TABLE OF CONTENTS

CHAPTER 1 PRINCIPLES OF IMAGE FORMATION *J.M. COWLEY*

1.1 Introduction 1
Electron Scattering and Diffration
The Physical Optics Analogy
Diffraction Patterns
Mathematical Formulation

1.2 The Abbe Theory of Imaging 7
Incident Beam Convergence
Chromatic Aberration
Mathematical Formulation

1.3 Inelastic Scattering 12

1.4 STEM and CTEM 13
STEM Imaging Modes
Mathematical Description

1.5 Thin, Weakly Scattering Specimens 18
Beam Convergence and Chromatic Aberration
Mathematical Formulation

1.6 Thin, Strongly Scattering Specimens 23
Mathematical Formulation

1.7 Thin, Periodic Objects: Crystals 25
Special Imaging Conditions
Mathematical Formulation

1.8 Thicker Crystals 30
Lattice Fringes
Mathematical Considerations

1.9 Very Thick Specimens 34
Mathematical Descriptions

1.10 Conclusions 36

Classical and General References

Other References

CHAPTER 2 INTRODUCTORY ELECTRON OPTICS *R.H. GEISS*

2.1 Introduction 43

2.2 Geometrical Optics 43
Refraction
Cardinal Elements
Real and Virtual Images
Lens Equations
Paraxial Rays

2.3 Electrostatic Lenses 49
Refraction
Action of Electrostatic Lenses
Types of Electrostatic Lenses

2.4 Magnetic Lenses 53
Action of a Homogeneous Field
Action of an Inhomogeneous Field
Paraxial Ray Equations
Bell Shaped Field
Lens Excitation Parameters ω and k^2
Cardinal Elements of Magnetic Lenses
Objective Lenses
Lens Aberrations and Defects
Special Magnetic Lenses
2.5 Prism Optics 69
Magnetic Sectors
Electrostatic Sectors
Wein Filter
2.6 Optics of the Electron Microscope 72
Introduction
Electron Gun
Condenser Lens System
Coherence
Magnification Lens Systems
2.7 Comparison of CTEM and STEM Optics 77
2.8 Conclusion
References

CHAPTER 3 PRINCIPLES OF THIN FILM X-RAY MICROANALYSIS *J.I. GOLDSTEIN*

3.1 Introduction 83
3.2 Quantitative X-ray Analysis 84
Primary Emitted X-ray Intensities
Quantitative X-ray Analysis Using the Ration Technique and Thin Film Criterion
Limitations of the Thin Film Criterion
Absorption Correction
Fluorescence Correction
3.3 Spatial Resolution 100
Analytical and Computer Models
Measurements of Spatial Resolution
3.4 Sensitivity Limits 109
3.5 Summary 117
Acknowledgments
References

CHAPTER 4 QUANTITATIVE X–RAY MICROANALYSIS: INSTURMENTAL CONSIDERATIONS AND APPLICATIONS TO MATERIALS SCIENCE *N.J. ZALUZEC*

4.1 Introduction 121
4.2 Instrumental Limitations in AEM Based X-ray Microanalysis 121
4.3 Instrumental Artifacts: Systems Background 122
Fluorescence by Uncollimated Radiation: Remote Sources
Fluorescence by Uncollimated Radiation: Local Sources
Specimen Contamination
Detector Artifacts
4.4 Optimum Experimental Conditions for X-ray Analysis 130
Detector/Specimen Geometry
Detector Collimation
Selection of Incident Beam Energy and Electron Source
Imaging and Diffraction Conditions During Analysis
Specimen Preparation Artifacts
4.5 Data Reduction for Quantitative Analysis 137

4.6 Application of Quantitative X-ray Microanalysis: 139
Parameters of Standardless Analysis
Absorption Correction
4.7 Applications of Standardless Analysis 149
Standardless Analysis Using the Thin-Film Approximation: Fe-13Cr-40Ni
Standardless Analysis Using the Absorption Correction: NiAl
4.8 Standardless Analysis in Complex Systems 155
Analysis of Totally Buried Peaks
Quantitative Analysis of Precipitated Phases
Procedures for Analysis of Radioactive Specimens
4.9 Summary
Acknowledgments
References
Tables

