

Handbook of Microscopy

Applications in Materials Science,
Solid-State Physics and Chemistry

Edited by

S. Amelinckx, D. van Dyck,

J. van Landuyt, G. van Tendeloo

Methods II



Weinheim · New York · Basel · Cambridge · Tokyo

This Page Intentionally Left Blank

S. Amelinckx, D. van Dyck, J. van Landuyt,
G. van Tendeloo

Handbook of Microscopy

Methods II



Handbook of Microscopy

Applications in Materials Science, Solid-State Physics and Chemistry

Methods I

1997. ISBN 3-527-29280-2.

Methods II

1997. ISBN 3-527-29473-2.

Applications

1997. ISBN 3-527-29293-4.

Further Reading from VCH

S. N. Magonor, M.-U. Whangbo

Surface Analysis with STM and AFM

Experimental and Theoretical Aspects of Image Analysis

ISBN 3-527-29313-2

D. A. Bonnell

Scanning Tunnelling Microscopy and Spectroscopy

Theory, Techniques and Applications

ISBN 3-527-27920-2

© VCH Verlagsgesellschaft mbH, D-69451 Weinheim (Federal Republic of Germany), 1997

Distribution:

VCH, P.O. Box 10 11 61, D-69451 Weinheim (Federal Republic of Germany)

Switzerland: VCH, P.O. Box, CH-4020 Basel (Switzerland)

United Kingdom and Ireland: VCH (UK) Ltd., 8 Wellington Court, Cambridge CB1 1HZ (England)

USA and Canada: VCH, 333 7th Avenue, New York, NY 10001 (USA)

Japan: VCH, Eikow Building, 10-9 Hongo 1-chome, Bunkyo-ku, Tokyo 113 (Japan)

ISBN 3-527-29473-2

Handbook of Microscopy

Applications in Materials Science,
Solid-State Physics and Chemistry

Edited by

S. Amelinckx, D. van Dyck,

J. van Landuyt, G. van Tendeloo

Methods II



Weinheim · New York · Basel · Cambridge · Tokyo

Prof. S. Amelinckx
Electron Microscopy for
Materials Science (EMAT)
University of Antwerp - RUCA
Groenenborgerlaan 171
2020 Antwerp
Belgium

Prof. D. van Dyck
Electron Microscopy for
Materials Science (EMAT)
University of Antwerp - RUCA
Groenenborgerlaan 171
2020 Antwerp
Belgium

Prof. J. van Landuyt
Electron Microscopy for
Materials Science (EMAT)
University of Antwerp - RUCA
Groenenborgerlaan 171
2020 Antwerp
Belgium

Prof. G. van Tendeloo
Electron Microscopy for
Materials Science (EMAT)
University of Antwerp - RUCA
Groenenborgerlaan 171
2020 Antwerp
Belgium

This book was carefully produced. Nevertheless, authors, editors and publisher do not warrant the information contained therein to be free of errors. Readers are advised to keep in mind that statements, data, illustrations, procedural details or other items may inadvertently be inaccurate.

Published by
VCH Verlagsgesellschaft mbH, Weinheim (Federal Republic of Germany)

Editorial Directors: Dr. Peter Gregory, Dr. Ute Anton, Dr. Jörn Ritterbusch
Production Manager: Dipl.-Wirt.-Ing. (FH) Hans-Jochen Schmitt

Every effort has been made to trace the owners of copyrighted material; however, in some cases this has proved impossible. We take this opportunity to offer our apologies to any copyright holders whose rights we may have unwittingly infringed.

Library of Congress Card No. applied for.

A catalogue record for this book is available from the British Library.

Die Deutsche Bibliothek – CIP-Einheitsaufnahme
Handbook of microscopy : applications in materials science,
solid state physics and chemistry / ed. by S. Amelinckx ... -
Weinheim ; New York ; Basel ; Cambridge ; Tokyo : VCH.
NE: Amelinckx, Severin [Hrsg.]
Methods 2 (1997)
ISBN 3-527-29473-2

© VCH Verlagsgesellschaft mbH, D-69451 Weinheim (Federal Republic of Germany), 1997

Printed on acid-free and chlorine-free paper.

All rights reserved (including those of translation into other languages). No part of this book may be reproduced in any form – by photoprinting, microfilm, or any other means – nor transmitted or translated into a machine-readable language without written permission from the publishers. Registered names, trademarks, etc. used in this book, even when not specifically marked as such, are not to be considered unprotected by law.

Composition: Alden Bookset, England
Printing: betz-druck, D-64291 Darmstadt
Bookbinding: W. Osswald, D-67433 Neustadt

Short biography of the editors



Severin Amelinckx was born in Willebroek, Belgium, in 1922. He studied at the University of Ghent, receiving his first degree (licence) in mathematics in 1944, his doctorate in physics in 1952, and his aggregation in physics in 1955. Currently he is Emeritus Professor of General Physics and Crystallography associated with the EMAT laboratory of the University of Antwerp (RUCA). Until 1987 he was Director General of the Belgian Nuclear Research Establishment at Mol. He is a member of the European Academy and of the Koninklijke Academie voor Wetenschappen, Letteren en Schone Kunsten van België and former chairman of the division of sciences of this academy.

