Electron Microdiffraction

Electron Microdiffraction

J. C. H. Spence and J. M. Zuo

*Arizona State University
Tempe, Arizona*

Springer Science+Business Media, LLC

Library of Congress Cataloging-in-Publication Data

Spence, John C. H.

Electron microdiffraction / J.C.H. Spence and J.M. Zuo.

p. cm.

Includes bibliographical references and index.

ISBN 978-1-4899-2355-4

1. Thin films--Optical properties. 2. Crystal optics.
3. Electrons--Diffraction. 4. Solid state physics. 5. Solid state
chemistry. I. Zuo, J. M. II. Title.

QC176.64.O7S65 1992

530.4'275--dc20

92-33963

CIP

ISBN 978-1-4899-2355-4

ISBN 978-1-4899-2353-0

DOI 10.1007/978-1-4899-2353-0

© Springer Science+Business Media New York 1992

Originally published by Plenum Press, New York in 1992

Softcover reprint of the hardcover 1st edition 1992

All rights reserved

No part of this book may be reproduced, stored in a retrieval system, or transmitted in any form or by any means, electronic, mechanical, photocopying, microfilming, recording, or otherwise, without written permission from the Publisher

Large-angle convergent beam pattern recorded at 100 kV from silicon in the [111] orientation. The Omega filter of the Zeiss 912 has been used to remove most inelastic scattering, together with a very small selected area aperture, resulting in high contrast. (Courtesy of Dr. J. Mayer, M.P.I., Stuttgart, Germany)

Preface

Much of this book was written during a sabbatical visit by J.C.H.S. to the Max Planck Institute in Stuttgart during 1991. We are therefore grateful to Professors M. Ruhle and A. Seeger for acting as hosts during this time, and to the Alexander von Humbolt Foundation for the Senior Scientist Award which made this visit possible. The Ph.D. work of one of us (J.M.Z.) has also provided much of the background for the book, together with our recent papers with various collaborators.

Of these, perhaps the most important stimulus to our work on convergent-beam electron diffraction resulted from a visit to the National Science Foundation's Electron Microscopy Facility at Arizona State University by Professor R. Høier in 1988, and from a return visit to Trondheim by J.C.H.S. in 1990. We are therefore particularly grateful to Professor Høier and his students and co-workers for their encouragement and collaboration. At ASU, we owe a particular debt of gratitude to Professor M. O'Keeffe for his encouragement. The depth of his understanding of crystal structures and his role as passionate skeptic have frequently been invaluable. Professor John Cowley has also been an invaluable sounding board for ideas, and was responsible for much of the experimental and theoretical work on coherent nanodiffraction. The sections on this topic derive mainly from collaborations by J.C.H.S. with him in the seventies. Apart from that, we have tried to review the literature as impartially as possibly and, at the same time, bring out the underlying concepts in a clear and unified manner, so that the book will be useful for graduate students. We are particularly grateful to Dr. J. A. Eades for his critical review of Chapter 7 and to Dr. J. Mayer. We apologize to those authors whose work may have been overlooked among the many hundreds of papers. In order to make the book more practically useful, we have included some FORTRAN source listings, together with POSTSCRIPT code, which allows the direct printing of Kikuchi and HOLZ line patterns on modern laser printers from the programs. Support from NSF award DMR-9015867 ("Electron Crystallography") and the facilities of the NSF-ASU National Center for High Resolution Electron Microscopy is gratefully acknowledged.