CHAPTER 5 EDS QUANTITATION AND APPLICATION TO BIOLOGY *T.A. Hall and B.L. GUPTA*

5.1 Introduction 169
5.2 Measurements on Thin or Ultrathin Sections Mounted on Thin Films 170
Elemental Ratios
Millimoles of Element Per Unit Volume
Millimoles of Element Per kg of Dried Tissue (Continuum Method)
Millimoles of Element Per kg Wet Weight (Continuum Method, Frozen-Hydrated Sections)
Dry-Weight and Aqueous Fractions
Conversion to mM of Element Per Litre of Water
Absorption Corrections
Standards
5.3 Effects of Contamination Within the Microscope Column 180
5.4 Effects of Beam Damage 181
5.5 Specimen Preparation 182
5.6 Specimens Other Than Sections Mounted on Thin Film 183
Main Literature References
List of Symbols Used in this Article
Subscripts
References
Appendix I
Derivation of Equation 5.12 for Dry-Weight Determination
Appendix II
Sample Calculations
Calculations

CHAPTER 6 MONTE CARLO SIMULATION IN ANALYTICAL ELECTRON MICROSCOPY *DAVID F. KYSER*

6.1 Introduction 199
6.2 Basic Physical Concepts in Monte Carlo Simulation 200
Electron Scattering
Energy Loss Between Elastic Scattering Events
Sequence of Calculations
Spatial Distribution of Energy Loss and X-ray Production
6.3 Design, Implementation, and Output of a Monte Carlo Program 206
Computer Generation and Utilization of Random Numbers
Computational Time and Its Control
Condensation and Output of Results Obtained
6.4 Applications to X-ray Microanalysis 208
Depth Distribution of X-ray Production
Total X-ray Production in Foils
Radial Distribution of X-ray Production
Electron Trajectory Plotting

6.5 Summary 219
Acknowledgments
References

CHAPTER 7 THE BASIC PRINCIPLES OF ELECTRON ENERGY LOSS SPECTROSCOPY *DAVID C. JOY*

7.1 What is Electron Energy Loss Spectroscopy? 223
7.2 What is Required? 223
7.3 Describing the Energy Loss Spectrum 225
7.4 The Micro-Analytical Information in the EEL Specimen 227
Region 1 - Around Zero-Loss
Region 2 - The Low-Loss Region
Region 3 - Higher Energy Losses
7.5 Collecting the Energy Loss Spectrum 236
7.6 Recording and Analyzing the Data 239
7.7 The Effects of Specimen Thickness 241
7.8 To Summarize 242
References

CHAPTER 8 ENERGY LOSS SPECTROMETRY FOR BIOLOGICAL RESEARCH *DALE E. JOHNSON*

8.1 Introduction 245
8.2 Characteristics of a Typical Spectrum 245
8.3 Sensitivity of ELS Techniques 247
Elemental Microanalysis
Chemical and Molecular Microanalysis
8.4 Approaches to the Quantitative Use of ELS 248
Elemental Microanalysis
Chemical Microanalysis
Molecular Microanalysis
Dielectric Constant Determination
8.5 Examples of Typical Experimental Results 249
Experimental Spectra
Low Z Elemental Mapping
Molecular Species Mapping
Extended Fine Structure (EXAFS)
8.6 Practical Limitations 253
Radiation Damage
i Mass Loss
ii Bond Scission
Specimen Thickness
i Effect on Background and Peak Heights
ii Specimen Mass Thickness Effects in Mapping
8.7 Summary 257
References
Classic References

CHAPTER 9 ELEMENTAL ANALYSIS USING INNER-SHELL EXCITATIONS: A MICROANALYTICAL TECHNIQUE FOR MATERIALS CHARACTERIZATION *DENNIS M. MAHER*

9.1 Introduction 259
9.2 Basic Considerations 260
Spectrum
Dynamic Range
Spectral Background
Edge Shapes

9.3 Progress in Quantitation 265
Analysis Methods
Method 1: Efficiency Factors
Method 2: Calculated Partial Cross Sections
Method 3: Standards
Tests of Analysis Methods
Stability of Quantitation Methods
Relative Accuracy of Atomic Ratios
Absolute Accuracy of Quantitation
Future Considerations
9.4 Elemental Identification 281
Threshold Energy
Shape Analysis
Elemental Maps
9.5 Detection Limits 285
Importance of β
Minimum Detectable Limits
9.6 Summary 287
References

CHAPTER 10 ANALYSIS OF THE ELECTRONIC STRUCTURE OF SOLIDS *JOHN SILCOX*

10.1 Introduction 295
10.2 Scattering Kinematics 296
10.3 Inner-Core Excitations 300
10.4 Valence Electron Excitations 302
Final Comments
Acknowledgments
References

