Contents

1 Present State of Electron Backscatter Diffraction and Prospective Developments **1**
Robert A. Schwarzer, David P. Field, Brent L. Adams, Mukul Kumar, and Adam J. Schwartz
1.1 Introduction 1
1.2 Generation and Interpretation of Electron Backscatter Diffraction Patterns 2
1.3 Experimental Set-Up of an EBSD System 3
1.4 The Components of an Automated EBSD System 4
1.4.1 The Pattern Acquisition Device 4
1.4.2 Mechanical Stage and Digital Beam Scanning 5
1.5 Spatial Resolution 7
1.6 SEM Specifications for Good EBSD Performance 9
1.7 The Radon or Hough Transformation for Band Localization . . . 11
1.8 Indexing 12
1.9 Fast EBSD 13
1.10 Ion Blocking Patterns 15
1.11 Conclusions 19

2 Dynamical Simulation of Electron Backscatter Diffraction Patterns **21**
Aimo Winkelmann
2.1 Introduction 21
2.2 Model of Electron Backscatter Diffraction 21
2.3 Dynamical Electron Diffraction in EBSD 22
2.3.1 Dynamical Electron Diffraction in EBSD 22
2.3.2 Dynamical Electron Diffraction in EBSD 23
2.3.3 Dynamical Electron Diffraction in EBSD 24
2.4 Applications 25
2.4.1 A Real-Space View of EBSD 25
2.4.2 Full Scale Simulation of EBSD Patterns 27
2.4.3 The Influence of the Energy Spectrum of the Backscattered Electrons 28
2.4.4 Dynamical Effects of Anisotropic Backscattering 30
2.5 Summary 32

3 Representations of Texture **35**
Jeremy K. Mason and Christopher A. Schuh
3.1 Introduction 35
3.2 Rotations and Orientations 36
3.2.1 Defining a Rotation 36
3.2.2 Defining an Orientation 37
3.3 Pole Figures 38
3.4 Discrete Orientations 40
3.4.1 Axis-Angle Parameters 41
3.4.2 Rodrigues Vectors 42
3.4.3 Quaternions 42
3.4.4 Euler Angles 45
3.5 Orientation Distribution Functions 46
3.5.1 Circular Harmonics 46
3.5.2 Spherical Harmonics 47
3.5.3 Hyperspherical Harmonics 48
3.5.4 Generalized Spherical Harmonics 49
3.5.5 Symmetrized Harmonics 49
3.6 Conclusion 50

4 Energy Filtering in EBSD **53**
Alwyn Eades, Andrew Deal, Abhishek Bhattacharyya, and Tejpal Hooghan
4.1 Introduction 53
4.2 Background 53
4.3 Energy Filters 54
4.4 Operating the Filter 56
4.5 Early Results 57
4.6 Patterns at Different Energies 60
4.7 Localization of the Signal 61
4.8 Future Energy Filters in EBSD 62
4.9 Summary and Conclusions 62

5 Spherical Kikuchi Maps and Other Rarities **65**
Austin P. Day
5.1 Introduction 65
5.2 Electron Backscatter Patterns 65
5.3 Spherical Kikuchi Maps 65
5.4 EBSP Detectors 65
5.5 EBSP Imaging and Uniformity 68
5.6 EBSP Simulation 68
5.7 Spherical Kikuchi Maps from EBSPs 68
5.8 Kikuchi Band Profiles 72
5.9 Spherical Kikuchi Map Inversion 74
5.10 Uses for Spherical Kikuchi Maps 75
5.11 Colour Orientation Contrast Images 76
5.12 STEM in the SEM 76
5.13 Unusual Features in EBSPs 77

6 Application of Electron Backscatter Diffraction to Phase Identification 81
Bassem El-Dasher and Andrew Deal
6.1 Introduction 81
6.2 Considerations for Phase ID with EBSD 82
6.3 Case Studies 84
6.3.1 Simultaneous EBSD/EDS Phase Discrimination 85
6.3.2 Distinguishing γ and γ' in Ni Superalloys 86
6.3.3 Volume Fraction Determination in a Multiphase Alloy . . 89

7 Phase Identification Through Symmetry Determination in EBSD Patterns 97
David J. Dingley and S.I. Wright
7.1 Introduction 97
7.2 Basis of the Phase Identification Method 97
7.3 Determination of the Crystal Unit Cell 98
7.4 Discovering the Lattice Symmetry 100
7.5 Re-Indexing the Pattern According to the Discovered Crystal Class 101
7.6 Examples 102
7.6.1 Case 1, A Cubic Crystal 102
7.6.2 Case 2, A Hexagonal Crystal 104
7.6.3 Case 3, A Trigonal Crystal 104
7.7 Discussion 106