His research interests include electron diffraction contrast imaging, defects in solids, phase transformations and their resulting domain structures, crystal growth, dislocations, fullerenes and nanotubes, the structure of high- T_c superconductors, modulated structures, and order-disorder in alloys.



Joseph Van Landuyt, who was born in St. Amandsberg, Belgium, in 1938, obtained both his licence (1960) and doctorate in physics (1965) from the University of Ghent. At present he is Professor of General Physics and Crystallography at the University of Antwerp (RUCA and UIA) and of Electron Microscopy at UIA and the Flemish University of Brussels (VUB). He is a member of the Koninklijke Academic voor Wetenschappen, Letteren en Schone Kunsten van België.

His research interests are centered on the study of nanostructural features in alloys, ceramics, and minerals (in particular gems), with special interest in defects in semiconductors and their relation to device performance. More general subjects of interest are structural variants, defects, and phase transitions in various solids.



Gustaaf Van Tendeloo, born in Lier, Belgium, in 1950, received his licence in physics from the University of Brussels (VUB) in 1972, his doctorate from the University of Antwerp (UIA) in 1974, and his aggregation from the University of Brussels (VUB) in 1981. He has been associated with the University of Antwerp (RUCA) since 1972, but has spent extended periods of time as a researcher in Berkeley (USA), Caen (France), and elsewhere. He is currently Professor of Solid-State Physics at the University of Brussels (VUB) and of the Physics of Materials at the University of Antwerp (RUCA and UIA).

His research interests include the electron microscopy of inorganic solids (in particular high- T_c superconductors), solid-state phase transitions, modulated structures, fullerenes, defects in crystals order-disorder in alloys, and nanostructural features in general.



Dirk Van Dyck was born in Wilrijk, Belgium, in 1948. He studied physics, receiving his licence from the University of Brussels (VUB) in 1971 before moving to the University of Antwerp (UIA) for his doctorate (1977) and aggregation (1987). He has been associated with the University of Antwerp since 1971, and is at present Professor of Theoretical Mechanics, Digital Techniques and Image Processing at the University of Antwerp.

Among his research interests are theoretical aspects of dynamic electron diffraction and imaging, holographic reconstruction and structural retrieval, image processing and pattern recognition, and artificial intelligence. In particular, he is involved in the development of a 1 Å resolution microscope in the framework of the Brite/Euram program of the European Union.

The four editors belong to the Electron Microscopy for Materials Science (EMAT) laboratory, University of Antwerp (RUCA), which was founded in 1965. All four have contributed significantly to the development of electron microscopy and its application by numerous publications in journals and books and are members of editorial boards of several international journals in the field of materials science. They have also delivered numerous invited lectures at international conferences in their respective areas of research.

List of Contributors

Bauer, Ernst (V:3)
Physikal. Institut
Technische Universität Clausthal
38678 Clausthal
Germany

Bonnet, Noël (VIII:2)
INSERM Unit 314
University of Reims
21, rue Clément Ader
51100 Reims
France

Cerezo, Alfred; Smith, George D. W.
(VI:2)
Department of Materials
University of Oxford
Parks Road
Oxford OX1 3PH
United Kingdom

Cory, David G.; Choi, Sungmin (V:1)
Dept. of Nuclear Engineering,
NW 14-4111
Massachusetts Institute of Technology
Cambridge, MA 02139
USA

Cowley, John M. (IV:2.2)
Arizona State University
Dept. of Physics & Astronomy
Box 87 15 04
Tempe, AZ 8528 -1504
USA

DiNardo, N. John (VII:4)
Department of Physics and
Atmospheric Science
Drexel University
Philadelphia, PA 19104
USA

Fiermans, Lucien; De Gryse, Roger
(IV:2.4)
Department of Solid State Sciences
Surface Physics Division
University of Gent
Krijgslaan 281/S1
9000 Gent
Belgium

Herrmann, Karl Heinz (VIII:1)
Institute of Applied Physics
University of Tübingen
Auf der Morgenstelle 10
72076 Tübingen
Germany

Joy, David C. (IV:2.1)
The University of Tennessee
EM Facility – Programm in Analytical
Microscopy
F241 Walters Life Sciences Building
Knoxville, Tennessee 37996/0810
USA

Kruit, Peter (IX:1)
Delft University of Technology
Dept. of Applied Physics
Lorentzweg 1
2628 CS Delft
The Netherlands

Mundschau, Michael V. (VI:1)
Bowling Green State University
Dept. of Chemistry
Bowling Green, Ohio 43403-0213
USA

Oleshko*, Vladimir; Gijbels, Renaat
(IV:2.5)
Department of Chemistry
University of Antwerp (UIA)
2610 Wilrijk-Antwerpen
Belgium

