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Transmission Electron Microscopy and Diffractometry of Materials

Fourth Edition

Springer

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ISSN 1868-4513
ISSN 1868-4521 (electronic)
Graduate Texts in Physics
ISBN 978-3-642-29760-1
ISBN 978-3-642-29761-8 (eBook)
DOI 10.1007/978-3-642-29761-8
Springer Heidelberg New York Dordrecht London

Library of Congress Control Number: 2012949821

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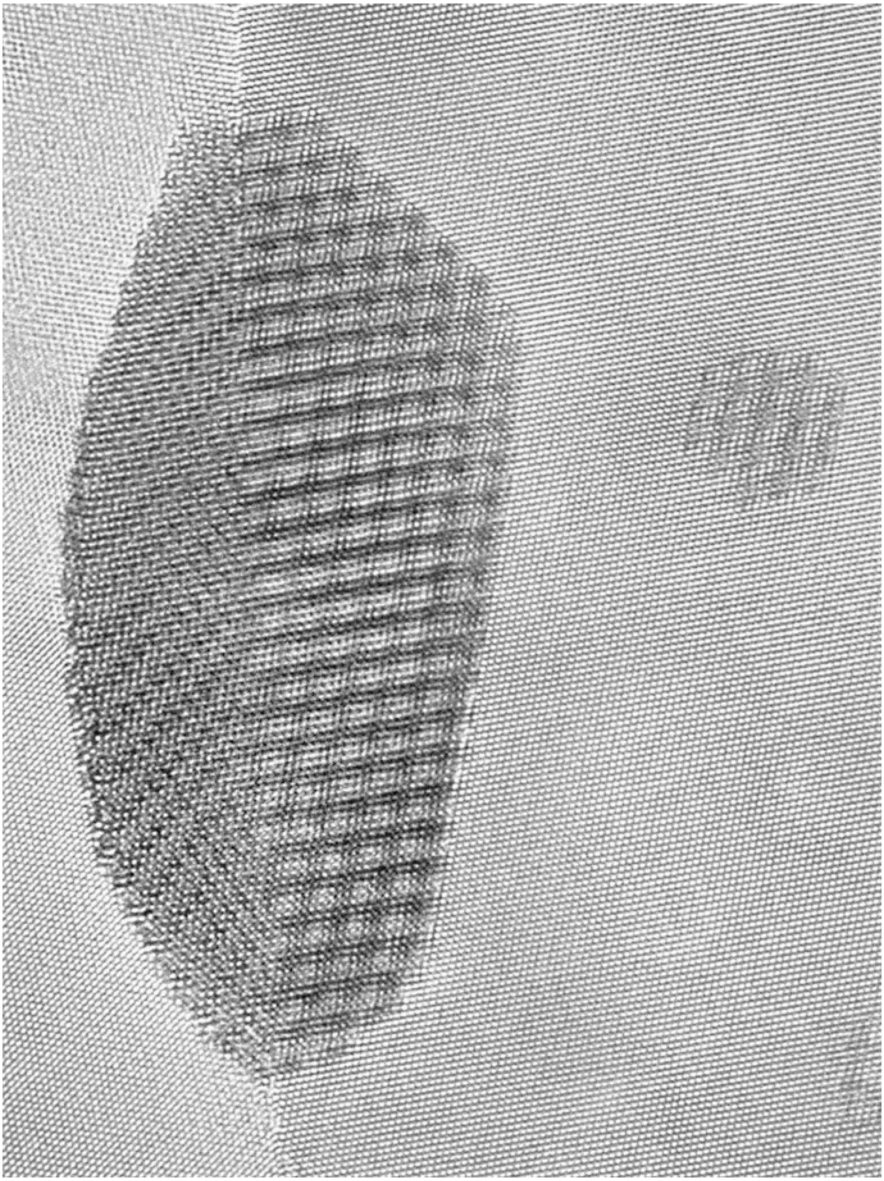

High resolution transmission electron microscope (HRTEM) image of a lead crystal between two crystals of aluminum (i.e., a Pb precipitate at a grain boundary in Al). The two crystals of Al have different orientations, evident from their different patterns of atom columns. Note the commensurate atom matching of the Pb crystal with the Al crystal at right, and incommensurate atom matching at left. An isolated Pb precipitate is seen to the right. The HRTEM method is the topic of Chap. 11. Image courtesy of U. Dahmen, National Center for Electron Microscopy, Berkeley

This book is dedicated to our students—past,
present and future.