8 Three-Dimensional Orientation Microscopy by Serial Sectioning and EBSD-Based Orientation Mapping in a FIB-SEM 109
Stefan Zaefferer and Stuart I. Wright
8.1 Introduction 109
8.2 The Geometrical Set-Up for 3D Characterisation in a FIB-SEM . 110
8.3 Automatic 3D Orientation Microscopy 113
8.4 Software for 3D Data Analysis 113
8.5 Application Examples 114
8.5.1 The 3D Microstructure and Crystallography of Pearlite Colonies 114
8.5.2 Microstructure of "Nanocrystalline" NiCo Deposits . . . 115
8.6 Discussion 119
8.6.1 Accuracy and Application Limits 119
8.6.2 Materials Issues 120
8.7 Conclusions 120

9 Collection, Processing, and Analysis of Three-Dimensional EBSD Data Sets 123
Michael A. Groeber, David J. Rowenhorst, and Michael D. Uchic
9.1 Introduction 123
9.2 Data Collection 123
9.3 Processing Strategies 124
9.3.1 Registration and Alignment of Sections 124
9.3.2 Segmentation of Grains 126
9.3.3 Clean-Up Routines 127
9.4 Analysis Capabilities 129
9.4.1 Morphological Descriptors 129
9.4.2 Crystallographic Descriptors 133
9.5 Summary 135

10 3D Reconstruction of Digital Microstructures 139
Stephen D. Sintay, Michael A. Groeber, and Anthony D. Rollett
10.1 Motivation 139
10.2 Background 139
10.2.1 2D–3D Inference 139
10.2.2 3D Polycrystal Microstructure Generation 140
10.3 Data Collection and Analysis 140
10.3.1 Data Sources 140
10.3.2 Identifying Features 141
10.3.3 Statistical Description of Features 141
10.4 Methods for 3D Structure Inference 141
10.4.1 Monte Carlo-Based Histogram Fitting 143
10.4.2 Observation-Based Domain Constraint 145
10.5 Generation of 3D Structure 147
10.5.1 Packing of Ellipsoids 147
10.5.2 Relaxation of Boundaries 149
10.6 Quality Analysis 149
10.6.1 Size Distribution Comparison 149
10.6.2 Shape Distribution Comparison 149
10.6.3 Neighborhood Comparison 151
10.6.4 Boundary Structure Comparison 151
10.7 Thoughts on Current Conditions and Future Work 151

11 Direct 3D Simulation of Plastic Flow from EBSD Data 155
Nathan R. Barton, Joel V. Bernier, Ricardo A. Lebensohn, and Anthony D. Rollett
11.1 Introduction 155
11.2 Material and Microstructural Model 156
11.2.1 Three-Dimensional Microstructure Generation 157
11.2.2 Micromechanical Model 158
11.2.3 Finite Element Model 159
11.3 Simulation Results 159
11.4 Directions for Further Computational Development 162
11.5 Conclusions 165

12 First-Order Microstructure Sensitive Design Based on Volume Fractions and Elementary Bounds 169
Surya R. Kalidindi, David T. Fullwood, and Brent L. Adams
12.1 Introduction 169
12.2 Quantification of Microstructure 170
12.3 Microstructure Sensitive Design Framework 170
12.4 Property Closures 172

13 Second-Order Microstructure Sensitive Design Using 2-Point Spatial Correlations 177
David T. Fullwood, Surya R. Kalidindi, and Brent L. Adams
13.1 Introduction 177
13.2 Definition and Properties of the 2-Point Correlation Functions . . 178
13.2.1 Boundary Conditions 179
13.2.2 Properties of the 2-Point Functions 179
13.2.3 Visualization of the 2-Point Functions 179
13.2.4 Metrics from 2-Point Correlations 180
13.2.5 Collecting 2-Point Correlations from Material Samples . 180
13.3 Structure Property Relations 181
13.3.1 Localization Tensors 182
13.3.2 Effective Tensors 184
13.4 Microstructure Design 186

14 Combinatorial Materials Science and EBSD: A High Throughput Experimentation Tool 189
Krishna Rajan
14.1 Introduction 189
14.2 Introduction to Combinatorial Methods 189
14.2.1 High Throughput EBSD Screening 190
14.2.2 Informatics and Data 194
14.3 Summary 196