*On leave from the Russian Academy
of Sciences
N. N. Semenov Institute of Chemical
Physics
117421 Moscow
Russia

Pennycook, Stephen J. (IV:2.3)
Solid State Division
Oak Ridge National Laboratory
P. O. Box 2008
Oak Ridge, TN 37831-6030
USA

Scheinfein, Mike R. (V:2)
Dept. of Physics and Astronomy
Arizona State University
Tempe, AZ 85287-1504
USA

Unguris, John; Kelley, Michael H.;
Gavrin, Andrew; Celotta, Robert J.;
Pierce, Daniel T. (V:2)
Electron Physics Group
Nat. Institute of Standards and
Technology
Gaithersburg, MD 20899
USA

Schwarz, Udo D. (VII:2)
Institute of Applied Physics
University of Hamburg
Jungiusstr. 11
20355 Hamburg

Spence, John C. H. (IX:2)
Dept. of Physics
A. S. U.
Tempe, AZ 85287
USA

Ventrice, Carl A., Jr. (VII:4)
Department of Physics
Rensselaer Polytechnic Institute
Troy, NY 12180
USA

Wadas, Andrzej (VII:3)
Institute of Applied Physics
University of Hamburg
Jungiusstr. 11
20355 Hamburg
Germany

Wiesendanger, Roland (VII:1)
University of Hamburg
Institute of Applied Physics and
Microstructure Research Center
Jungiusstr. 11
20355 Hamburg
Germany

Van Espen, Pierre; Janssens, Gert
(IV:2.6)
Micro and Trace Analysis Centre
Dept. of Chemistry
University of Antwerpen (UIA)
Universiteitsplein 1
2610 Antwerpen
Belgium

Outline

Volume 1: Methods I

I Light Microscopy

- 1 **Fundamentals of Light Microscopy**
F. Mücklich
- 2 **Optical Contrasting of Microstructures**
F. Mücklich
- 3 **Raman Microscopy**
P. Dhamelincourt, J. Barbillat
- 4 **Three-Dimensional Light Microscopy**
E. H. K. Stelzer
- 5 **Near Field Optical Microscopy**
D. Courjon, M. Spajer
- 6 **Infrared Microscopy**
J. P. Huvenne, B. Sombret

II X-Ray Microscopy

- 1 **Soft X-Ray Imaging**
G. Schmahl
- 2 **X-Ray Microradiography**
D. Mouze
- 3 **X-Ray Microtomography**
J. Cazaux
- 4 **Soft X-Ray Microscopy by Holography**
D. Joyeux
- 5 **X-Ray Diffraction Topography**
M. Schlenker, J. Baruchel

III Acoustic Microscopy

- 1 **Acoustic Microscopy**
A. Briggs

IV Electron Microscopy

1 Stationary Beam Methods

- 1.1 Transmission Electron Microscopy
- 1.1.1 Diffraction Contrast Transmission Electron Microscopy
S. Amelinckx
- 1.1.2 High-Resolution Electron Microscopy
D. Van Dyck
- 1.2 Reflection Electron Microscopy
J. M. Cowley
- 1.3 Electron Energy-Loss Spectroscopy Imaging
C. Colliex
- 1.4 High Voltage Electron Microscopy
H. Fujita
- 1.5 Convergent Beam Electron Diffraction
D. Cherns, J. W. Steeds, R. Vincent
- 1.6 Low-Energy Electron Microscopy
E. Bauer
- 1.7 Lorentz Microscopy
J. P. Jakubovics
- 1.8 Electron Holography Methods
H. Lichte

Volume 2: Methods II

IV Electron Microscopy

2 Scanning Beam Methods

- 2.1 Scanning Reflection Electron Microscopy
D. C. Joy
- 2.2 Scanning Transmission Electron Microscopy
J. M. Cowley
- 2.3 Scanning Transmission Electron Microscopy: Z Contrast
S. J. Pennycook
- 2.4 Scanning Auger Microscopy (SAM) and Imaging X-Ray Photoelectron Microscopy (XPS)
R. De Gryse, L. Fiermans
- 2.5 Scanning Microanalysis
R. Gijbels
- 2.6 Imaging Secondary Ion Mass Spectrometry
P. van Espen, G. Janssens

V Magnetic Methods

- 1 Nuclear Magnetic Resonance**
D. G. Cory, S. Choi
- 2 Scanning Electron Microscopy with Polarization Analysis (SEMPA)**
J. Unguris, M. H. Kelley, A. Gavrin, R. J. Celotta, D. T. Pierce, M. R. Scheinfein
- 3 Spin-Polarized Low-Energy Electron Microscopy**
E. Bauer

VI Emission Methods

- 1 Photoelectron Emission Microscopy**
M. Mundschau
- 2 Field Emission and Field Ion Microscopy (Including Atom Probe FIM)**
A. Cerezo, G. D. W. Smith

VII Scanning Point Probe Techniques

- General Introduction
- 1 Scanning Tunneling Microscopy**
R. Wiesendanger
 - 2 Scanning Force Microscopy**
U. D. Schwarz
 - 3 Magnetic Force Microscopy**
A. Wadas
 - 4 Ballistic Electron Emission Microscopy**
J. DiNardo

