Journal of
Applied Crystallography
ISSN 0021-8898

book reviews

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X-ray diffraction by polycrystalline materials. By René Guinebretière. Pp. 351. London: ISTE, 2007. Price (hardcover) EUR 124.83. ISBN 978-1905209217.

In his book, R. Guinebretière covers a wide range of X-ray diffraction applied to polycrystalline materials, from randomly oriented bulks to strongly epitaxial thin films. This work is organized as a very practical textbook, including a historical introduction, with the remaining content divided into two main parts, 'basics' and 'microstructure', organized into four and three chapters, respectively. Though any teacher will find in this book a solid base to help masters students get to grips with X-ray diffraction, one will also find inside much more specialized themes that are not treated in as much detail in the corresponding literature. In this respect, this book certainly deserves the attention of any researcher or PhD confronted with crystallite imperfections, profile analysis or even highly perfect structures best probed by reciprocal space mapping.

The historical introduction deals with the discovery of and first studies using X-rays. We of course find related the usually mentioned first discoveries in this 115 year old field, but in a way that will please anyone interested in the history of science. None of the keynote bibliography covering the first 20 years of X-ray discovery seems to have been missed, and the few figures illustrating these ten pages are really the starting points of any lecture about X-ray scattering.

Chapter 1, *Kinematic and geometric theories of X-ray diffraction*, introduces the main origins of scattering, from single free electron coherent and incoherent scattering, to bound electron and multi-electron atom scattering. Diffraction by an ideally perfect crystal serves as an introduction to a crystallographic description of crystals using direct and reciprocal lattices, which leads to a description of diffracted intensity within the kinematic theory. Classical geometrical theory is then used to illustrate the Bragg, Laue and Ewald descriptions of crystal diffraction. The last part of this chapter focuses on diffraction by ideally imperfect, or size-limited, crystals, and this discussion is naturally followed by an elaboration of diffraction by polycrystalline samples.

All the equations introduced are justified by demonstrations that masters students having a grounding in physics will understand easily, and therefore this chapter makes a readily operational text for a diffraction course. The chapter is illustrated using 18 figures.

Chapter 2 addresses instrumentation for X-ray diffraction. All aspects of the measuring line are treated, from source to detector. X-ray sources are detailed, including classical and high-brilliance sources, X-ray spot geometry, window absorption, and target contamination. Monochromatization of the beam is discussed with particular emphasis on the description of the various types of monochromators and their respective geometries, including multilayers. There follows a description of point, linear, curved and two-dimensional detectors, in their gas or solid versions. The rest of the chapter concentrates on diffractometer design, discriminating between diffractometers designed for bulk and thin-film (eventually strongly textured) sample analyses. We encounter in this part the usual Debye–Scherrer, Seemann–Bohlin, Bragg–Brentano and parallel configurations, using zero-, one- or two-dimensional detectors and various monochromating optics. Particular attention is payed to the study of thin films with respect to penetration depth and irradiated volumes, and to the description of multi-axis diffractometers designed for the study of epitaxial films.

I particularly liked in this chapter the extended description of the modern configurations of diffractometers that are currently used worldwide and that all derive from well known geometries such as Bragg–Brentano. Not many books on diffraction treat these aspects, which, however, are very important for experimentalists and analysts. If *Clean measurements for neat results* had been the title of this chapter, it would not have been constructed differently! An impressive number of nicely drawn figures (70) help the overall understanding of the consequences imposed by such 'geometry variations'.

The third chapter deals with data processing to extract information. Instrumental aberrations taking account of X-ray sources, collimating slits, spectral widths, axial divergence and sample transparency are dealt with here, with a comparison of the various diffractometer geometries in terms of instrumental resolution functions. An introduction to diffraction peak fits using the most usual peak functions and peak-by-peak, whole-pattern or Rietveld analysis is then given. This section does not aim to re-explain Rietveld analysis, which has been described many times, but the author raises in this chapter some points that are essential for an understanding of material covered later in the book (15 figures).

