomogeneous distribution of dislocations in plastically deformed single crystals usually results in asymmetric line profiles. The evaluation of these peaks enables the determination of the long-range internal stresses besides the dislocation densities in the dislocation cell walls and interiors.

In chapter nine, 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 sources of instrumental broadening are overviewed and some procedures for instrumental correction of diffraction profiles are presented. Additionally, it is shown how the prevailing dislocation slip systems and twin boundary types in hexagonal polycrystals can be determined from line profiles. It is revealed that 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 TEM. It is shown that for plastically deformed metallic materials, the crystallite size determined by XLPA is usually much smaller than the grain or particle size obtained by TEM or SEM, respectively. This phenomenon is attributed to the fact that the

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crystallites are equivalent to the domains in the microstructure, which scatter X-rays coherently. As the coherency of X-rays breaks even if they are scattered from volumes having quite small misorientation, the crystallite size corresponds rather to the subgrain size in the severely deformed microstructures. In this chapter, the limitations of line profile analysis in the determination of crystallite size and defect densities are also discussed. Finally, the microstructural parameters obtained by XLPA for ultrafine-grained and nanocrystalline metals and ceramics as well as for polymers are overviewed.

REFERENCES

Balogh, L., Ribárik, G., & Ungár, T. (2006). Stacking faults and twin boundaries in fcc crystals determined by x-ray diffraction profile analysis. *Journal of Applied Physics*, *100*, 023512. doi:10.1063/1.2216195

Balogh, L., Tichy, G., & Ungár, T. (2009). Twinning on pyramidal planes in hexagonal close packed crystals determined along with other defects by x-ray line profile analysis. *Journal of Applied Crystallography*, *42*, 580–591. doi:10.1107/S0021889809022936

Bertaut, F. (1950). Raies de Debye-Scherrer et répartition des dimensions des domaines de bragg dans les poudres polycristallines. *Acta Crystallographica*, *3*, 14–18. doi:10.1107/S0365110X50000045

Estevez-Rams, E., Leoni, M., Scardi, P., Aragon-Fernandez, B., & Fuess, H. (2003). On the powder diffraction pattern of crystals with stacking faults. *Philosophical Magazine*, *83*, 4045–4057. doi:10.1080/14786430310001613219

Groma, I., Ungár, T., & Wilkens, M. (1988). Asymmetric x-ray line broadening of plastically deformed crystals: Theory. *Journal of Applied Crystallography*, *21*, 47–54. doi:10.1107/S0021889887009178

Krivoglaz, M. A. (1969). *Theory of x-ray and thermal neutron scattering by real crystals*. New York: Plenum Press.

Krivoglaz, M. A., & Ryaboshapka, K. P. (1963). Theory of x-ray scattering by crystals containing dislocations, screw and edge dislocations randomly distributed throughout the crystal. *The Physics of Metals and Metallography*, *15*, 14–26.

Kuzel, R. Jr, & Klimanek, P. (1988). X-ray diffraction line broadening due to dislocations in non-cubic materials: The case of elastic anisotropy applied to hexagonal crystals. *Journal of Applied Crystallography*, *21*, 363–368. doi:10.1107/S002188988800336X

Langford, J. I. (2004). Line profile analysis: a historical overview. In P. Scardi, & E. J. Mittemeijer (Eds.), *Diffraction analysis of the microstructure of materials* (pp. 413–454). Berlin: Springer Verlag. doi:10.1007/978-3-662-06723-9_1

Langford, J. I., & Louer, D. (1996). Powder diffraction. *Reports on Progress in Physics*, *59*, 131–234. doi:10.1088/0034-4885/59/2/002

Leineweber, A., & Mittemeijer, E. J. (2004). Diffraction line broadening due to lattice-parameter variations caused by a spatially varying scalar variable: Its orientation dependence caused by locally varying nitrogen content in ε-FeN$_{0.433}$. *Journal of Applied Crystallography*, *37*, 123–135. doi:10.1107/S0021889803026906

Leineweber, A. (2009). Description of anisotropically microstrain-broadened line profiles by Edgeworth series. *Zeitschrift fur Kristallographie*, *224*, 432–445.

Leoni, M., & Scardi, P. (2004). Grain surface relaxation effects in powder diffraction. In P. Scardi, & E. J. Mittemeijer (Eds.), *Diffraction analysis of the microstructure of materials* (pp. 413–454). Berlin: Springer Verlag. doi:10.1007/978-3-662-06723-9_16

Levine, L. E., Larson, B. C., Yang, W., Kassner, M. E., Tischler, J. Z., & Delos-Reyes, M. A. et al. (2006). X-ray microbeam measurements of individual dislocation cell elastic strains in deformed single-crystal copper. *Nature Materials*, *5*, 619–622. doi:10.1038/nmat1698 PMID:16845413

Louer, D., Auffredic, J. P., Langford, J. I., Ciosmak, D., & Niepce, J. C. (1983). A precise determination of the shape, size and distribution of size of crystallites in zinc oxide by x-ray line-broadening analysis. *Journal of Applied Crystallography*, *16*, 183–191. doi:10.1107/S0021889883010237

Mittemeijer, E. J., & Delhez, R. (1980). Determination of compositional variations by x-ray diffraction line profile analysis. In S. Block, & C. R. Hubbard (Eds.), *Proceedings of symposium on accuracy in powder diffraction,* (pp. 271-314). Washington, DC: US Department of Commerce.

Ribárik, G. (2009). *Modeling of diffraction patterns based on microstructural properties.* (PhD dissertation). Eotvos Lorand University, Budapest, Hungary.

Ribárik, G., Ungár, T., & Gubicza, J. (2001). MWP-fit: A program for multiple whole profile fitting of diffraction profiles by ab-initio theoretical functions. *Journal of Applied Crystallography*, *34*, 669–676. doi:10.1107/S0021889801011451

Scardi, P., & Leoni, M. (2002). Whole powder pattern modelling. *Acta Crystallographica. Section A, Foundations of Crystallography*, *58*, 190–200. doi:10.1107/S0108767301021298 PMID:11832590

Scherrer, P. (1918). Bestimmung der grösse und der inneren struktur von kolloidteilchen mittels röntgenstrahlen. *Nachrichten von der Königliche Gesellschaft der Wissenschaften zu Göttingen. Mathematisch-Physikalische Klasse*, *1*, 98–100.

Stokes, A. R., & Wilson, A. J. C. (1944). The diffraction of X rays by distorted crystal aggregates – I. *Proceedings of the Physical Society, London*, *56*, 174–181. doi:10.1088/0959-5309/56/3/303

Ungár, T., & Borbély, A. (1996). The effect of dislocation contrast on x-ray line broadening: A new approach to line profile analysis. *Applied Physics Letters*, *69*, 3173–3175. doi:10.1063/1.117951

Velterop, L., Delhez, R., de Keijser, Th. H., Mittemeijer, E. J., & Reefman, D. J. (2000). X-ray diffraction analysis of stacking and twin faults in f.c.c. metals: A revision and allowance for texture and non-uniform fault probabilities. *Journal of Applied Crystallography*, *33*, 296–306. doi:10.1107/S0021889800000133

Warren, B. E. (1959). X-ray studies of deformed metals. *Progress in Metal Physics*, *8*, 147–202. doi:10.1016/0502-8205(59)90015-2

Wilkens, M. (1970). Theoretical aspects of kinematical x-ray diffraction profiles from crystals containing dislocation distributions. In J. A. Simmons, R. de Wit, & R. Bullough (Eds.), *Fundamental aspects of dislocation theory* (Vol. 2, pp. 1195–1221). Washington, DC: National Bureau of Standards.

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