VIII Image Recording, Handling and Processing

- 1 Image Recording in Microscopy**
K.-H. Herrmann
- 2 Image Processing**
N. Bonnet

IX Special Topics

- 1 Coincidence Microscopy**
P. Kruit

- 2 Low Energy Electron Holography and Point-Projection
Microscopy**
J. C. H. Spence

Volume 3: Applications

I Classes of Materials

- 1 Metals and Alloys**
J. Th. M. De Hosson
G. van Tendeloo
- 2 Microscopy of Rocks and Minerals**
D. J. Barber
- 3 Semiconductors and Semiconducting Devices**
H. Oppolzer
- 4 Optoelectronic Materials**
I. Berbezier, J. Derrien
- 5 Domain Structures in Ferroic Materials**
E. K. H. Salje
- 6 Structural Ceramics**
M. Rühle
- 7 Microscopy of Gemmological Materials**
J. van Landuyt, M. H. G. van Bockstael, J. van Royen
- 8 Superconducting Ceramics**
G. van Tendeloo
- 9 Non-Periodic Structures**
- 9.1 High-Resolution Imaging of Amorphous Materials
P. H. Gaskell
- 9.2 Quasi-Crystalline Structures
K. H. Kuo
- 10 Medical and Dental Materials**
K. Yasuda, K. Hisatsune, H. Takahashi, K.-I. Udoh, Y. Tanaka
- 11 Carbon**
D. Bernaerts and S. Amelinckx
- 12 Composite Structural Materials**
*O. Van der Biest, P. Lust, K. Lambrinou, J. Ivens, I. Verpoest,
L. Froyen*
- 13 The Structure of Polymers and Their Monomeric Analogs**
I. G. Voigt-Martin

- 14 **Nuclear Materials**
H. Blank, Hj. Matzke, H. Maußner, I. L. F. Ray
- 15 **Magnetic Microscopy**
A. Hubert

II **Special Topics**

- 1 **Small Particles**
(Catalysis, Photography, Magnetic Recording)
H. W. Zandbergen, C. Træholt
- 2 **Structural Phase Transformations**
H. Warlimont
- 3 **Preparation Techniques**
for Transmission Electron Microscopy
A. Barna, G. Radnóczy, B. Pécz
- 4 **Environmental Problems**
W. Jambers, R. E. Van Grieken
- 5 **Quantitative Hyleography:**
The Determination of Quantitative Data From Micrographs
P. J. Goodhew

This Page Intentionally Left Blank

Contents

Volume 2: Methods II

IV Electron Microscopy

- 2 Scanning Beam Methods 537**
- 2.1 Scanning Reflection Electron Microscopy 539
D. C. Joy
 - 2.1.1 Introduction 539
 - 2.1.2 Instrumentation 540
 - 2.1.3 Performance 542
 - 2.1.4 Modes of Operation 544
 - 2.1.4.1 Secondary Electron Imaging 544
 - 2.1.4.2 Backscattered Electrons 548
 - 2.1.4.3 Special Techniques 553
 - 2.1.5 Conclusions 561
 - 2.1.6 References 561
- 2.2 Scanning Transmission Electron Microscopy 563
J. M. Cowley
 - 2.2.1 Introduction 563
 - 2.2.2 Scanning Transmission Electron Microscopy Imaging Modes 566
 - 2.2.3 Scanning Transmission Electron Microscopy Theory 570
 - 2.2.4 Inelastic Scattering and Secondary Radiations 574
 - 2.2.5 Convergent-Beam and Nanodiffraction 577
 - 2.2.6 Coherent Nanodiffraction, Electron Holography, Ptychology 578
 - 2.2.7 Holography 581
 - 2.2.8 STEM Instrumentation 584
 - 2.2.9 Applications of Scanning Transmission Electron Microscopy 587
 - 2.2.10 References 592
- 2.3 Scanning Transmission Electron Microscopy: Z Contrast 595
S. J. Pennycook
 - 2.3.1 Introduction 595

- 2.3.2 Incoherent Imaging with Elastically Scattered Electrons 598
- 2.3.3 Incoherent Imaging with Thermally Scattered Electrons 601
- 2.3.4 Incoherent Imaging using Inelastically Scattered Electrons 604
- 2.3.5 Probe Channeling 606
- 2.3.6 Applications to Materials Research 610
 - 2.3.6.1 Semiconductors 610
 - 2.3.6.2 Ceramics 613
 - 2.3.6.3 Nanocrystalline Materials 616
- 2.3.7 References 619

