

Contents

1	Light Microscopy	1
1.1	Optical Principles	1
1.1.1	Image Formation	1
1.1.2	Resolution	3
1.1.2.1	Effective Magnification	5
1.1.2.2	Brightness and Contrast	5
1.1.3	Depth of Field	6
1.1.4	Aberrations	7
1.2	Instrumentation	9
1.2.1	Illumination System	9
1.2.2	Objective Lens and Eyepiece	13
1.2.2.1	Steps for Optimum Resolution	15
1.2.2.2	Steps to Improve Depth of Field	15
1.3	Specimen Preparation	15
1.3.1	Sectioning	16
1.3.1.1	Cutting	16
1.3.1.2	Microtomy	17
1.3.2	Mounting	17
1.3.3	Grinding and Polishing	19
1.3.3.1	Grinding	19
1.3.3.2	Polishing	21
1.3.4	Etching	23
1.4	Imaging Modes	26
1.4.1	Bright-Field and Dark-Field Imaging	26
1.4.2	Phase-Contrast Microscopy	27
1.4.3	Polarized-Light Microscopy	30
1.4.4	Nomarski Microscopy	35
1.4.5	Fluorescence Microscopy	37
1.5	Confocal Microscopy	39
1.5.1	Working Principles	39
1.5.2	Three-Dimensional Images	41

	References	45
	Further Reading	45
2	X-Ray Diffraction Methods	47
2.1	X-Ray Radiation	47
2.1.1	Generation of X-Rays	47
2.1.2	X-Ray Absorption	50
2.2	Theoretical Background of Diffraction	52
2.2.1	Diffraction Geometry	52
2.2.1.1	Bragg's Law	52
2.2.1.2	Reciprocal Lattice	53
2.2.1.3	Ewald Sphere	55
2.2.2	Diffraction Intensity	58
2.2.2.1	Structure Extinction	60
2.3	X-Ray Diffractometry	62
2.3.1	Instrumentation	62
2.3.1.1	System Aberrations	64
2.3.2	Samples and Data Acquisition	65
2.3.2.1	Sample Preparation	65
2.3.2.2	Acquisition and Treatment of Diffraction Data	65
2.3.3	Distortions of Diffraction Spectra	67
2.3.3.1	Preferential Orientation	67
2.3.3.2	Crystallite Size	68
2.3.3.3	Residual Stress	69
2.3.4	Applications	70
2.3.4.1	Crystal-Phase Identification	70
2.3.4.2	Quantitative Measurement	72
2.4	Wide-Angle X-Ray Diffraction and Scattering	75
2.4.1	Wide-Angle Diffraction	76
2.4.2	Wide-Angle Scattering	79
	References	82
	Further Reading	82
3	Transmission Electron Microscopy	83
3.1	Instrumentation	83
3.1.1	Electron Sources	84
3.1.1.1	Thermionic Emission Gun	85
3.1.1.2	Field Emission Gun	86
3.1.2	Electromagnetic Lenses	87
3.1.3	Specimen Stage	89
3.2	Specimen Preparation	90
3.2.1	Prethinning	91
3.2.2	Final Thinning	91
3.2.2.1	Electrolytic Thinning	91
3.2.2.2	Ion Milling	92

3.2.2.3	Ultramicrotomy	93
3.3	Image Modes	94
3.3.1	Mass–Density Contrast	95
3.3.2	Diffraction Contrast	96
3.3.3	Phase Contrast	101
3.3.3.1	Theoretical Aspects	102
3.3.3.2	Two-Beam and Multiple-Beam Imaging	105
3.4	Selected-Area Diffraction (SAD)	107
3.4.1	Selected-Area Diffraction Characteristics	107
3.4.2	Single-Crystal Diffraction	109
3.4.2.1	Indexing a Cubic Crystal Pattern	109
3.4.2.2	Identification of Crystal Phases	112
3.4.3	Multicrystal Diffraction	114
3.4.4	Kikuchi Lines	114
3.5	Images of Crystal Defects	117
3.5.1	Wedge Fringe	117
3.5.2	Bending Contours	120
3.5.3	Dislocations	122
	References	126
	Further Reading	126
4	Scanning Electron Microscopy	127
4.1	Instrumentation	127
4.1.1	Optical Arrangement	127
4.1.2	Signal Detection	129
4.1.2.1	Detector	130
4.1.3	Probe Size and Current	131
4.2	Contrast Formation	135
4.2.1	Electron–Specimen Interactions	135
4.2.2	Topographic Contrast	137
4.2.3	Compositional Contrast	139
4.3	Operational Variables	141
4.3.1	Working Distance and Aperture Size	141
4.3.2	Acceleration Voltage and Probe Current	144
4.3.3	Astigmatism	145
4.4	Specimen Preparation	145
4.4.1	Preparation for Topographic Examination	146
4.4.1.1	Charging and Its Prevention	147
4.4.2	Preparation for Microcomposition Examination	149
4.4.3	Dehydration	149
4.5	Electron Backscatter Diffraction	151
4.5.1	EBSD Pattern Formation	151
4.5.2	EBSD Indexing and Its Automation	153
4.5.3	Applications of EBSD	155
4.6	Environmental SEM	156