CHAPTER 11 STEM IMAGING OE CRYSTALS AND DEFECTS *C.J. HUMPHREYS*

11.1 Introduction 305
11.2 Principle of Reciprocity in STEM and CTEM 306
Reciprocity of Electron Microscopes
Reciprocity and the Coherence of the Source and Detector
The Inapplicability of Reciprocity for Thick Specimens
Qualitative Reciprocity of the Top-Bottom Effect
Procedure if Reciprocity is not Applicab!e
11.3 Image Recording and Signal/Noise 310
Signal/Noise and Reciprocity
Z-Contrast Applied to Materials
11.4 The Optimum Beam Divergences for Imaging Crystal Defects 312
Typical Values of α and β in CTEM and STEM
Effects of Varying β_s on STEM Images
Two-Beam Dynamical Theory Interpretation
Choice of Optimum β_s Value
11.5 The Identification of Crystal Defects 317
Properties of Dislocation Images
Properties of Stacking Fault Images
11.6 The Breakdown of the Column Approximation in STEM 320
The Nature of the Column Approximation
High Resolution STEM Image Calculations Without the Column Approximation
11.7 Penetration in Crystals Using CTEM and STEM 323
Definition of Penetration
Factors Limiting the Penetration of CTEM (W Filament)
Penetration in STEM
The Penetration in STEM and CTEM
The Top-Bottom Effect

11.8 Current Developments in the STEM Imaging of Defects 327
Post Specimen Lenses
On-Line Optical Image Processing
Lattice Imaging
High Voltage STEM
In-Situ Imaging and Analysis
Acknowledgments
References
Classical References

CHAPTER 12 BIOLOGICAL SCANNING TRANSMISSION ELECTRON MICROSCOPY *J. WALL*

12.1 Introduction 333
12.2 Quantitative Measurement with the STEM 335
Length
Mass
Substrate Noise
Heavy Atom Signal
Resolution
Specimen Modification During Imaging
12.3 Conclusion 341
Acknowledgments
References

CHAPTER 13 ELECTRON MICROSCOPY OF INDIVIDUAL ATOMS *M. ISAACSON, M.OHTSUKI and M. UTLAUT*

13.1 Introduction 343
13.2a Basics - Electron Scattering 344
13.2b Basics - Operation 346
13.3a Practical Considerations - Electron Optics 347
Probe Formation
Further Stability Requirements
13.3b Practical Considerations - Specimen Preparation 352
Low Noise Support Films
13.3c Practical Considerations - Clean Support Films 355
Heavy Atom Contamination
13.3d Practical Considerations - Organic Contaminants 357
13.4 How to Visualize an Atom 357
13.5 Some Examples of Single Atom Microscopy 360
13.6 Conclusion 366
Acknowledgment
References

CHAPTER 14 MICRODIFFRACTION *J.B. WARREN*

14.1 Introduction 369
14.2 Focused Probe Microdiffraction 371
14.3 Focused Aperture Microdiffraction 373
14.4 Rocking Beam Microdiffraction 375
14.5 Applications 377
14.6 Summary 383
References

CHAPTER 15 CONVERGENT BEAM ELECTRON DIFFRACTION *J.W. STEEDS*

15.1 Introduction 387
Development of Convergent Beam Diffraction
The Microscope
TEM Mode
STEM Mode
Intermediate Configurations
Effects Connected with the Specimen
Beam Broading
Beam Heating
Perfection of the Specimen
Contamination
Goniometry

15.2 The Dimensional Electron Diffraction 395
Higher Order Laue Zones
Diameters of Holz Rings
Indexing and Origin of Holz Lines
i Indexing
ii Origin of Lines
Lattice Parameter Determination
Determination of the Reciprocal Lattice
Space Group Determination
Measurement of Chemical Variations and Strains

15.3 Crystal Point and Space Groups 406
Use of High Symmetry Zone Axes
Point Group Determination
Determination of the Reciprocal Lattice
Space Group Determination
Handedness of a Crystal

15.4 Atomic Arrangements 412
Intensities of Holz Reflections
Atomic String Approximation

15.5 Finger Printing Techniques 416

15.6 Crystal Potential and Thickness Determination 417

Acknowledgments

References

CHAPTER 16 RADIATION DAMAGE WITH BIOLOGICAL SPECIMENS AND ORGANIC MATERIALS *ROBERT M. GLAESER*

16.1 Introduction 423

16.2 Primary Events in Radiation Physics and Radiation Chemistry 425

16.3 Empirical Studies of Radiation Damage Effects Measured Under Conditions Used in the Electron Microscope 428

16.4 Signal-to-Noise Considerations at Safe Electron Exposures 429

16.5 Additional Processes of Radiation Damage that Occur at Very High Electron Exposures 432

Acknowledgments

References

CHAPTER 17 RADIATION EFFECTS IN ANALYSIS OF INORGANIC SPECIMENS BY TEM *L.W. HOBBS*

17.1 Introduction 437
Radiation Damage in Compact Lattices
Electron-Atom Inelastic Interaction
Electron-Beam Heating
Charge Acquisition by Insulating Specimens

