

Contents

Color plates appear between pages 306 and 307.

Preface to the Third Edition	v
1 Introduction to Polymer Morphology	1
1.1 POLYMER MATERIALS	1
1.1.1 Introduction	1
1.1.2 Definitions	2
1.2 POLYMER MORPHOLOGY	3
1.2.1 Amorphous Polymers	4
1.2.2 Semicrystalline Polymers	5
1.2.3 Liquid Crystalline Polymers	7
1.2.4 Multiphase Polymers	8
1.2.5 Composites	8
1.3 POLYMER PROCESSES	8
1.3.1 Fiber and Film Formation	9
1.3.2 Extrudates and Moldings	11
1.4 POLYMER CHARACTERIZATION	17
1.4.1 General Techniques	17
1.4.2 Microscopy Techniques	18
1.4.3 Specimen Preparation Methods	19
1.4.4 Applications of Microscopy to Polymers	20
1.4.5 Emerging Microscopy Techniques	21
References	21
2 Fundamentals of Microscopy	27
2.1 INTRODUCTION	28
2.1.1 Lens-Imaging Microscopes	29
2.1.2 Scanning-Imaging Microscopes	30
2.2 OPTICAL MICROSCOPY	31
2.2.1 Introduction	31
2.2.2 Objective Lenses	32
2.2.3 Imaging Modes	32
2.2.4 Measurement of Refractive Index	34
2.2.5 Polarizing Microscopy	34

2.3 SCANNING ELECTRON MICROSCOPY	35
2.3.1 Introduction	35
2.3.2 Imaging Signals	37
2.3.3 Electron Sources	39
2.3.4 SEM Types	41
2.3.5 SEM Optimization	41
2.4 TRANSMISSION ELECTRON MICROSCOPY	42
2.4.1 Conventional TEM	42
2.4.2 Scanning TEM	43
2.4.3 Electron Diffraction	44
2.4.4 High Resolution Electron Microscopy	45
2.5 SCANNING PROBE MICROSCOPY	45
2.5.1 Introduction	45
2.5.2 Atomic Force Microscopy	47
2.5.3 SPM Probes	50
2.6 RADIATION SENSITIVE MATERIALS	51
2.6.1 SEM Operation	52
2.6.2 Low Dose TEM Operation	52
2.7 ANALYTICAL MICROSCOPY	53
2.7.1 X-ray Microanalysis	53
2.7.2 X-ray Analysis: SEM versus AEM	55
2.7.3 Elemental Mapping	55
2.8 QUANTITATIVE MICROSCOPY	56
2.8.1 Image Processing and Analysis	56
2.8.2 Three Dimensional Reconstruction	57
2.8.3 Calibration	57
2.9 DYNAMIC MICROSCOPY	59
2.9.1 Mechanical Deformation Stages	59
2.9.2 Hot and Cold Stages	60
References	60
3 Image Formation in the Microscope	67
3.1 IMAGING WITH LENSES	68
3.1.1 Basic Optics	68
3.1.2 Diffraction	68
3.1.3 Image Formation	71
3.1.4 Resolution and Contrast	72
3.1.5 Phase Contrast and Lattice Imaging	76
3.1.6 Illumination Systems	78
3.1.7 Polarized Light	80
3.2 IMAGING BY SCANNING ELECTRON BEAM	85
3.2.1 Probe Formation	85
3.2.2 Probe-Specimen Interactions	88
3.2.3 Image Formation in the SEM	92
3.2.4 Low Voltage SEM	94
3.2.5 Variable Pressure SEM	96
3.3 IMAGING IN THE ATOMIC FORCE MICROSCOPE	97
3.3.1 Microscope Components	97
3.3.2 Probe-Specimen Interaction	100

3.3.3 Contact Mode AFM	102
3.3.4 Intermittent Contact AFM	105
3.3.5 Noncontact AFM	112
3.3.6 Practical Considerations for AFM Imaging	113
3.3.7 Artifacts in SPM Imaging	114
3.4 SPECIMEN DAMAGE IN THE MICROSCOPE	118
3.4.1 Effect of Radiation on Polymers	118
3.4.2 Radiation Doses and Specimen Heating	120
3.4.3 Effects of Radiation Damage on the Image	121
3.4.4 Noise Limited Resolution	123
References	124
4 Specimen Preparation Methods	130
4.1 SIMPLE PREPARATION METHODS	132
4.1.1 Optical Preparations	132
4.1.2 SEM Preparations	133
4.1.3 TEM Preparations	133
4.1.4 SPM Preparations	140
4.2 POLISHING	142
4.2.1 Limiting Artifacts	142
4.2.2 Polishing Specimen Surfaces	143
4.3 MICROTOMY	146
4.3.1 Peelback of Fibers/Films for SEM	146
4.3.2 Microtomy for OM	147
4.3.3 Microtomy for SEM	150
4.3.4 Microtomy for TEM and SPM	150
4.3.5 Cryomicrotomy for TEM and SPM	154
4.3.6 Microtomy for SPM	158
4.3.7 Limiting Artifacts in Microtomy	160
4.4 STAINING	160
4.4.1 Introduction	160
4.4.2 Osmium Tetroxide	162
4.4.3 Ruthenium Tetroxide	166
4.4.4 Chlorosulfonic Acid and Uranyl Acetate	173
4.4.5 Phosphotungstic Acid	175
4.4.6 Ebonite	177
4.4.7 Silver Sulfide	178
4.4.8 Mercuric Trifluoroacetate	178
4.4.9 Iodine and Bromine	179
4.4.10 Summary	179
4.5 ETCHING	181
4.5.1 Solvent and Chemical Etching	181
4.5.2 Acid Etching: Overview	183
4.5.3 Permanganate Etching	184
4.5.4 Plasma and Ion Etching	188
4.5.5 Focused Ion Beam Etching	194
4.5.6 Summary	195
4.6 REPLICATION	196
4.6.1 Simple Replicas	197
4.6.2 Replication for TEM	198

