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book reviews

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Thin film analysis by X-ray scattering. By Mario Birkholz, with contributions by P. F. Fewster and C. Genzel. Pp. xxii+356. Weinheim: Wiley-VCH Verlag GmbH Co., 2005. Price (hardcover) EUR 119, SFR 188. ISBN-10: 3-527-31052-5; ISBN-13: 978-3-527-31052-4.

In this book, M. Birkholz encompasses the broad field of X-ray scattering by thin layers. This work includes phase identification, line profile analysis, grazing-incidence scattering, texture and residual stress analyses, and high-resolution X-ray diffraction, described in seven chapters. The treatment of each of these fields becomes progressively specialized, focusing on particular aspects, although the scattering patterns reflect all these effects, often in a combined manner.

Chapter 1, *Principles of X-ray Diffraction*, introduces the basis necessary to gain a first interpretation of diffraction patterns of powders. By introducing the basic phenomenon of interaction of X-rays with matter, the author provides the fundamental knowledge that will be used in the rest of the book, such as the scattered field amplitude, the interference function for crystals, Miller indices and the Bragg equation, crystal systems, *etc*. This opens the way to measurements using θ–2θ scans, for which the intensity of Bragg reflections is derived using the usual atomic form and structure factors, line multiplicity, polarization, absorption and preferred orientation factors, as in classical Rietveld analysis. The chapter includes two 'instrumental panels' on Bragg–Brentano geometry and the generation of X-rays by X-ray tubes, and one 'structure panel' dealing with elementary metals. This chapter ends with a paragraph on the application of some metal layers. This chapter could, by itself, be used for teaching Rietveld analysis by X-rays.

Chapter 2 concentrates on the identification of chemical phases, with initial special emphasis on the use of crystallographic databases in the case of thin films that highlights the deviations currently observed between real sample patterns and models. Then linear and mass absorption coefficients are introduced for the analysis of phase mixtures, including amorphous thin films. A paragraph dedicated to accurate lattice-parameter determination provides an introduction to the usual effects present in diffraction patterns, such as sample transparency, angular and height shifts, axial divergence, *etc*.

In this chapter, an instrumental panel on divergent beam optics aids the understanding of the Bragg–Brentano geometry relative to geometrical effects, and detailed examples of structure-factor calculations are illustrated in two structure panels, for the diamond, sphalerite and wurtzite structures.

The third chapter, on line profile analysis, describes the usual model functions and their integral breadth, before focusing on the instrumental broadening and profile, the latter including the special cases of pure Cauchy and Gaussian profiles. The deconvolution of diffraction profiles by Fourier techniques is then described. In this chapter, line broadening due to only crystallite finite size is described in detail. The approach of Scherrer is developed, before introducing the concept of column-height distribution, which enables the description of crystallite shapes. The determination of crystallite size distribution is also described, using Gauss and lognormal functions.

In a second part of chapter 3, the concomitant occurrence of size and strain broadening is detailed, using the Williamson–Hall and Warren–Averbach techniques. The special case of single-line observation is introduced before the more complete whole-pattern fitting approach. All this is illustrated by examples concerning CuO_2, W–C:H and TiN films. This chapter provides a complete overview of size and microstrain analyses using conventional techniques, as applied to thin films. It also includes an instrumental panel on numerical data analysis, which details reliability factors, and a structure panel which describes crystalline lattice faults. I would have liked to find in this section perhaps a few words about crystal shape and microstrain description using harmonic series, well adapted to whole-pattern fitting.

Chapter 4 deals with grazing-incidence configurations (grazing-incidence X-ray diffraction, GIXRD). Linked to the configurations, the penetration and information depths are introduced in order to demonstrate the depth-dependent probing inherent to GIXRD. X-ray specular reflectivity is described and applied to single and multilayers. Some examples of application are given for Pr_2O_3 and GaAS layers. Since small-angle X-ray scattering (SAXS) generally uses parallel-beam optics, an instrumental panel accordingly describes such a configuration. A structure panel for rutile is included in this chapter. Readers looking for non-specular reflectivity, Yoneda wings or DWBA approaches will perhaps miss something here, but these are more specialized questions, though the classical analyses are well illustrated.

The following chapter is a large chapter on *Texture and Preferred Orientation*. Most of the examples presented in this chapter concern perovskite-related structures, and are summarized in a structure panel. Introducing first the texture factors, the author describes pole figures and their measurement, referring to an instrumental panel on Euler cradles and inverse pole-figure minimum sectors. An illustration of direction, orientations and inverse pole figures follows, as a preamble to orientation distribution function (ODF) determination by spherical harmonics and whole-pattern approaches. In this part, one might notice the absence of ODF refinement by methods other than spherical harmonics. The more restricted 'rocking curve' technique is described and compared with the ODF approach. This chapter is presented in a progressive way, from the more to the less symmetric ODFs and their associated scans. The chapter finishes with a small paragraph on depth-dependent textures, and applications to several films.

Chapter 6, on residual stress analysis, written in collaboration with C. Genzel, starts with the generalization of Hooke's law before introducing the fundamental equation for residual stress analysis, and the associated measured strains by X-ray diffraction. Measurements using the Ψ and Ω modes are described and supported by an instrumental panel on detectors, including CCDs. The concept of diffraction elastic constants is then detailed, followed by a paragraph on grain interaction models. The effect of texture is considered in this chapter and an interesting development on the classification of stresses is presented, which includes multiphase materials, extrinsic and intrinsic stresses. Stress gradients are then considered, as applied to thin-film characterization. This part focuses largely on measurement methods of stress gradients, using asymmetric geometry, grazing incidence and the scattering-vector method. The structure panel on rock-salt structure suits well the presented examples. The absence of both whole-pattern approaches and geometric mean modelling of elastic constants, which are becoming increasingly used, is perhaps regrettable, but this does not detract from the interest of this chapter.

The last chapter, written in collaboration with P. F. Fewster, deals with high-resolution X-ray diffraction, of major interest with respect to strongly epitaxic layered structures. Coherently, a structure panel on epitaxic thin films for electronic applications is presented, together with an instrumental panel on crystals as X-ray beam conditioners. After concerns about the comparison between instrumental resolution and lattice changes, the various strain-inducing mechanisms in epitaxic layers are described. Subsequently, high-resolution rocking curves are detailed and illustrated for Ge-related stacks. Mosaicity and extinctions are introduced for comparison with texture, and to serve the discussion of the dynamical theory of rocking curves. Finally, reciprocal-space mapping and diffuse scattering are briefly illustrated.

The whole makes a very interesting book, providing necessary insights, from basic to more sophisticated analyses. It is an easy-to-read ensemble which does not present confusing details, nor scatters notions that would reduce cohesion. The author has produced a textbook that is genuinely suited to undergraduate scientists, beginners in crystallography, postgraduates and researchers alike. The educational benefits are strongly supported by not less than 101 exercises, fully backed-up by an associated Web server. The materials scientist involved in setting-up, monitoring and analysing X-ray diffraction from thin polycrystalline layers will find here a useful set of tools. To dig deeper, the reader will find a bibliography of 37 monographs and proceedings, and 306 references to articles. The author concentrates on the basis of each discipline without elaborating sophisticated methodologies already developed in specialized literature.

With 356 pages and a nicely constructed index, this book will be of considerable interest to students with the necessary mathematical knowledge, teachers and scientists working in the field of X-ray diffraction and materials science. It should find a place on the bookshelves of every thin-film team.

Daniel Chateigner

Université de Caen Basse-Normandie, Bd. M. Juin, F-14050 Caen, France