Chapter 4, *Interpreting the results*, covers classical X-ray diffraction phase and structure analyses. Phase identification is treated from a database point of view, before quantitative phase analysis (QPA) is discussed in detail. In this part, problems due to grain statistics and differential absorption are highlighted, together with methods to extract integrated intensities using the appropriate peak fitting technique. QPA techniques using direct, external or internal standard methods are described. Indexing and cell parameter refinement techniques are then introduced, before an introduction to structural analysis. This latter deals with the relationship of electron density to diffraction intensity, and then with the Patterson method and Fourier projections, before applying

this in the case of structural refinement of X-ray diffraction from polycrystalline randomly oriented samples. This chapter contains 14 figures and tables.

The *Microstructural Analysis* part of the book corresponds to an advanced level, perhaps less suited to masters students. It commences with Chapter 5, on scattering and diffraction by imperfect crystals. This chapter describes the effect of the presence of zero-, one-, two- and three-dimensional defects on X-ray diffraction diagrams, in a gradual way. Concerning point defects, the author describes the effects of atomic vacancies associated (or not) with structural relaxations, in the case of monoatomic crystals and with eventual clusterization of vacancies. Linear defects like screw and edge dislocations are then taken into account; this section incorporates a discussion on the contrast factor. Stacking fault planar defects in the X-ray diffraction signal are then introduced. Volume defects (finite crystal sizes and microstrains) are finally modelled, either separately or simultaneously within the Fourier analysis framework. Both specialists and neophytes will appreciate this chapter, containing ten illustrations and all the necessary mathematical developments.

Chapter 6 is on microstructural studies of randomly oriented polycrystalline samples. The first part of this chapter concentrates on the extraction of the sample profile using deconvolution of the instrumental component. The Stokes and iterative methods for profile deconvolution are first detailed, taking account of some stabilization. More recently described techniques (maximum entropy or likelihood, Bayesian, convolutive) are mentioned and briefly described. Microstructural characterization using integral breadth techniques (Williamson–Hall and modified Williamson–Hall) is then exemplified in case studies incorporating anisotropic size and stacking fault determination using peak-by-peak or whole-pattern fitting approaches. Fourier series (Bertaut–Warren–Averbach) and indirect Fourier series (Balzar) techniques are finally thoroughly described. This chapter, illustrated by 18 figures and tables, is essential reading for anyone interested in the characterization of crystal defects.

Chapter 7 is the twin of Chapter 6, although these two chapters have nothing in common except their titles, since working with thin structures presents so many differences in experimental and analytical constraints. The author explicitly sets out the specific requirements needed when working with films, from the positioning and orientation of the sample on the goniometer, to the incorporation of the measurement needs when studying textured films. Rotation transformations using diffractometer angles are first illustrated using matrix notation. Examples of diffraction diagrams are then given for randomly oriented or textured films in the context of showing the effect of incident angle variation. The pole figure concept is introduced in the case of textured films. The determination of epitaxial relationships is the focus of a larger section dealing with the use of φ scans. The rest of the chapter deals with the microstructural determination of epitaxial films. Reciprocal space mapping methodology is introduced in detail, and the effects of microstructural defects on the reciprocal space maps are nicely illustrated. The author targets also the effect of the measurement mode on the maps and the quantitative determination of defects using fitting approaches. 35 figures are incorporated in this chapter.

Overall, this book is a very interesting view on X-ray diffraction. All the necessary insights, from basic to more sophisticated analyses, are included, with a prerequisite in basic crystallography. Only important details are given, and the chapters make a coherent ensemble. The author has achieved an effective textbook, as useful to students and people without expertise in diffractometry as it will be to PhDs and researchers. It is also a ready-to-use support for teachers in this field. Furthermore, the materials scientist needing to set up, monitor and analyse X-ray diffraction from bulk or thin layers will find here a useful set of tools. Around 420 references will help the reader in getting further.

At 351 pages, with few preface and historic pages, this book does not resemble others in the same field. It shows the differences in the experimental equipment needed to study thin films compared with bulk studies, and I have not seen such an extensive review of modern diffractometers anywhere else. The microstructural aspects are undoubtedly the major part of the work, from randomly oriented polycrystalline samples to strongly epitaxial films.

Daniel Chateigner

CRISMAT–ENSICAEN, Boulevard Maréchal Juin, F-14050 Caen Cedex, France