# Contents

Preface to the Second Edition xi
Preface to the First Edition xiii

## 1 The Concept of Microstructure
1.1 Microstructural Features 7
   1.1.1 Structure–Property Relationships 7
   1.1.2 Microstructural Scale 10
   1.1.3 Microstructural Parameters 19
1.2 Crystallography and Crystal Structure 24
   1.2.1 Interatomic Bonding in Solids 25
   1.2.2 Crystalline and Amorphous Phases 30
   1.2.3 The Crystal Lattice 30
Summary 42
Bibliography 46
Worked Examples 46
Problems 51

## 2 Diffraction Analysis of Crystal Structure 55
2.1 Scattering of Radiation by Crystals 56
   2.1.1 The Laue Equations and Bragg’s Law 56
   2.1.2 Allowed and Forbidden Reflections 59
2.2 Reciprocal Space 60
   2.2.1 The Limiting Sphere Construction 60
   2.2.2 Vector Representation of Bragg’s Law 61
   2.2.3 The Reciprocal Lattice 61
2.3 X-Ray Diffraction Methods 63
   2.3.1 The X-Ray Diffractometer 67
   2.3.2 Powder Diffraction–Particles and Polycrystals 73
   2.3.3 Single Crystal Laue Diffraction 76
   2.3.4 Rotating Single Crystal Methods 78
2.4 Diffraction Analysis 79
   2.4.1 Atomic Scattering Factors 80
   2.4.2 Scattering by the Unit Cell 81
   2.4.3 The Structure Factor in the Complex Plane 83
   2.4.4 Interpretation of Diffracted Intensities 84
   2.4.5 Errors and Assumptions 85
2.5 Electron Diffraction 90
   2.5.1 Wave Properties of Electrons 91
5.6.4 Electron Backscattered Diffraction Patterns 289
5.6.5 OIM Resolution and Sensitivity 291
5.6.6 Localized Preferred Orientation and Residual Stress 292

5.7 Specimen Preparation and Topology 294
5.7.1 Sputter Coating and Contrast Enhancement 295
5.7.2 Fractography and Failure Analysis 295
5.7.3 Stereoscopic Imaging 298
5.7.4 Parallax Measurements 298

5.8 Focused Ion Beam Microscopy 301
5.8.1 Principles of Operation and Microscope Construction 302
5.8.2 Ion Beam–Specimen Interactions 304
5.8.3 Dual-Beam FIB Systems 306
5.8.4 Machining and Deposition 306
5.8.5 TEM Specimen Preparation 310
5.8.6 Serial Sectioning 314

Summary 315
Bibliography 318
Worked Examples 318
Problems 326

6 Microanalysis in Electron Microscopy 333
6.1 X-Ray Microanalysis 334
6.1.1 Excitation of Characteristic X-Rays 334
6.1.2 Detection of Characteristic X-Rays 338
6.1.3 Quantitative Analysis of Composition 343

6.2 Electron Energy Loss Spectroscopy 357
6.2.1 The Electron Energy-Loss Spectrum 360
6.2.2 Limits of Detection and Resolution in EELS 361
6.2.3 Quantitative Electron Energy Loss Analysis 364
6.2.4 Near-Edge Fine Structure Information 365
6.2.5 Far-Edge Fine Structure Information 366
6.2.6 Energy-Filtered Transmission Electron Microscopy 367

Summary 370
Bibliography 375
Worked Examples 375
Problems 386

7 Scanning Probe Microscopy and Related Techniques 391
7.1 Surface Forces and Surface Morphology 392
7.1.1 Surface Forces and Their Origin 392
7.1.2 Surface Force Measurements 396
7.1.3 Surface Morphology: Atomic and Lattice Resolution 397

7.2 Scanning Probe Microscopes 400
7.2.1 Atomic Force Microscopy 403
7.2.2 Scanning Tunnelling Microscopy 410

7.3 Field-Ion Microscopy and Atom Probe Tomography 413
8 Chemical Analysis of Surface Composition 423
8.1 X-Ray Photoelectron Spectroscopy 424
  8.1.1 Depth Discrimination 426
  8.1.2 Chemical Binding States 428
  8.1.3 Instrumental Requirements 429
  8.1.4 Applications 431
8.2 Auger Electron Spectroscopy 431
  8.2.1 Spatial Resolution and Depth Discrimination 433
  8.2.2 Recording and Presentation of Spectra 434
  8.2.3 Identification of Chemical Binding States 435
  8.2.4 Quantitative Auger Analysis 436
  8.2.5 Depth Profiling 437
  8.2.6 Auger Imaging 438
8.3 Secondary-Ion Mass Spectrometry 440
  8.3.1 Sensitivity and Resolution 442
  8.3.2 Calibration and Quantitative Analysis 444
  8.3.3 SIMS Imaging 445
Summary 446
Bibliography 448
Worked Examples 448
Problems 453

9 Quantitative and Tomographic Analysis of Microstructure 457
9.1 Basic Stereological Concepts 458
  9.1.1 Isotropy and Anisotropy 459
  9.1.2 Homogeneity and Inhomogeneity 461
  9.1.3 Sampling and Sectioning 463
  9.1.4 Statistics and Probability 466
9.2 Accessible and Inaccessible Parameters 467
  9.2.1 Accessible Parameters 468
  9.2.2 Inaccessible Parameters 476
9.3 Optimizing Accuracy 481
  9.3.1 Sample Size and Counting Time 483
  9.3.2 Resolution and Detection Errors 485
  9.3.3 Sample Thickness Corrections 487
  9.3.4 Observer Bias 489
  9.3.5 Dislocation Density Revisited 490
9.4 Automated Image Analysis 491
  9.4.1 Digital Image Recording 494
  9.4.2 Statistical Significance and Microstructural Relevance 495