Contents

1. A Brief History of Electron Microdiffraction	1
2. The Geometry of CBED Patterns	7
2.1. One-Dimensional (Systematics) CBED	9
2.2. Two-Dimensional CBED	15
2.3. The Geometry of HOLZ and Kikuchi Lines (Kinematic)	20
3. Theory	29
3.1. Electron and X-Ray Structure Factors. Temperature Factors	30
3.2. Many-Beam Theory	35
3.3. Two-Beam Theory and the Bethe Potentials. How Bragg's Law May Fail If More Than Two Beams Are Excited	40
3.4. The Concept of the Dispersion Surface	44
3.5. Degenerate and Nondegenerate Perturbation Theory	48
3.6. Three-Beam Theory and Particular Solutions for Centrosymmetric Crystals	52
3.7. Three-Beam Theory—Noncentrosymmetric Crystals and the Phase Problem	56
3.8. Dynamic HOLZ Intensities and Positions. Dispersion Surfaces for HOLZ Lines. How the Bragg Law Depends on Local Composition	63
3.9. Absorption and Its Effects	75
3.10. Channeling, Bound States, and Atomic Strings	82
4. The Measurement of Low-Order Structure Factors and Thickness	85

4.1.	Two-Beam Methods for Thickness Determination	86
4.2.	Automated Dynamical Least-Squares Refinement Methods	88
4.3.	Error Propagation in Refinement by CBED	98
4.4.	Measurement of Debye–Waller Factors by CBED	100
4.5.	What Is Measured. Charge Density and the Total Crystal Energy	101
4.6.	The Significance of the Mean Inner Potential V_0	104
5.	Applications of Three- and Many-Beam Theory	107
5.1.	Finding Atom Positions. Structure-Factor Phase Measurement. Enantiamorphs	107
5.2.	Critical Voltages	120
5.3.	The Intersecting Kikuchi-Line and HOLZ-Line Methods	123
5.4.	Local Measurement of Strains, Composition, and Accelerating Voltage Using HOLZ Lines	125
5.5.	Polarity Determination in Acentric Crystals	131
5.6.	The Inversion Problem	134
6.	Large-Angle Methods	137
6.1.	Large-Angle Methods for the ZOLZ	137
6.2.	Large-Angle Methods for the HOLZ	143
7.	Symmetry Determination	145
7.1.	Point Groups	148
7.2.	Bravais Lattice Determination	158
7.3.	Space Groups	160
7.4.	Phase Identification	166
8.	Coherent Nanoprobes. STEM. Defects and Amorphous Materials	169
8.1.	Subnanometer Probes, Coherence, and the Best Focus for CBED	170
8.2.	Lattice Imaging in STEM Using Overlapping CBED Orders	177
8.3.	CBED from Defects—Coherent Nanoprobes. Site Symmetry, Atom Positions	181

8.4. Ronchigrams, Point Projection Shadow Lattice Images, and Holograms	191
8.5. CBED from Defects—Incoherent. Dislocations and Planar Faults. Multilayers. CBIM	201
8.6. Microdiffraction from Amorphous Materials	210
9. Instrumentation and Experimental Technique	213
9.1. Instrumentation	214
9.2. Experimental Technique	217
9.3. Detectors and Energy Filters	219
9.4. Electron Sources	233
References	245
Appendix 1. Useful Relationships in Dynamical Theory	255
Appendix 2. Electron Wavelengths, Physical Constants, etc.	257
Appendix 3. Crystallographic Data	259
A3.1. The Reciprocal Lattice	259
A3.2. The Seven Crystal Systems	260
A3.3. Interplanar Spacings	261
A3.4. Extinction Conditions Resulting from Screw and Glide Symmetry	261
A3.5. Symmetries in Zone-Axis CBED Patterns	263
A3.6. Height H of HOLZ Planes for Zone (u, v, w)	265
A3.7. The Use of a Metric Matrix for Crystallographic Calculations	265
Appendix 4. Indexed Diffraction Patterns with HOLZ	267
Appendix 5. Computer Programs	277
A5.1. Plotting HOLZ Lines	277

A5.2. Bloch-wave Dynamical Programs	296
A5.3. Multislice Programs	308
 Appendix 6. Crystal Structure Data	 323
 Appendix 7. A Bibliography of CBED Applications Indexed by Material	 329
 References for the Appendixes	 353
 Index	 355

Symbols, Units, Sign Conventions, and Terminology Used in This Book

TERMINOLOGY

The terms *convergent-beam electron diffraction* (CBED) and *microdiffraction* are used interchangeably throughout the book, according to current usage. Both refer to the electron diffraction patterns which are formed when an electron source is focused onto a thin sample. “Coherent CBED” and “incoherent CBED” refer specifically to the degree of coherence in the illumination aperture. (In the first case the probe is necessarily coherent. In the second it is partially coherent. An incoherent probe is impossible in practice.) Roughly speaking, the coherent case is usually obtained only on field-emission electron microscopes.

