Contents

List of Contributor  XVII
Preface to Second Edition  XXIII

Part I  General  1

1  Microstructure and Properties of Engineering Materials  3
   Helmut Clemens, Svea Mayer, and Christina Scheu
   1.1  Introduction  3
   1.2  Microstructure  4
   1.2.1  Crystal Defects  7
   1.2.2  Grain (Phase) Boundaries and Twins  7
   1.2.3  Precipitates and Dispersions  8
   1.3  Microstructure and Properties  10
   1.4  Microstructural Characterization  12
   References  19

2  Internal Stresses in Engineering Materials  21
   Anke Kaysser-Pyzalla
   2.1  Definition  21
   2.1.1  Stress Tensor, Strain Tensor, and Elasticity Tensor  21
   2.1.1.1  Stress Tensor  21
   2.1.1.2  Strain Tensor  22
   2.1.2  Definitions, Residual Stresses  23
   2.1.2.1  Stress Equilibrium  23
   2.1.2.2  Residual Macro- and Microstresses  24
   2.2  Origin of Residual Macro- and Microstresses  25
   2.2.1  Residual Stress Formation in Primary Forming Processes  26
   2.2.2  Residual Stress Formation in Heat Treatment Processes  28
   2.2.2.1  Residual Stresses in a Material without Phase Transformation (Pure Cooling Residual Stresses)  29
   2.2.2.2  Residual Stresses in a Material with Phase Transformation  30
   2.2.2.3  Residual Stress Formation in Surface-Hardening Processes (Nitriding, Carbo-Nitriding, and Case Hardening)  32
   2.2.3  Residual Stress Formation in Forming Processes  32
2.2.3.1 Deep-Rolling Residual Stresses 33
2.2.3.2 Cold Extrusion Residual Stresses 34
2.2.4 Residual Stress Formation in Metal Cutting Manufacturing 36
2.2.4.1 Grinding Residual Stresses 37
2.2.5 Residual Stress Formation in Joining Processes 39
2.2.6 Residual Stress Formation in Coatings 43
2.3 Relevance 45
2.3.1 Failure due to Residual Stress Formation or Residual Stress Relief Induced by Temperature Changes 46
2.3.2 Influence of Residual Stresses on Component Failure under Static and Dynamic Mechanical Loads 47
2.3.3 Influence of Residual Stresses on Component Failure in Corrosive Environments 49
2.3.4 Influence of Residual Stresses on Wear 49
References 51

3 Textures in Engineering Materials 55
Heinz G. Brokmeier and Sangbong Yi
3.1 Introduction 55
3.2 Measurement of Preferred Orientations 58
3.3 Presentation of Preferred Orientations 59
3.3.1 Pole Figure 60
3.3.2 Orientation Distribution Function 62
3.3.3 Inverse Pole Figures 62
3.4 Interpretation of Textures 62
3.5 Errors 67
3.5.1 Grain Statistics 67
3.5.2 Pole Figure Coverage 68
References 71

4 Physical Properties of Photons and Neutrons 73
Andreas Schreyer
4.1 Introduction 73
4.2 Interaction of X-ray Photons and Neutrons with Individual Atoms 74
4.2.1 Neutrons 75
4.2.2 X-rays 76
4.3 Scattering of X-ray Photons and Neutrons from Ensembles of Atoms 79
Acknowledgment 81
References 81

5 Radiation Sources 83
Ina Lommatzsch, Wolfgang Knop, Philipp K. Pranzas, and Peter Schreiner
5.1 Generation and Properties of Neutrons 83
5.1.1 Introduction 83
5.1.2 Generation of Neutrons 83
5.1.2.1 Research Reactors 83
5.1.2.2 Spallation Sources 87
5.1.3 Instrumentation 87
5.1.3.1 Structure Research 89
5.1.3.2 Large-Scale Structures 89
5.1.3.3 Spectroscopy 89
5.1.3.4 Imaging and Analysis 89
5.1.3.5 Particle Physics 89

References 90

5.2 Production and Properties of Synchrotron Radiation 90
Rolf Treusch

5.2.1 Introduction 90
5.2.2 Properties of Synchrotron Radiation 92
5.2.3 Sources of Synchrotron Radiation 96
5.2.3.1 Bending Magnets 97
5.2.3.2 Wigglers and Undulators 98
5.2.4 Outlook: Free Electron Lasers 100
5.2.5 Summary 102

References 102

Part II Methods 105

6 Stress Analysis by Angle-Dispersive Neutron Diffraction 107
Peter Staron

6.1 Introduction 107
6.2 Diffractometer for Residual Stress Analysis 108
6.2.1 Setup of a Diffractometer for Strain Scanning 108
6.2.2 Monochromator 109
6.2.3 Slit System 110
6.2.4 Sample Positioning 111
6.2.5 Detector 111
6.3 Measurement and Data Analysis 112
6.3.1 Gage Volume and Sample Positioning 112
6.3.2 Data Reduction and Analysis 113
6.3.2.1 Data Reduction and Peak Fitting 113
6.3.2.2 Calculation of Stresses 114
6.3.2.3 Macro and Microstresses 115
6.3.2.4 Stress-Free Reference 116
6.4 Examples 116
6.4.1 Residual Stresses in Friction Stir Welded Aluminum Sheets 116
6.4.2 Residual Stresses in Water-Quenched Turbine Disks 117
6.5 Summary and Outlook 120