- 2.4 Scanning Auger Microscopy (SAM) and Imaging X-Ray Photoelectron Microscopy (XPS) 621
R. De Gryse, L. Fiermans
- 2.4.1 Introduction 621
- 2.4.2 Basic Principles of Auger Electron Spectroscopy (AES) and X-Ray Photoelectron Spectroscopy (XPS) 622
 - 2.4.2.1 Auger Electron Spectroscopy (AES) 622
 - 2.4.2.2 X-Ray Photoelectron Spectroscopy (XPS) 625
 - 2.4.2.3 Quantitative Analysis in AES and XPS 627
- 2.4.3 Scanning Auger Microscopy (SAM) and Imaging XPS 630
 - 2.4.3.1 Basic Principles of Imaging 630
 - 2.4.3.2 General Aspects of Analyzers 632
 - 2.4.3.3 Energy Resolution of Deflecting Electrostatic Analyzers 635
 - 2.4.3.4 Cylindrical Mirror Analyzer (CMA) versus the Concentric Hemispherical Analyzer (CHA) 637
 - 2.4.3.5 Imaging Techniques 644
 - 2.4.3.6 Magnetic Fields in Imaging XPS 652
- 2.4.4 Characteristics of Scanning Auger Microscopy Images 654
 - 2.4.4.1 General Aspects 654
 - 2.4.4.2 Background Slope Effects 656
 - 2.4.4.3 Substrate Backscattering Effects 656
 - 2.4.4.4 Topographic Effects 656
 - 2.4.4.5 Beam Current Fluctuation Effects 657
 - 2.4.4.6 Edge Effects 657
- 2.4.5 Conclusion 658
- 2.4.6 References 658

- 2.5 Scanning Microanalysis 661
R. Gijbels
- 2.5.1 Physical Basis of Electron Probe Microanalysis 661
 - 2.5.1.1 Electron Interactions with Solids 661
 - 2.5.1.2 X-Ray Emission Spectra 664
 - 2.5.1.3 Characteristic X-Ray Spectra 666

2.5.1.4	Soft X-Ray Spectra	668
2.5.1.5	X-Ray Continuum	669
2.5.1.6	Overview of Methods of Scanning Electron Beam Analysis	669
2.5.1.7	Electron Probe X-Ray Microanalyzers	669
2.5.1.8	Analytical Electron Microscopes	673
2.5.1.9	Multipurpose Electron Probe Analytical Systems	675
2.5.1.10	X-Ray Emission Spectrometry	679
2.5.1.11	Wavelength-Dispersive Spectrometry	679
2.5.1.12	Energy-Dispersive Spectrometry	680
2.5.1.13	X-Ray Mapping	681
2.5.2	Introduction to Quantitative X-Ray Scanning Microanalysis	682
2.5.2.1	ZAF Method	683
2.5.2.2	Atomic Number Correction	683
2.5.2.3	X-Ray Absorption Correction	684
2.5.2.4	Fluorescence Corrections	684
2.5.2.5	$f(pz)$ Methods	685
2.5.2.6	Standardless Analysis	686
2.5.2.7	Analysis of Thin Films and Particles	687
2.5.3	Conclusions	688
2.5.4	References	689
2.6	Imaging Secondary Ion Mass Spectrometry	691
	<i>P. Van Espen, G. Janssens</i>	
2.6.1	Introduction	691
2.6.1.1	Types of Secondary Ion Mass Spectrometry Measurements	691
2.6.1.2	Dynamic and Static Secondary Ion Mass Spectrometry	692
2.6.1.3	Ion Microscope and Ion Microprobe	692
2.6.1.4	Characteristics of Secondary Ion Mass Spectrometry	693
2.6.2	Secondary Ion Formation	694
2.6.3	Instrumentation	695
2.6.3.1	Primary Ion Sources	695
2.6.3.2	Sample Chamber	696
2.6.3.3	Mass Spectrometer	696
2.6.3.4	Ion Detection and Image Registration	696
2.6.3.5	Typical Configurations	698
2.6.4	Comparison of Ion Microprobe and Ion Microscope Mode	702
2.6.5	Ion Image Acquisition and Processing	704
2.6.5.1	Dynamic Range of Secondary Ion Mass Spectrometry Ion Images	704
2.6.5.2	Influence of Mass Resolution	705
2.6.5.3	Image Sequences	705
2.6.5.4	Interpretation and Processing of Ion Images	706

2.6.5.5	Analysis of Image Depth Sequences	707
2.6.5.6	Analysis of Multivariate Ion Images	709
2.6.6	Sample Requirements	711
2.6.7	Application Domain	712
2.6.8	References	714

V **Magnetic Methods**

1	Nuclear Magnetic Resonance	719
	<i>D. G. Cory, S. Choi</i>	
1.1	Introduction	719
1.2	Background	720
1.3	Magnetic Field Gradients, Magnetization Gratings, and <i>k</i> -Space	723
1.4	Nuclear Magnetic Resonance	727
1.5	Echoes and Multiple-Pulse Experiments	727
1.6	Two-Dimensional Imaging	730
1.7	Slice Selection	731
1.8	Gratings and Molecular Motions	732
1.9	Solid State Imaging	733
1.10	References	734
2	Scanning Electron Microscopy with Polarization Analysis (SEMPA)	735
	<i>J. Unguris, M. H. Kelley, A. Gavrin, R. J. Celotta, D. T. Pierce, M. R. Scheinfein</i>	
2.1	Introduction	735
2.2	Principle of SEMPA	737
2.3	Instrumentation	739
2.3.1	Scanning Electron Microscopy Probe Forming Column	739
2.3.2	Transport Optics	740
2.3.3	Electron Spin Polarization Analyzers	740
2.3.4	Electronics and Signal Processing	742
2.4	System Performance	743
2.5	Data Processing	744
2.6	Examples	745
2.6.1	Iron Single Crystals	745
2.6.2	CoPt Magneto-optic Recording Media	746
2.6.3	Exchange Coupling of Magnetic Layers	746