A recommendation for future usage might be that the coherent case be known as nanodiffraction and the incoherent case as CBED. Both are examples of electron microdiffraction. CBED patterns are also known as Kossel–Möllenstedt patterns. A Kossel pattern is an incoherent CBED pattern with overlapping orders. A Kikuchi pattern is generated by inelastic scattering (often covering a range greater than the Bragg angle) from within a crystal. A ronchigram is a coherent CBED pattern with overlapping orders (often formed with the illumination aperture removed entirely), and is usually obtained from rather thin crystals. LACBED means large-angle convergent-beam electron diffraction, and refers to incoherent CBED patterns in which special techniques have been used to prevent overlap of the orders, so that an angular view of an order may be obtained which is larger than the Bragg angle. The terms “sample,” “crystal,” “specimen,” and “foil” are used synonymously throughout the book. The uncrystallographic terms “horizontal” and “vertical” have sometimes been used for brevity, and are defined by the direction of the electron beam.

We adopt the crystallographic conventions for planes and directions, thus (hkl) denotes a set of parallel crystal planes, $\{hkl\}$ a family of planes equivalent by symmetry, $[hkl]$ a direction, and $\langle hkl \rangle$ a set of equivalent directions.

SIGN CONVENTIONS

A plane wave is taken to have the form $\exp(+2\pi i \mathbf{K} \cdot \mathbf{r})$ throughout this book, in accordance with the most widespread usage in quantum mechanics texts and in the dynamical electron diffraction literature. (Since inelastic scattering is not considered, the time dependence of the wavefunction is suppressed.) This convention is the opposite to that used throughout the *International Tables for Crystallography*, in X-ray crystallography generally, in Cowley (1981b), Spence (1988a), and in many of the multislice multiple electron scattering programs. The choice of signs thus presents an unavoidable dilemma in electron crystallography, as discussed in Saxton *et al.* (1983) and Spence (1988a). Our choice means that our formulas for structure factors [such as Eq. (3.3)] are unconventional. [The minus signs in Eqs. (3.3) and (3.8) would be replaced by plus signs in X-ray crystallography.] Broadly speaking, reversing the sign of the phase of the incident plane wave requires a change of sign in the exponential kernel of the Fourier transform between real and reciprocal space, which has the effect of conjugating all structure factors. For historical reasons, the Bloch-wave program listed in Appendix 5.2 uses the crystallographic convention, $\exp(-2\pi i \mathbf{K} \cdot \mathbf{r})$.

For the determination of structure-factor phase invariants, either convention, if used consistently, leads to the same conclusions. Thus, for example, the use of an incident plane wave $\exp(-2\pi i \mathbf{K} \cdot \mathbf{r})$ would reverse the sign of every term in the three-phase invariant given in Eq. (3.59). As shown at the end of Section 5.1, a compensating sign change elsewhere then results in the same predicted intensity for all crystals. (Two crystals which are enantiomorphous pairs, however, differ only in the sign of the phase invariant, and can therefore be distinguished using CBED intensity measurements.)

Interferometry and holography experiments (of which three-beam dynamical scattering is an example) always measure magnitudes of phase differences, which are again independent of the sign of the phase of the incident wave.

Section 5.1 describes the “absolute” determination of individual structure-factor phases in BeO, for an origin taken on the Be site. Reversing the sign of the phase of the incident wave would reverse the

sign of these phases, and this must be considered (together with the choice of origin) when comparing this work with results from other researchers.