4.6.1	ESEM Working Principle	156
4.6.2	Applications	158
	References	160
	Further Reading	160
5	Scanning Probe Microscopy	163
5.1	Instrumentation	163
5.1.1	Probe and Scanner	165
5.1.2	Control and Vibration Isolation	165
5.2	Scanning Tunneling Microscopy	166
5.2.1	Tunneling Current	166
5.2.2	Probe Tips and Working Environments	167
5.2.3	Operational Modes	168
5.2.4	Typical Applications	169
5.3	Atomic Force Microscopy	170
5.3.1	Near-Field Forces	170
5.3.1.1	Short-Range Forces	171
5.3.1.2	van der Waals Forces	171
5.3.1.3	Electrostatic Forces	171
5.3.1.4	Capillary Forces	172
5.3.2	Force Sensors	172
5.3.3	Operational Modes	174
5.3.3.1	Static Contact Modes	176
5.3.3.2	Lateral Force Microscopy	177
5.3.3.3	Dynamic Operational Modes	177
5.3.4	Typical Applications	180
5.3.4.1	Static Mode	180
5.3.4.2	Dynamic Noncontact Mode	181
5.3.4.3	Tapping Mode	182
5.3.4.4	Force Modulation	183
5.4	Image Artifacts	183
5.4.1	Tip	183
5.4.2	Scanner	185
5.4.3	Vibration and Operation	187
	References	189
	Further Reading	189
6	X-Ray Spectroscopy for Elemental Analysis	191
6.1	Features of Characteristic X-Rays	191
6.1.1	Types of Characteristic X-Rays	193
6.1.1.1	Selection Rules	193
6.1.2	Comparison of K, L, and M Series	194
6.2	X-Ray Fluorescence Spectrometry	196
6.2.1	Wavelength Dispersive Spectroscopy	199
6.2.1.1	Analyzing Crystal	200

6.2.1.2	Wavelength Dispersive Spectra	201
6.2.2	Energy Dispersive Spectroscopy	203
6.2.2.1	Detector	203
6.2.2.2	Energy Dispersive Spectra	204
6.2.2.3	Advances in Energy Dispersive Spectroscopy	204
6.2.3	XRF Working Atmosphere and Sample Preparation	206
6.3	Energy Dispersive Spectroscopy in Electron Microscopes	207
6.3.1	Special Features	208
6.3.2	Scanning Modes	210
6.4	Qualitative and Quantitative Analysis	211
6.4.1	Qualitative Analysis	211
6.4.2	Quantitative Analysis	213
6.4.2.1	Quantitative Analysis by X-Ray Fluorescence	214
6.4.2.2	Fundamental Parameter Method	215
6.4.2.3	Quantitative Analysis in Electron Microscopy	216
	References	219
	Further Reading	219
7	Electron Spectroscopy for Surface Analysis	221
7.1	Basic Principles	221
7.1.1	X-Ray Photoelectron Spectroscopy	221
7.1.2	Auger Electron Spectroscopy	222
7.2	Instrumentation	225
7.2.1	Ultrahigh Vacuum System	225
7.2.2	Source Guns	227
7.2.2.1	X-Ray Gun	227
7.2.2.2	Electron Gun	228
7.2.2.3	Ion Gun	229
7.2.3	Electron Energy Analyzers	229
7.3	Characteristics of Electron Spectra	230
7.3.1	Photoelectron Spectra	230
7.3.2	Auger Electron Spectra	233
7.4	Qualitative and Quantitative Analysis	235
7.4.1	Qualitative Analysis	235
7.4.1.1	Peak Identification	239
7.4.1.2	Chemical Shifts	239
7.4.1.3	Problems with Insulating Materials	241
7.4.2	Quantitative Analysis	246
7.4.2.1	Peaks and Sensitivity Factors	246
7.4.3	Composition Depth Profiling	247
	References	250
	Further Reading	251