- 2.6.4 Magnetic Singularities in Fe–SiO₂ Films 747
- 2.7 References 748

- 3 Spin-Polarized Low-Energy Electron Microscopy 751**
E. Bauer
 - 3.1 Introduction 751
 - 3.2 Theoretical Foundations 751
 - 3.3 Instrumentation 753
 - 3.4 Areas of Application 755
 - 3.5 Discussion 758
 - 3.6 Concluding Remarks 758
 - 3.7 References 758

VI Emission Methods

- 1 Photoelectron Emission Microscopy 763**
M. Mundschau
 - 1.1 Introduction 763
 - 1.2 Photoelectron Emission 763
 - 1.3 Microscopy with Photoelectrons 765
 - 1.4 Applications 768
 - 1.4.1 Monolayer Epitaxial Growth 768
 - 1.4.2 Chemical Kinetic Reaction-Diffusion Fronts in Monolayers 769
 - 1.4.3 Magnetic Materials 769
 - 1.5 Choice and Preparation of Samples 771
 - 1.6 References 771

- 2 Field Emission and Field Ion Microscopy (Including Atom Probe FIM) 775**
A. Cerezo, G. D. W. Smith
 - 2.1 Field Emission Microscopy 775
 - 2.2 Field Ion Microscopy 777
 - 2.2.1 Principle of the Field Ion Microscope 778
 - 2.2.2 Field Ionization 779
 - 2.2.3 Field Evaporation 780
 - 2.2.4 Image Formation, Magnification, and Resolution 781

2.2.5	Contrast from Lattice Defects and Alloys, and Analysis of Field Ion Microscope Images	784
2.2.6	Specimen-Preparation Techniques	787
2.3	Atom Probe Microanalysis	788
2.3.1	Principles of the Atom Probe Field Ion Microscope	788
2.3.2	Energy Deficits and Energy Compensation	791
2.3.3	Accuracy and Precision of Atom Probe Analysis	792
2.3.4	Atomic Plane Depth Profiling	793
2.3.5	Analysis of Semiconductor Materials	795
2.4	Three-Dimensional Atom Probes	795
2.4.1	Position-Sensing Schemes	797
2.4.2	Mass Resolution in the Three-Dimensional Atom Probe	798
2.4.3	Three-Dimensional Reconstruction of Atomic Chemistry	798
2.5	Survey of Commercially Available Instrumentation	799
2.6	References	800

VII Scanning Point Probe Techniques

General Introduction 805

1	Scanning Tunneling Microscopy	807
	<i>R. Wiesendanger</i>	
1.1	Introduction	807
1.2	Topographic Imaging in the Constant-Current Mode	807
1.2.1	Effects of Finite Bias	809
1.2.2	Effects of Tip Wave Functions with Angular Dependence	810
1.2.3	Imaging of Adsorbates	811
1.2.4	Spatial Resolution in Constant-Current Topographs	812
1.3	Local Tunneling Barrier Height	815
1.3.1	Local Tunneling Barrier Height Measurements at Fixed Surface Locations	816
1.3.2	Spatially Resolved Local Tunneling Barrier Height Measurements	816
1.4	Tunneling Spectroscopy	817
1.4.1	Scanning Tunneling Spectroscopy at Constant Current	819
1.4.2	Local Spectroscopic Measurements at Constant Separation	820
1.4.3	Current Imaging Tunneling Spectroscopy	820
1.5	Spin-Polarized Scanning Tunneling Spectroscopy	821
1.6	Inelastic Tunneling Spectroscopy	823
1.6.1	Phonon Spectroscopy	824