UNITS

SI units are used throughout, except where stated. Angstrom ($\text{\AA}$), centimeter (cm), and esu units are also given occasionally for convenience. Electron scattering factors are specified in both the old (Born approximation) system in units of angstroms and the new SI system in units of volt meter³ (V m^3). See Section 3.1 for more details.

SYMBOLS

In the old system, based on the Born approximation and given in the *International Tables for Crystallography* before 1989, electron structure factors F_g^B were defined in terms of atomic scattering factors f_i^B by

$$F_g^B = \sum_j f_j^B(s) \exp(-2\pi i \mathbf{g} \cdot \mathbf{r}_j) \quad \text{in angstroms}$$

In the new system (*International Tables* since 1989) SI units have been used, and the structure factors made a property of the material alone, independent of accelerating voltage or scattering theory, so that

$$F_g = \sum_i f_i(s) \exp(-2\pi i \mathbf{g} \cdot \mathbf{r}_i) \quad \text{in volt meter}^3$$

where

$$f_i(s) = \int V_i(\mathbf{r}) \exp(-4\pi i \mathbf{s} \cdot \mathbf{r}) d\tau$$

Here $V(\mathbf{r})$ is the atomic potential (in volts), and $2\mathbf{s} = \mathbf{u} = \mathbf{K}' - \mathbf{K}$ with $|\mathbf{u}| = 2 \sin \theta / \lambda$. In addition $\mathbf{K}$ and $\mathbf{K}'$ are the incident and elastically scattered electron wavevectors, and θ is half the total scattering angle Θ . The symbol $f_i^e(s)$ has also been used for the new electron atomic scattering factor.

Fourier transforms are defined with signs such that, for example, the (positive) crystal potential is

$$V(\mathbf{r}) = \sum_{\mathbf{g}} V_{\mathbf{g}} \exp(-2\pi i \mathbf{g} \cdot \mathbf{r})$$

in volts, therefore

$$V_g = \frac{F_g}{\Omega} \quad \text{in volts}$$

where Ω is the volume of the unit cell in meter³.

The old system is related to the new by

$$f_j^B(\mathbf{s}) = \left(\frac{2\pi m_0 |\mathbf{e}|}{h^2} \right) f_j(\mathbf{s})$$

Here $|\mathbf{s}| = \sin \theta / \lambda \approx \Theta / 2\lambda$ is half the scattering vector, as used in the *International Tables for Crystallography*. Scattering factors are expressed in terms of $f(\mathbf{s})$ rather than $f(u)$.

B	$= 8\pi^2 \langle u^2 \rangle$ The temperature factor is $\exp(-Bs^2)$ for amplitudes
λ	Relativistic electron wavelength $= hc/W\beta$
$\mathbf{K}_0$	Incident electron wavevector in vacuum, $ \mathbf{K}_0 = 1/\lambda$
$\mathbf{K}'_0$	Scattered wavevector
$\mathbf{K}$	Wavevector corrected for mean inner potential, $\mathbf{K}^2 = \mathbf{K}_0^2 + U_0$
$\mathbf{k}^{(j)}$	Labeling wavevector for Bloch wave j inside the crystal
S_g	Excitation error, positive if $\mathbf{g}$ is inside the Ewald sphere
θ_c	Incident beam divergence, the semiangle subtended by the effective source at the sample
θ_B	The Bragg angle
Θ	Total scattering angle, the angle between $\mathbf{K}'$ and $\mathbf{K}$, twice θ and θ_{Bragg}
$\mathbf{g}$	Reciprocal lattice vector, $ \mathbf{g} = 1/d_{hkl}$
W	Total relativistic energy of an electron, $W = mc^2 = \gamma m_0 c^2$
m	Relativistic mass of an electron $= \gamma m_0 = \gamma m_e$
σ	Interaction constant $= 2\pi m e \lambda / h^2$
U_g	Dynamical structure factor $= \sigma V_g / \lambda \pi = \gamma F_g^B / \pi \Omega$ in units of length ⁻² . In noncentrosymmetric crystals, $U_g = U_g^c + iU_g^i$, where U_g^c and U_g^i are each the complex Fourier coefficients of real potentials
V_g	$= h^2 U_g / (2m e)$, Fourier coefficient of the crystal potential, in volts
$V(\mathbf{r})$	Crystal potential, in volts
ξ_g	Two-beam extinction distance, $\xi_g = 1/(\lambda U_g) = \pi/(V_g \sigma) = \pi \Omega / (\gamma \lambda F_g^B)$
t	Sample thickness