17.2 Knock-On Displacement 444
Displacement Energy
Momentum Transfer
17.3 Radiolysis 450
Electronic Excitations
Energy-to-Momentum Conversion
Influence of Temperature, Impurity and Radiation Flux
17.4 Degradation Kinetics 457
17.5 Radiation-Induced Structural Changes During Analysis 460
Frenkel Defect Condensation
i Planar Aggregates
ii Volume Inclusions
Ordering and Disordering
Segregation and Precipitation
17.6 Minimizing The Effects of Irradiation 472
Reducing the Electron Dose
Reducing the Temperature
17.7 Conclusions 476
References

CHAPTER 18 BARRIERS TO AEM: CONTAMINATION AND ETCHING *J.J. HREN*

18.1 Introduction 481
18.2 Some Definitions 481
18.3 Early Observations of Contamination 482
18.4 The Nature of the Contaminant 483
18.5 Relationship Between Contamination and Etching 484
18.6 Surface Diffusion and Beam Size Effects 486
18.7 Recent Studies of Contamination and Etching 487
18.8 Summary of Phenomenological Observations 490
18.9 The Mechanisms of Contamination 491
Physiosorption of Hydrocarbon Molecules
Surface Diffusion of Hydrocarbon Molecules
Polymerization and Fragmentation of Hydrocarbon Molecules in the Electron Beam
Beam Induced Thermal Gradients
Electrical Gradients in the Surface
18.10 The Mechanisms of Etching 495
Physiosorption of a Potentially Reactive Gas
Activation of the Reactive Gas by Electrons
Specimen or Contaminant Molecules that Will React with the Excited Physiosorbed Gas
The Reactant Molecules must be Volatile
18.11 Working Solutions: Proven and Potential 497
18.12 Some Effects of Contamination and Etching on AEM 500
References

CHAPTER 19 MICROANALYSIS BY LATTICE IMAGING *ROBERT SINCLAIR*

19.1 Introduction 507
19.2 Theoretical Considerations 508
Fringe Imaging
Specimen-Related Parameters
Microscope Parameters
Multi-Beam Imaging
19.3 Experimental Procedures 515
19.4 Analysis of Fringe Images 520
19.5 Composition Determination 521
19.6 Experimental Examples 524
19.7 Future Directions 527

19.8 Summary 530
Acknowledgments
References
Note on Key References

CHAPTER 20 WEAK-BEAM MICROSCOPY *JOHN B. VANDER SANDE*

20.1 Introduction 535
20.2 Theoretical Background 536
Strong-Beam Images
Weak-Beam Images
20.3 The Practice of Weak-Beam Microscopy 539
Instrumental Needs
Establishing a Weak-Beam Condition
i The Ewald Sphere Construction for Strong-Beam Microscopy
ii The Ewald Sphere Construction for Weak-Beam Microscopy
iii Determining the Deviation Parameter, s
20.4 Applications of Weak-Beam Microscopy 543
Separation of Partial Dislocations
Dense Defect Arrays: Dislocation Dipoles, Dislocation Tangles, and Dislocation Cell Walls
Precipitation on Dislocation Lines: Second Phase Particle Interfaces
Comments
Acknowledgments
References

CHAPTER 21 THE ANALYSIS OF DEFECTS USING COMPUTER SIMULATED IMAGES *PETER HUMBLE*

21.1 Introduction 551
21.2 Theory and Computational Considerations 552
21.3 Experimental Method and the Collection of Information 559
21.4 Examples of the Use of Simulated Images in the Analysis of Defects 560
21.5 The Context of this Technique in AEM 571
References
Major References

CHAPTER 22 THE STRATEGY OF ANALYSIS *RON ANDERSON and J.N. RAMSEY*

22.1 Introduction 575
22.2 Where to Begin? 575
22.3 Specimen Type Strategy 576
22.4 Identification-Solving Unknown Phases and Structures with AEM Input 583
22.5 AEM and Complimentary Technique Examples 586
Al-Cu Thin Film Corrosion
Al-Cr Films and Al-Hf Films
Organic Residue on Fired Thick Film Conductors
Premature Collector - Base Breakdown
Acknowledgments
References