- 1.6.2 Molecular Vibrational Spectroscopy 824
- 1.7 References 825

2 Scanning Force Microscopy 827

U. D. Schwarz

- 2.1 Introduction 827
- 2.2 Experimental Aspects 829
 - 2.2.1 The Force Sensor 829
 - 2.2.2 Deflection Sensors 831
 - 2.2.3 Imaging Modes 832
 - 2.2.3.1 Constant Force Mode 832
 - 2.2.3.2 Variable Deflection Mode 833
 - 2.2.3.3 Noncontact Dynamic Modes 834
 - 2.2.3.4 Imaging Friction, Elasticity, and Viscosity 835
 - 2.2.3.5 Other Imaging Modes 836
 - 2.2.4 Force–Distance Curves 836
 - 2.2.5 Tip Artefacts 837
 - 2.2.6 Scanning Force Microscopy as a Tool for Nanomodifications 838
- 2.3 Theoretical Aspects 838
 - 2.3.1 Forces in Force Microscopy 838
 - 2.3.1.1 Pauli Repulsion and Ionic Repulsion 838
 - 2.3.1.2 Van der Waals Forces 839
 - 2.3.1.3 Adhesion 839
 - 2.3.1.4 Capillary Forces 840
 - 2.3.1.5 Interatomic and Intermolecular Bonds 840
 - 2.3.1.6 Frictional and Elastic Forces 840
 - 2.3.1.7 Magnetic and Electrostatic Forces 841
 - 2.3.2 Contrast Mechanism and Computer Simulations 841
- 2.4 References 842

3 Magnetic Force Microscopy 845

A. Wadas

- 3.1 Introduction 845
- 3.2 Force Measurement 845
- 3.3 Force Gradient Measurement 849
- 3.4 References 852

4	Ballistic Electron Emission Microscopy	855
	<i>J. DiNardo</i>	
4.1	Introduction	855
4.2	Experimental Considerations	857
4.3	First Demonstrations of Ballistic Electron Emission Microscopy	858
4.4	Theoretical Considerations	860
4.5	Ballistic Electron Emission Microscopy Analysis of Schottky Barrier Interfaces	864
4.5.1	Epitaxial Interfaces	865
4.5.2	Nonepitaxial Interfaces	867
4.5.3	Au/Si Interfaces	868
4.5.4	Metal-Film Dependence	869
4.5.5	Surface Gradients	869
4.5.6	Interfacial Nanostructures	870
4.5.7	Local Electron Tunneling Effects	872
4.5.8	Impact Ionization	873
4.6	Probing Beneath the Schottky Barrier	874
4.7	Ballistic Hole Transport and Ballistic Carrier Spectroscopy	879
4.8	Summary	881
4.9	References	881

VIII Image Recording, Handling and Processing

1	Image Recording in Microscopy	885
	<i>K.-H. Herrmann</i>	
1.1	Introduction	885
1.2	Fundamentals	885
1.2.1	The Primary Image	885
1.2.2	The General Recorder	886
1.2.3	Quantum Efficiency of Conversion Processes	889
1.2.3.1	Photographic Recording	890
1.2.3.2	Photoeffect	890
1.2.3.3	Scintillators	892
1.2.3.4	Light Optical Elements	893
1.2.3.5	Secondary Emission	895
1.2.3.6	Electron Beam-Induced Conduction	896
1.2.3.7	Imaging Plate	896
1.2.4	Composed Systems and Optoelectronic Components	896

- 1.2.4.1 Scintillator-Photosensor Combination 897
- 1.2.4.2 Image Intensifiers 897
- 1.2.4.3 Microchannel Plates 899
- 1.2.4.4 Television Camera Tubes 899
- 1.2.4.5 Charge-Coupled Devices 901
- 1.2.5 Resolution and Sampling 904
- 1.3 Light Microscopy 905
 - 1.3.1 Video Recording 905
 - 1.3.2 Low-Light-Level Detection 905
- 1.4 Electron Microscopy 906
 - 1.4.1 Photographic Recording 906
 - 1.4.2 Imaging Plate 907
 - 1.4.3 Electronic Recording 908
 - 1.4.3.1 Television Chains 908
 - 1.4.3.2 Slow-Scan Charge-Coupled Device Converters with a Scintillator 909
 - 1.4.3.3 Directly Back-Illuminated Charge-Coupled Devices 912
- 1.5 X-Ray Microscopy 912
 - 1.5.1 Photographic Film and Imaging Plate 913
 - 1.5.2 Resist 914
 - 1.5.3 Transmission Photocathodes 915
 - 1.5.4 Microchannel Plates 916
 - 1.5.5 Television Chains 916
 - 1.5.5.1 X-ray-Sensitive Vidicons 917
 - 1.5.5.2 Conversion to Visible Radiation 917
 - 1.5.6 Slow-Scan Charge-Coupled Device Chains 917
 - 1.5.7 Directly Illuminated Charge-Coupled Device Sensors 918
- 1.6 References 919

2 Image Processing 923

N. Bonnet

- 2.1 Introduction 923
- 2.2 Image Preprocessing 924
 - 2.2.1 Global Methods for Image Preprocessing 925
 - 2.2.1.1 Examples of Global Image Preprocessing in Image Space or Image Frequency Space 925
 - 2.2.1.2 Examples of Global Image Preprocessing in Parameter Space 926
 - 2.2.2 Local Methods for Image Preprocessing 927
 - 2.2.2.1 Example of Algorithm for Local Contrast Enhancement 928
 - 2.2.2.2 Example of Algorithm for Improving the Signal-to-Noise-Ratio 929