E_0	Accelerating voltage
E_r	Relativistic accelerating voltage $[= E_0 + (e/2m_0c^2)E_0^2]$
E	An electric field
γ	Relativistic factor $(1 - v^2/c^2)^{-1/2}$
γ^i	Eigenvalue of the structure matrix
α_c	Coherence angle. $\alpha_c = \lambda/d_s$

In expressions such as Eq. (8.1) we adopt the convention that all quantities following the solidus are to be divided into those preceeding it.

Introduction: A Note to Students in Materials Science

Electron microdiffraction is a technique for extracting crystal-structure information from submicron regions of material, using a transmission electron microscope (TEM). Information on the atomic structure of defects, on local crystal symmetry, and on strain fields may be extracted from regions as small as a nanometer in diameter or less by this method. This book is about the interpretation of these transmission electron diffraction patterns. In particular, we have attempted to summarize and develop all the useful knowledge which has been gained over the years from the study of the multiple electron scattering problem, and to apply it to practical problems in the interpretation of microdiffraction patterns. The book covers theory, applications, and experimental methods. It has been said that it is now impossible to write a book on electron microscopy—the subject is simply too large. Several volumes would be required to cover in detail all the relevant knowledge, from ultrahigh vacuum systems to X-ray detectors, dynamical scattering theory in transmission and reflection, electron optics, and electron crystallography for organic materials. We have concentrated in this book on the signal which is available from most modern TEM instruments that we feel is most accurately quantifiable—the transmission electron diffraction pattern.

From the beginning it was realized that the convergent-beam electron microdiffraction method in particular is an extraordinarily efficient technique. The wide range of illumination angles used provides a large amount of information in the form of rocking curves for many diffracted orders, presented simultaneously from the smallest possible region of crystal. In addition, different regions of the pattern are sensitive to different crystal parameters—the outer reflections and HOLZ line intensities to atomic position parameters and temperature factors, the inner reflections for bonding effects, and HOLZ line positions for lattice

parameters and strain. This book shows how this information may be extracted by a process of automated dynamical refinement, weighted according to the type of information required and the portion of the CBED pattern used.

Students embarking on a course in materials science at the graduate level will want to know where this book fits into the overall scheme of modern materials science. It is often said that materials science is the study of the structure, properties, and synthesis of materials. The relationship between structure and properties is heavily emphasized in recent work. Characterization forms an essential part of all the pertinent analysis. This book concerns one mode (microdiffraction) of operation for an electron microscope. It therefore treats a rather specialized topic, which is part of the overall characterization effort in materials science. A considerable effort is now under way in many countries aimed at understanding and predicting the properties of new materials in terms of atomic mechanisms. Microdiffraction contributes to this effort by giving us crystallographic information on a near-atomic scale. The following brief list of recent applications gives some impression of the contribution microdiffraction has made to our understanding of atomic processes in solids.

1. In the semiconductor industry, considerable effort has gone into the production of multilayer devices using molecular beam epitaxy in order to grow devices at low temperatures, where atomically abrupt interfaces can be generated and diffusion avoided. For silicon-germanium multilayers, for example, it has proven possible to grow metastable films in which there are no misfit dislocations to interfere with device performance, provided the layers are sufficiently thin. Then the electronic structure of the layers depends critically on the strains stored in them. Only by nanodiffraction has it been possible to measure this strain within a single layer (Pike *et al.*, 1991) and at these interfaces (Eagelsham *et al.*, 1989). In another example of the application of the same method [Zuo, 1991a; see also Boe and Gjønnes (1991) for related work], the change in lattice constant at two points a few hundred nanometers apart in the oxide superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{6-x}$ was measured, and the result related to changes in the local oxygen concentration x , using data from neutron diffraction. The onset of superconductivity is known to be strongly correlated with oxygen concentration and ordering.
2. For many applications it is required to form a strong bond