15 Grain Boundary Networks 201
Bryan W. Reed and Christopher A. Schuh
15.1 Introduction 201
15.2 Measurement and Classification of Local Network Elements . . . 202
15.2.1 General Definitions for Single Boundaries 202
15.2.2 Structures with More than One Boundary 203
15.3 Geometry of the Network Structure 204
15.3.1 Percolation Measures of the Grain Boundary Network . . 205
15.3.2 Crystallographic Constraints 206
15.4 Microstructure-Property Connections 208
15.4.1 Composite Averaging vs. Percolation Theory 209
15.4.2 Crystallographic Correlations 211
15.5 Conclusions and Future Outlook 212

16 Measurement of the Five-Parameter Grain Boundary Distribution from Planar Sections 215
Gregory S. Rohrer and Valerie Randle
16.1 Introduction: Grain Boundary Planes and Properties 215
16.2 Serial Sectioning 216
16.3 Single-Surface Trace Analysis 217
16.4 Five-Parameter Stereological Analysis 218
16.4.1 Parameterization and Discretization of the Space of Grain Boundary Types 218
16.4.2 Measurement of the Grain Boundary Characterization Distribution 219
16.4.3 Performance of the Stereological Analysis 221
16.4.4 Comparison GBCDs Measured Stereologically and by Serial Sectioning in the Dual Beam FIB 223
16.5 Examples of Five-Parameter Analyses 224

17 Strain Mapping Using Electron Backscatter Diffraction 231
Angus J. Wilkinson, David J. Dingley, and Graham Meaden
17.1 Introduction 231
17.1.1 The Need for Local Strain Assessment 231
17.1.2 Competing Strain Mapping Techniques 231
17.1.3 Review of Applications of EBSD to Analysis of Elastic Strains 232
17.2 Cross-Correlation-Based Analysis of EBSD Patterns 234
17.2.1 Geometry: Linking Pattern Shifts to Strain 234
17.2.2 Pattern Shift Measurement 235
17.2.3 Sensitivity Analysis 237
17.2.4 Illustrative Applications 239
17.3 Concluding Remarks 247

18 Mapping and Assessing Plastic Deformation Using EBSD 251
Luke N. Brewer, David P. Field, and Colin C. Merriman
18.1 Plastic Deformation Effects on the EBSD Pattern and Orientation Map 251
18.2 Pattern Rotation Approaches 253
18.2.1 Mapping Orientations and Misorientations 253
18.2.2 Average Misorientation Approaches 255
18.2.3 Measurement and Calculation of GND Densities 258

19 Analysis of Deformation Structures in FCC Materials Using EBSD and TEM Techniques 263
Oleg V. Mishin, Andrew Godfrey, and Dorte Juul Jensen
19.1 Introduction 263
19.2 Orientation Noise in EBSD Data 265
19.2.1 A Quantitative Description of Orientation Noise 265
19.2.2 Postprocessing Orientation Filtering Operations 266
19.3 Quantitative TEM–EBSD Comparison 268
19.4 Heterogeneity in Microstructural Refinement 271

19.4.1 Analysis of Local Heterogeneity . . . 271
19.4.2 Potential for Analysis of Large-Scale Heterogeneities . . 272
19.5 Summary and Conclusions . . . 273

20 Application of EBSD Methods to Severe Plastic Deformation (SPD) and Related Processing Methods . . . 277
Terry R. McNelley, Alexandre P. Zhilyaev, Srinivasan Swaminathan, Jianqing Su, and E. Sarath Menon
20.1 Introduction . . . 277
20.2 Microstructures During the Initial ECAP Pass . . . 278
20.3 Microstructures Developed by Machining . . . 282
20.4 Grain Refinement During FSP . . . 284
20.5 Conclusions . . . 288

21 Applications of EBSD to Microstructural Control in Friction Stir Welding/Processing . . . 291
Sergey Mironov, Yutaka S. Sato, and Hiroyuki Kokawa
21.1 Introduction . . . 291
21.2 Brief Explanations of FSW/P Terminology . . . 292
21.3 Microstructural Evolution . . . 292
21.4 Material Flow . . . 296
21.5 Structure-Properties Relationship . . . 298
21.6 Summary and Future Outlook . . . 299