2.3	Processing of Single Images	931
2.3.1	Image Restoration	931
2.3.1.1	Restoration of Linear Degradations	931
2.3.1.2	Restoration of Partly Linear Degradations: Very High Resolution Electron Microscopy	932
2.3.1.3	Example of a Completely Nonlinear Restoration: Near-Field Microscopies	933
2.3.2	Image Segmentation	934
2.3.2.1	Segmentation on the Basis of Grey Levels Only	934
2.3.2.2	Segmentation on the Basis of Grey-Level Gradients	935
2.3.2.3	Segmentation on the Basis of Grey-Level Homogeneity and the Concept of Connectivity	935
2.3.2.4	Segmentation on the Basis of Grey Levels, Gradients, and Connectivity: Functional Minimization	936
2.3.2.5	Mathematical Morphology	936
2.4	Analysis of Single Images	937
2.4.1	Object Features	937
2.4.2	Pattern Recognition	937
2.4.3	Image Analysis without Image Segmentation	938
2.4.3.1	Texture Analysis	938
2.4.3.2	Fractal Analysis	939
2.4.3.3	Stereology	939
2.4.4	Mathematical-Morphology Approach to Image Analysis	940
2.4.4.1	Granulometry	940
2.4.4.2	Distance Function	940
2.4.4.3	Skeleton	942
2.5	Processing/Analysis of Image Series	942
2.5.1	Three-Dimensional Reconstruction	942
2.5.1.1	Serial Sections	943
2.5.1.2	Stereoscopy	943
2.5.1.3	Microtomography	944
2.5.1.4	Three-Dimensional Display	945
2.5.2	Processing and Analysis of Spectral, Temporal and Spatial Image Series	945
2.6	Conclusion	950
2.7	References	950

IX Special Topics

- 1 Coincidence Microscopy 955**
P. Kruit
 - 1.1 Introduction 955
 - 1.2 Instrumentation 956
 - 1.3 Coincidence Count Rates 958
 - 1.4 Signal Combinations 958
 - 1.4.1 EELS–Emitted Electron 958
 - 1.4.2 EELS–X-ray 961
 - 1.4.3 EELS–Cathodoluminescence 961
 - 1.4.4 Backscattered Electron–Secondary Electron 962
 - 1.4.5 Other Combinations 962
 - 1.5 References 962

- 2 Low Energy Electron Holography and Point-Projection Microscopy 963**
J. C. H. Spence
 - 2.1 Introduction and History 963
 - 2.2 Electron Ranges in Matter: Image Formation 966
 - 2.3 Holographic Reconstruction Algorithms 970
 - 2.4 Nanotips, Tip Aberrations, Coherence, Brightness, Resolution Limits, and Stray Fields 973
 - 2.5 Instrumentation 978
 - 2.6 Relationship to Other Techniques 981
 - 2.7 Future Prospects, Radiation Damage, and Point Reflection Microscopy 982
 - 2.8 References 985

General Reading

List of Symbols and Abbreviations

List of Techniques

Index

2 Scanning Beam Methods

This Page Intentionally Left Blank

2.1 Scanning Electron Microscopy

2.1.1 Introduction

The scanning electron microscope (SEM) is the most widely used of all electron beam instruments. It owes its popularity to the versatility of its various modes of imaging, the excellent spatial resolution of its images, the ease with which the micrographs that are generated can be interpreted, the modest demands that are made on specimen preparation, and its 'user-friendliness'. At one end of its operating range the SEM provides images which can readily be compared to those of conventional optical microscopes, while at the other end its capabilities are complementary to instruments such as scanning tunneling (STM) or atomic force (AFM) microscopes. While its resolution can now approach 0.5 nm, rivaling that of a transmission electron microscope, it can handle specimens as large as production size silicon wafers.

The SEM had its origins in the work of von Ardenne [1, 2] who added scanning coils to a transmission electron microscope. A photographic plate beneath the electron transparent sample was mechanically scanned in synchronism with the beam to produce the image. The first recognizably modern SEM was described by Zworykin et al. [3]. This instrument

incorporated most of the features of current instruments, such as a cathode-ray-tube display and a secondary electron detector, and achieved a resolution of 5 nm on solid specimens. In 1948 Oatley [4] and his students commenced their work on the development of the SEM leading in 1965 to the first commercial machine the Cambridge Scientific Instruments Mark 1 'Stereoscan'. There are now seven or eight manufacturers of these instruments in Europe, the USA, and Japan, and it is estimated that about 20 000 SEMs are in use worldwide.

The SEM is a mapping, rather than an imaging, device (Fig. 1) and so is a member of the same class of instruments as the facsimile machine, the scanning probe microscope, and the confocal optical microscope. The sample is probed by a beam of electrons scanned across the surface. Radiations from the specimen, stimulated by the incident beam, are detected, amplified, and used to modulate the brightness of a second beam of electrons scanned, synchronously with the first beam, across a cathode ray tube display. If the area scanned on the display tube is $A \times A$ and the corresponding area scanned on the sample is $B \times B$ then the linear magnification $M = A/B$. The magnification is therefore geometric in origin and may be changed by varying the area