between a ceramic and a metal. The strength of such a bond depends crucially on the interface energies involved. If a thin layer of a third phase is present at the interface, the relevant interface energies will differ from those at a bilayer. Thus the resistance to crack propagation at the interface may be controlled by a layer of material just a few unit cells thick. Only by nanodiffraction has it been possible to identify the crystal structure of such an intergranular phase (Lodge and Cowley, 1984).

3. The performance of the new high-temperature superalloys used in turbine blade applications frequently depends on the presence of many fine intermetallic precipitates and a complex microstructure. These microcrystals may be as small as a few nanometers in diameter. There now exist several examples of phase identification in these and similar alloys using convergent-beam diffraction to identify the space group of the microcrystal; see, for example, Fraser (1983).
4. In the field of catalysis, information is required on heavy metal particles, perhaps 5 nm in diameter or smaller, lying on a low-atomic-number substrate. The method of high-angle annular detector (HAAD) scanning transmission electron microscopy (STEM) combined with microdiffraction has proven extremely powerful for the study of these particles, since it is possible to suppress diffraction contrast effects from the substrate (Treacy *et al.*, 1978) and to combine the method with secondary electron imaging (Liu and Cowley, 1990). The HAAD signal depends on the atomic number of the sample, its thickness, and the vibration amplitude. The secondary electron image provides topographic information on a subnanometer scale. The microdiffraction technique is indispensable for the identification of individual particles and their crystallographic relationship with the substrate.

More generally, the need for the information which microdiffraction provides arises in many fields, from fundamental experiments in condensed matter physics, through mineralogy and solid state chemistry, to physical metallurgy and materials science generally. In all these fields there has been a growing recognition of the importance of the effect of crystalline microstructure on the behavior and properties of materials. In condensed matter physics, for example, the method has been used to study changes in symmetry during phase transitions on a local scale, and to map out the distribution of valence electrons in semiconductors. Other

uses of the technique include the very accurate measurement of electron microscope accelerating voltage, of sample thickness, of crystal lattice spacings, and of crystal structure-factor amplitudes and phases. Recent developments, using an automated refinement cycle similar to the Rietvelt method in neutron diffraction, suggest that atomic positions and lattice strains may be measured with high accuracy, allowing deductions to be made concerning ordering and impurity segregation in crystals. The method also provides a powerful technique for microphase identification, through its unique ability to determine the space group of very small microcrystals. When the smallest (subnanometer) coherent electron probes are used, the theory of CBED becomes the theory of scanning transmission electron microscopy (STEM) and, for very thin crystals, of in-line electron holography. This technique, while yet to be fully developed, possesses in principle the same power to determine the atomic structure of crystalline defects as high-resolution electron microscopy, while allowing simultaneous microanalysis.

Microdiffraction patterns are obtainable on all modern analytical electron microscopes. They differ from conventional selected area ("spot" or "point") diffraction patterns in that a small electron probe is focused onto the sample, causing the diffraction spots to broaden out into disks. We shall use the terms convergent-beam electron diffraction (CBED) and microdiffraction synonymously throughout this book to refer to all electron diffraction patterns in which a submicron electron probe is focused onto a sample. The term "nanodiffraction" will be used to refer to similar patterns, in which the probe size is smaller than about a nanometer in diameter. These patterns are also known as Kossel-Möllenstedt patterns, after their discoverers. (A "Kossel pattern" is a CBED pattern whose orders overlap.) The book covers four main topics: Space-group determination by CBED, structure-factor measurement by CBED, coherent CBED (holograms, ronchigrams) from defects using subnanometer probes, and strain measurement using HOLZ-line shifts. A chapter on instrumentation and experimental technique is included. We have also tried to provide a unified and coherent account of all the relevant theoretical background. We have emphasized fundamentals in the theory chapters, because our experience has been that this is the way in which progress is made. We hope that our unified treatment of the theory will suggest new experiments and new signals which can be used for materials characterization. For example, only quite recently has it been widely appreciated in the materials community that Bragg's law can be in error by as much as 5% for electron diffraction (due to multiple scattering), and that this has important consequences for the accurate measurement of strains near interfaces.