22 Characterization of Shear Localization and Shock Damage with EBSD . . . 301
John F. Bingert, Veronica Livescu, and Ellen K. Cerreta
22.1 Introduction . . . 301
22.2 Shear Localization . . . 302
22.2.1 Constrained Shear in Pure Fe—Shear Zone Geometry . . 302
22.2.2 Constrained Shear in Pure Fe—Texture Development . . 306
22.2.3 Effect of Morphology on Grain Instability in Cu . . . 307
22.3 Shock Loading Damage in Tantalum . . . 309
22.3.1 Effect of Shock Duration on Incipient Spall Structure . . 310
22.3.2 Effect of Pressure on Incipient Spall Structure . . . 313
22.4 Conclusions . . . 313

23 Texture Separation for α/β Titanium Alloys . . . 317
Ayman A. Salem
23.1 Introduction . . . 317
23.2 Microstructure of α/β Titanium Alloys . . . 317
23.3 Texture of Ti-6Al-4V . . . 318
23.3.1 Separation of Primary and Secondary Alpha Texture . . . 319
23.3.2 EBSD + BSE Imaging Technique . . . 319
23.3.3 EBSD or XRD + Heat Treatment Technique . . . 320
23.4 Texture Separation Using EBSD + EDS Technique . . . 320
23.4.1 Texture Separation Using EBSD + EDS Technique . . . 320
23.4.2 Microstructure Observations . . . 321
23.4.3 Chemical Composition Maps (EDS) . . . 321
23.5 Industrial Application: Controlling Texture During Hot-Rolling of Ti-6Al-4V . . . 322

23.5.1 Microstructure Evolution . . . 323
23.5.2 Overall Texture Evolution . . . 323
23.5.3 Primary-Alpha (α_p) Textures . . . 324
23.5.4 Secondary-Alpha (α_s) Texture . . . 325
23.6 Industrial Application: Controlling Texture During Hot-Rolling of Ti-6Al-4V . . . 326

24 A Review of In Situ EBSD Studies . . . 329
Stuart I. Wright and Matthew M. Nowell
24.1 Introduction . . . 329
24.2 In Situ Postmortem Experiments . . . 330
24.3 Deformation Stage Experiments . . . 331
24.4 Heating Stage Experiments . . . 332
24.4.1 Phase Transformation . . . 332
24.4.2 Recrystallization and Grain Growth . . . 333
24.5 Combined Heating and Tensile Stage Experiments . . . 335
24.6 Conclusions . . . 335

25 Electron Backscatter Diffraction in Low Vacuum Conditions . . . 339
Bassem S. El-Dasher and Sharon G. Torres
25.1 Introduction . . . 339
25.2 Considerations for Low Vacuum EBSD . . . 340
25.3 Example Applications . . . 341
25.3.1 Microstructural Analysis of AlN-TiB_2 Ceramic Composite . . . 341
25.3.2 Characterization of $CaHPO_4 \cdot 2H_2O$ Single Crystals . . . 342

26 EBSD in the Earth Sciences: Applications, Common Practice, and Challenges . . . 345
David J. Prior, Elisabetta Mariani, and John Wheeler
26.1 Development of EBSD in Earth Sciences . . . 345
26.2 Current Practice, Capabilities, and Limitations . . . 346
26.2.1 Range of Materials and Preparation . . . 346
26.2.2 Speed of Data Collection . . . 347
26.2.3 Spatial Resolution . . . 347
26.2.4 Misindexing . . . 348
26.2.5 Polyphase Samples . . . 350
26.3 Application of EBSD in Earth Sciences . . . 351
26.3.1 Rock Deformation and Solid Earth Geophysics . . . 352
26.3.2 Metamorphic Processes . . . 355
26.3.3 Meteorites . . . 356
26.3.4 Other Areas . . . 356
26.4 Conclusions . . . 357

27 Orientation Imaging Microscopy in Research on High Temperature Oxidation 361
Bae-Kyun Kim and Jerzy A. Szpunar
27.1 Introduction 361
27.2 High Temperature Oxidation 362
27.3 Experimental Procedure 363
27.3.1 Oxidation of Samples and Oxide Formation 363
27.3.2 Sample Preparation and Geometry in OIM 364
27.3.3 Microstructure and Texture Measurement 365
27.3.4 Oxidation of Low Carbon Steel 365
27.4 Results and Discussion 368
27.4.1 Grain Growth in Iron Oxide 368
27.4.2 Effect of the Oxidation Process on Microstructure 371
27.4.3 Oxidation of Pure Iron 373
27.5 Cracks and Defects 384
27.6 Conclusion 390

Index 395