8	Secondary Ion Mass Spectrometry for Surface Analysis	253
8.1	Basic Principles	253
8.1.1	Secondary Ion Generation	254
8.1.2	Dynamic and Static SIMS	257
8.2	Instrumentation	258
8.2.1	Primary Ion System	258
8.2.1.1	Ion Sources	259
8.2.1.2	Wien Filter	262
8.2.2	Mass Analysis System	262
8.2.2.1	Magnetic Sector Analyzer	263
8.2.2.2	Quadrupole Mass Analyzer	264
8.2.2.3	Time-of-Flight Analyzer	264
8.3	Surface Structure Analysis	266
8.3.1	Experimental Aspects	266
8.3.1.1	Primary Ions	266
8.3.1.2	Flood Gun	266
8.3.1.3	Sample Handling	267
8.3.2	Spectrum Interpretation	268
8.3.2.1	Element Identification	269
8.4	SIMS Imaging	272
8.4.1	Generation of SIMS Images	274
8.4.2	Image Quality	275
8.5	SIMS Depth Profiling	275
8.5.1	Generation of Depth Profiles	276
8.5.2	Optimization of Depth Profiling	276
8.5.2.1	Primary Beam Energy	278
8.5.2.2	Incident Angle of Primary Beam	278
8.5.2.3	Analysis Area	279
	References	282
9	Vibrational Spectroscopy for Molecular Analysis	283
9.1	Theoretical Background	283
9.1.1	Electromagnetic Radiation	283
9.1.2	Origin of Molecular Vibrations	285
9.1.3	Principles of Vibrational Spectroscopy	286
9.1.3.1	Infrared Absorption	286
9.1.3.2	Raman Scattering	287
9.1.4	Normal Mode of Molecular Vibrations	289
9.1.4.1	Number of Normal Vibration Modes	291
9.1.4.2	Classification of Normal Vibration Modes	291
9.1.5	Infrared and Raman Activity	292
9.1.5.1	Infrared Activity	293
9.1.5.2	Raman Activity	295
9.2	Fourier Transform Infrared Spectroscopy	297
9.2.1	Working Principles	298

9.2.2	Instrumentation	300
9.2.2.1	Infrared Light Source	300
9.2.2.2	Beamsplitter	300
9.2.2.3	Infrared Detector	301
9.2.2.4	Fourier Transform Infrared Spectra	302
9.2.3	Examination Techniques	304
9.2.3.1	Transmittance	304
9.2.3.2	Solid Sample Preparation	304
9.2.3.3	Liquid and Gas Sample Preparation	304
9.2.3.4	Reflectance	305
9.2.4	Fourier Transform Infrared Microspectroscopy	307
9.2.4.1	Instrumentation	307
9.2.4.2	Applications	309
9.3	Raman Microscopy	310
9.3.1	Instrumentation	310
9.3.1.1	Laser Source	311
9.3.1.2	Microscope System	311
9.3.1.3	Prefilters	312
9.3.1.4	Diffraction Grating	313
9.3.1.5	Detector	314
9.3.2	Fluorescence Problem	314
9.3.3	Raman Imaging	315
9.3.4	Applications	316
9.3.4.1	Phase Identification	317
9.3.4.2	Polymer Identification	319
9.3.4.3	Composition Determination	319
9.3.4.4	Determination of Residual Strain	321
9.3.4.5	Determination of Crystallographic Orientation	322
9.4	Interpretation of Vibrational Spectra	323
9.4.1	Qualitative Methods	323
9.4.1.1	Spectrum Comparison	323
9.4.1.2	Identifying Characteristic Bands	324
9.4.1.3	Band Intensities	327
9.4.2	Quantitative Methods	327
9.4.2.1	Quantitative Analysis of Infrared Spectra	327
9.4.2.2	Quantitative Analysis of Raman Spectra	330
	References	331
	Further Reading	332
10	Thermal Analysis	333
10.1	Common Characteristics	333
10.1.1	Thermal Events	333
10.1.1.1	Enthalpy Change	335
10.1.2	Instrumentation	335
10.1.3	Experimental Parameters	336

10.2	Differential Thermal Analysis and Differential Scanning Calorimetry	337
10.2.1	Working Principles	337
10.2.1.1	Differential Thermal Analysis	337
10.2.1.2	Differential Scanning Calorimetry	338
10.2.1.3	Temperature-Modulated Differential Scanning Calorimetry	340
10.2.2	Experimental Aspects	342
10.2.2.1	Sample Requirements	342
10.2.2.2	Baseline Determination	343
10.2.2.3	Effects of Scanning Rate	344
10.2.3	Measurement of Temperature and Enthalpy Change	345
10.2.3.1	Transition Temperatures	345
10.2.3.2	Measurement of Enthalpy Change	347
10.2.3.3	Calibration of Temperature and Enthalpy Change	348
10.2.4	Applications	348
10.2.4.1	Determination of Heat Capacity	348
10.2.4.2	Determination of Phase Transformation and Phase Diagrams	350
10.2.4.3	Applications to Polymers	351
10.3	Thermogravimetry	353
10.3.1	Instrumentation	354
10.3.2	Experimental Aspects	355
10.3.2.1	Samples	355
10.3.2.2	Atmosphere	356
10.3.2.3	Temperature Calibration	358
10.3.2.4	Heating Rate	359
10.3.3	Interpretation of Thermogravimetric Curves	360
10.3.3.1	Types of Curves	360
10.3.3.2	Temperature Determination	362
10.3.4	Applications	362
	References	365
	Further Reading	365
	Index	367