Working in an electron microscopy laboratory for many years has convinced us that most of the enormous amount of information contained in electron diffraction patterns is simply thrown away. This book is an attempt to reverse that trend, and to promote the development of a truly quantitative electron crystallography method. The book is intended for graduate students and professional research workers in materials science and condensed matter physics. We have tried to cover the theory, the practice, and some applications of the method, together with a very brief account of the history of the subject. An undergraduate knowledge of crystallography and some familiarity with electron microscopy are assumed.

The first half of the book is concerned mainly with theory, the second with applications, techniques, computer algorithms, and instrumentation. Students completing graduate courses in materials science may wish to read only the following: Chapter 2, Sections 3.3 and 3.4, Section 4.1, Section 5.5, and Chapters 7 and 9. For those wanting only an introduction to the subject, Chapters 2 and 9 are the most important.

While a large number of papers, a few review chapters, an atlas of CBED patterns, and two outstanding volumes of annotated examples of CBED patterns have appeared, there seems to be no other unified description of the theory and practice of convergent-beam electron diffraction (CBED) which aims at pedagogic soundness in an integrated treatment, using a consistent formalism throughout. Some useful companion review papers include: The two numbers of *J. Microsc. Tech.*, (1989) Vol. 13, Nos. 1 and 2 devoted to CBED, and Cowley (1978), Eades (1988), Muddle (1985), Spence and Carpenter (1986), Steeds (1979), and Williams (1987). The most important books on the subject are Cowley (1992) and Tanaka *et al.* (1988).

A recent literature survey (Sung and Williams, 1991) lists over 200 publications on CBED in 1991, as against 10 in 1975. This growth in the popularity of the method is due to advances both in computing power and in the performance of modern analytical electron microscopes. These include the appearance of convenient and reliable field-emission guns for TEM, of Omega energy-filtering devices, of cooled CCD detectors, and stable heating and cooling double-tilt specimen holders, of the new RISC work stations, together with the improved vacuum systems on modern instruments which reduce contamination and so make CBED possible. We shall see, however, that the basic theoretical work on which these modern developments are based occurred mainly in the 1960s and earlier.

We have written this book with the strong conviction that the intensity distribution in elastic (energy-filtered) electron microdiffraction patterns now provides the most accurately quantifiable information

available on a modern analytical transmission electron microscope (TEM). For example, using appropriate dynamical computations, it is possible to measure strains as small as 10^{-4} , structure-factor phases with an accuracy of much less than one degree, and sample thickness to an accuracy of a few angstroms. This information is obtainable from regions as small as a few nanometers in diameter. Our second motivation has been the fascinating and rich history of the subject, briefly outlined in Chapter 1.

On reading the literature, one is struck by the enormous variety of applications of CBED. These include studies of charge-density waves in layered structures, analyses of the symmetry changes which accompany low-temperature phase transitions, accurate mapping of the distribution of valence electrons in semiconductors, and phase identification and strain measurement around precipitates in alloys or semiconductors. To review all this work, published in over one-thousand papers, and to extract its implications for materials physics would be a herculean task. (We have, however, included a bibliographic index of applications indexed according to material in Appendix 7.) Thus, to experts in the field, the examples in this book may seem somewhat oversimplified. But our limited aim has been only to explain the principles of CBED, to provide all the useful theory in a consistent format, and to convey enough understanding to students and researchers to let them get started with microdiffraction. In order to make the book more practically useful, we have included some FORTRAN source code listings, together with the POSTSCRIPT code which will allow the direct printing of Kikuchi- and HOLZ-line patterns on modern laser printers.