4.7 CONDUCTIVE COATINGS	201
4.7.1 Coating Devices	202
4.7.2 Coatings for TEM	203
4.7.3 Coatings for SEM and STM	203
4.7.4 Artifacts	207
4.7.5 Gold Decoration	211
4.8 YIELDING AND FRACTURE	212
4.8.1 Fractography	212
4.8.2 Fracture: Standard Physical Testing	213
4.8.3 Crazing	217
4.8.4 <i>In Situ</i> Deformation	221
4.9 CRYOGENIC AND DRYING METHODS	226
4.9.1 Simple Freezing Methods	226
4.9.2 Freeze Drying	227
4.9.3 Critical Point Drying	230
4.9.4 Freeze Fracture-Etching	231
4.9.5 Cryomicroscopy	232
References	234
5 Applications of Microscopy to Polymers	248
5.1 FIBERS	250
5.1.1 Introduction	250
5.1.2 Textile Fibers	251
5.1.3 Problem Solving Applications	260
5.1.4 Industrial Fibers	267
5.1.5 High Performance Fibers	270
5.2 FILMS AND MEMBRANES	276
5.2.1 Introduction	276
5.2.2 Model Studies	278
5.2.3 Industrial Films	282
5.2.4 Flat Film Membranes	294
5.2.5 Hollow Fiber Membranes	305
5.3 ENGINEERING RESINS AND PLASTICS	308
5.3.1 Introduction	308
5.3.2 Process-Structure Considerations	311
5.3.3 Single Phase Polymers	316
5.3.4 Multiphase Polymers	321
5.3.5 Failure or Competitive Analysis	349
5.4 COMPOSITES	354
5.4.1 Introduction	354
5.4.2 Literature Review	355
5.4.3 Composite Characterization	357
5.4.4 Carbon and Graphite Fiber Composites	365
5.4.5 Particle Filled Composites	366
5.4.6 Nanocomposites	370
5.5 EMULSIONS, COATINGS AND ADHESIVES	380
5.5.1 Introduction	380
5.5.2 Emulsions and Latexes	381
5.5.3 Particle Size Measurements	385

5.5.4 Adhesives and Adhesion	386
5.5.5 Wettability and Coatings	388
5.6 HIGH PERFORMANCE POLYMERS	398
5.6.1 Introduction	398
5.6.2 Microstructure of LCPs	400
5.6.3 Molded Parts and Extrudates	403
5.6.4 High Modulus Fibers	409
5.6.5 Structure-Property Relations in LCPs	412
References	418
6 Emerging Techniques in Polymer Microscopy	435
6.1 INTRODUCTION	435
6.2 OPTICAL AND ELECTRON MICROSCOPY	436
6.2.1 Confocal Scanning Microscopy	436
6.2.2 Optical Profilometry	437
6.2.3 Birefringence Imaging	438
6.2.4 Aberration Corrected Electron Microscopy	438
6.2.5 Ion Microscopy	440
6.3 SCANNING PROBE MICROSCOPY	441
6.3.1 Chemical Force Microscopy	441
6.3.2 Harmonic Imaging	443
6.3.3 Fast Scanning SPM	444
6.3.4 Scanning Thermal Microscopy	445
6.3.5 Near Field Scanning Optical Microscopy	449
6.3.6 Automated SPM	449
6.4 THREE DIMENSIONAL IMAGING	451
6.4.1 Introduction	451
6.4.2 Physical Sectioning	452
6.4.3 Optical Sectioning	454
6.4.4 Tomography	455
6.5 ANALYTICAL IMAGING	459
6.5.1 FTIR Microscopy	459
6.5.2 Raman Microscopy	460
6.5.3 Electron Energy Loss Microscopy	461
6.5.4 X-ray Microscopy	462
6.5.5 Imaging Surface Analysis	464
References	468
7 Problem Solving Summary	478
7.1 WHERE TO START	479
7.1.1 Problem Solving Protocol	479
7.1.2 Polymer Structures	480
7.2 INSTRUMENTAL TECHNIQUES	480
7.2.1 Comparison of Techniques	480
7.2.2 Optical Techniques	484
7.2.3 SEM Techniques	485
7.2.4 TEM Techniques	486
7.2.5 SPM Techniques	487
7.2.6 Technique Selection	487

7.3 INTERPRETATION	488
7.3.1 Artifacts	489
7.3.2 Summary	492
7.4 SUPPORTING CHARACTERIZATIONS	492
7.4.1 X-ray Diffraction	493
7.4.2 Thermal Analysis	495
7.4.3 Spectroscopy	496
7.4.4 Small Angle Scattering	499
7.4.5 Summary	500
References	501
Appendices	505
Appendix I Abbreviation of Polymer Names	505
Appendix II Acronyms of Techniques	506
Appendix III Manmade Polymer Fibers	507
Appendix IV Common Commercial Polymers and Trade Names for Plastics, Films, and Engineering Resins	508
Appendix V General Suppliers of Microscopy Accessories	510
Appendix VI Suppliers of Optical and Electron Microscopes, Microanalysis Equipment, Image Analysis and Processing	512
Appendix VII Suppliers of Scanning Probe Microscopes and Related Supplies	513
Index	515