Preface

Aims and Scope of the Book This textbook was written for advanced undergraduate students and beginning graduate students with backgrounds in physical science. Its goal is to acquaint them, as quickly as possible, with the central concepts and some details of transmission electron microscopy (TEM) and x-ray diffractometry (XRD) that are important for the characterization of materials. The topics in this book are developed to a level appropriate for most modern materials research with TEM and XRD. The content of this book also provides the fundamental preparation needed for further study of advanced topics in scattering, diffraction, or microscopy. The book includes many practical details and examples, but it does not cover some topics important for laboratory work such as specimen preparation methods for TEM.

The methods of diffraction and microscopy have made rapid advances since this book was first published in the year 2001, driven in part by the growth of nanoscience and materials science. For TEM, a remarkable recent development has been a practical corrector for the spherical aberration of the objective lens. Image resolution below 1 Å can be achieved regularly now, and the energy resolution of electron spectrometry has improved dramatically. Locating and identifying individual atoms inside materials has been transformed from a dream of fifty years into experimental methods of today.

The entire field of x-ray spectrometry and diffractometry has benefited from advances in semiconductor detector technology. Another development has been the formation of a large community of scientists who are regular users of synchrotron x-ray facilities. Powerful new sources of neutrons have elevated the field of neutron scattering research. Increasingly, the most modern instrumentation for materials research with beams of x-rays, neutrons, and electrons is becoming available through an international science infrastructure of user facilities that grant access on the basis of scientific merit.

This fourth edition offers new content on recent advances in transmission electron microscopy including tomography and strain analysis. Neutron scattering is the subject of its own chapter that gives a general introduction to concepts of phase, energy and scattering factor. After publication of the third edition, many explanations and problems were sharpened to cover more clearly the fundamentals of scattering, diffractometry and microscopy, which are the core of the book.

Beneath the details of principle and practice lies a larger goal bringing out common themes of how waves and wavefunctions interact with matter, while highlighting the special features of x-rays, electrons, and neutrons. Coherence and wave interference are conceptually similar for both x-ray waves and electron wavefunctions. In probing the structure of materials, periodic waves and wavefunctions share concepts of the reciprocal lattice, crystallography, and effects of disorder. Besides efficiency in teaching, a further benefit of an integrated treatment is breadth—it builds strength to apply Fourier transforms and convolutions to examples from both TEM and XRD.

Content The first three chapters give general descriptions of scattering, diffraction, imaging, and instrumentation for XRD, TEM, and neutron scattering. This is followed in Chaps. 4 and 5 by electron and x-ray interactions with atoms. The atomic form factor for elastic scattering, and especially the cross sections for inelastic electron scattering, are covered with more depth than needed to understand Chaps. 6–8, which emphasize diffraction, crystallography, and diffraction contrast. In a course oriented towards diffraction and microscopy, it is possible to defer much of Chaps. 4 and 5 for advanced study.

The core of the book develops kinematical diffraction theory in the Laue formulation to treat diffraction phenomena from crystalline materials with increasing amounts of disorder. The phase-amplitude diagram is used heavily in Chap. 8 for the analysis of diffraction contrast in TEM images of defects. After a treatment of diffraction lineshapes in Chap. 9, the Patterson function is used in Chap. 10 to treat short-range order phenomena, thermal diffuse scattering, and amorphous materials. High-resolution TEM imaging and image simulation follow in Chap. 11, and many of the modern microscopy methods are presented in Chap. 12. The essentials of the dynamical theory of electron diffraction are presented in Chap. 13.

With a discussion of the effective extinction length and the effective deviation parameter from dynamical diffraction, we extend the kinematical theory as far as it can go for electron diffraction. We believe this approach is the right one for a textbook because kinematical theory provides a clean consistency between diffraction and the structure of materials. The phase-amplitude diagram, for example, is a practical device for interpreting defect contrast, and is a handy conceptual tool even when working in the laboratory or sketching new ideas. Furthermore, expertise with Fourier transforms is valuable outside the fields of diffraction and microscopy. Fourier transforms are mentioned early in the text, but their manipulations become more serious in Chaps. 5, 6 and 8. Chapter 9 presents convolutions, and the Patterson function is presented in Chap. 10. The student is advised to become comfortable with Fourier transforms at this level before reading Chaps. 11–13 on HRTEM and dynamical theory. The mathematical level is necessarily higher for HRTEM and dynamical theory, which are grounded in the quantum mechanics of the electron wavefunction.

Teaching This textbook evolved from a set of notes for the one-quarter course MS/APh 122 *Diffraction Theory and Applications*, offered to graduate students and advanced undergraduates at the California Institute of Technology, and notes for the one-semester graduate courses MSE 703 *Transmission Electron Microscopy* and MSE 706 *Advanced TEM*, at the University of Virginia. Most of the students in these courses were specializing in materials science or applied physics, and had some background in elementary crystallography and wave mechanics.

The choice of topics, depth, and speed of coverage are matters for the taste and discretion of the instructor, but to help with the selection of course content, the authors have indicated with an asterisk, "*," those sections of a more specialized nature. The double dagger, "‡," warns of sections containing a higher level of mathematics, physics, or crystallography. Each chapter includes several, sometimes many, problems to illustrate principles. The text for some of these problems includes explanations of phenomena that seemed too specialized for inclusion in the text itself. An online manual of worked solutions, many with insightful background information, is available. Access to this manual is offered only to course instructors, who are encouraged to contact the authors. (We apologize to ambitious students that we cannot grant access to you.)

When choosing the level of presentation for a concept, the authors faced the conflict of balancing rigor and thoroughness against clarity and conciseness. Our general guideline was to avoid direct citations of rules, but instead provide explanations of the underlying physical concepts. The mathematical derivations are usually presented in steps of equal height, and we try to highlight the central tricks even if this means reviewing elementary concepts. The authors are indebted to our former students for identifying explanations and calculations that needed clarification or correction.

It is not realistic to cover the entire content of this textbook in one academic quarter, or even one semester, so we offer these suggestions for using it. For a materials physics course that covers microscopy and the scattering of waves from materials, one of the authors follows the sequence of Chaps. 1, 2, 3.1–3.4, 4, 5.1–5.4, 5.6, 6, 7.1–7.3, 8, 9, 10.1–10.3, 11.1, 11.2, 12.1, 12.2, 13.1, 13.5. With a weekly problem set and a weekly TEM laboratory, this content is covered in a 10 week academic quarter. Exercises for an introductory laboratory course are presented in Appendix A.12.

For a one-semester course on TEM, the other author covers Sects. 1.1, 1.2, 2.1–2.8, 4.1, 4.3, 5.1–5.7, 6.1–6.9, 7.1–7.5, 8.1–8.14. This course includes an accompanying lab following Appendix A.12, except that the MoO_3 rotation calibration is replaced with exercises to correct the objective lens astigmatism.

Acknowledgements We are grateful for the advice and comments of Drs. C.C. Ahn, D.H. Pearson, H. Frase, U. Kriplani, N.R. Good, C.E. Krill, Profs. L. Anthony, L. Nagel, M. Sarikaya, and the help of P.S. Albertson with manuscript preparation. N.R. Good and J. Graetz performed much of the mathematical typesetting, and we are indebted to them for their careful work. Prof. P. Rez suggested an approach to treat dynamical diffraction in a unified manner, and he and Prof. A. Minor provided valuable advice on the new content for later editions. Updates to the solutions manual include excellent contributions from Profs. R. Gronsky and

O.A. Graeve. We thank our physics editor Dr. C. Ascheron of Springer-Verlag for his help. Finally, we thank the National Science Foundation for supporting our research efforts in microscopy and diffraction.

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