References 120

7 Stress Analysis by Energy-Dispersive Neutron Diffraction 123
Javier Santisteban

7.1 Introduction 123
7.2 Time-of-Flight Neutron Diffraction 123
7.2.1 TOF Peak Shape and Data Analysis Packages 124
7.3 TOF Strain Scanners 126
7.3.1 Counting Times and Resolution 128
7.3.2 Neutron Optics and Time Focusing 130
7.4 A Virtual Laboratory for Strain Scanning 131
7.5 Type II Stresses: Evolution of Intergranular Stresses 134
7.6 Type III Stresses: Dislocation Densities 135
7.7 Strain Imaging by Energy-Dispersive Neutron Transmission 138
7.8 Conclusions 140
Acknowledgments 141
References 141

8 Residual Stress Analysis by Monochromatic High-Energy X-rays 145
René V. Martins
8.1 Basic Setups 145
8.2 Principle of Slit Imaging and Data Reconstruction 148
8.3 The Conical Slit 149
8.3.1 Working Principle 149
8.3.2 Capabilities 149
8.3.3 Example 151
8.4 The Spiral Slit 152
8.4.1 Functional Principle 152
8.4.2 Capabilities 152
8.4.3 Example 153
8.5 Simultaneous Strain Measurements in Individual Bulk Grains 155
8.6 Coarse Grain Effects 156
8.7 Analysis of Diffraction Data from Area Detectors 157
8.8 Matrix for Comparison and Decision Taking Which Technique to Use for a Specific Problem 158
References 159

9 Residual Stress Analysis by Energy-Dispersive Synchrotron X-ray Diffraction 161
Christoph Genzel and Manuela Klaus
9.1 Introduction 161
9.2.1 The Basic Equation of Energy-Dispersive X-ray Diffraction 162
9.2.2 Near-Surface Depth Profiling in the Energy-Dispersive Diffraction Mode 162
9.2.3 Principles of Depth-Resolved X-ray Stress Analysis and Application to the Energy-Dispersive Case of Diffraction 164
9.3 Experimental Setup 167
9.4 Examples for Energy-Dispersive Stress Analysis 168
9.4.1 Near Surface Residual Stress Depth Profiling 168
9.4.2 Fast In situ Stress Analysis by Means of Energy-Dispersive Diffraction 171
9.5 Final Remarks 173
References 175
Contents

10 Texture Analyses by Synchrotron X-rays and Neutrons 179
Sangbong Yi, Weimin Gan, and Heinz G. Brokmeier
10.1 Texture Measurements on Laboratory Scale 179
10.1.1 X-ray Diffraction 179
10.1.2 Electron Diffraction 181
10.2 Texture Measurements at Large Scale Facilities 182
10.2.1 Neutron Diffraction 182
10.2.2 Texture Analysis Using Synchrotron X-rays 185
10.2.3 Examples of Texture Analyses Using Neutrons and Synchrotron X-rays 189
10.2.3.1 Local Texture Measurement of a Friction Welded Rod 189
10.2.3.2 Global Texture in Cu Wire 190
10.2.3.3 In situ Texture Measurement of Steel at Elevated Temperature 191
10.2.3.4 In situ Texture Measurement under Loading 192
10.3 Conclusion 193
References 194

11 Basics of Small-Angle Scattering Methods 197
Philipp K. Pranzas
11.1 Introduction 197
11.2 Common Features of a SAS Instrument 197
11.3 Contrast 198
11.4 Scattering Curve 198
11.5 Power Law/Scattering by Fractal Systems 200
11.6 Guinier and Porod Approximations 201
11.7 Macroscopic Differential Scattering Cross-section 202
11.8 Model Calculation of Size Distributions 202
11.9 Magnetic Structures 203
References 204

12 Small-Angle Neutron Scattering 207
Philipp K. Pranzas and André Heinemann
12.1 Introduction 207
12.2 Nanocrystalline Magnesium Hydride for the Reversible Storage of Hydrogen 208
12.3 Precipitates in Steel 210
12.4 SiO₂ Nanoparticles in a Polymer Matrix – An Industrial Application 213
12.5 Green Surfactants 213
Acknowledgments 215
References 215

13 Anomalous Small-Angle X-ray Scattering 217
Ulla Vainio
13.1 Introduction 217
13.2 Theory 218
13.2.1 Scattering Power of Elements 218
13.2.2 Contrast Variation 219
<table>
<thead>
<tr>
<th>Contents</th>
</tr>
</thead>
<tbody>
<tr>
<td>13.2.3 Partial Structure Factor Formalism</td>
</tr>
<tr>
<td>13.2.4 Model-Dependent ASAXS</td>
</tr>
<tr>
<td>13.2.5 Subtraction Method</td>
</tr>
<tr>
<td>13.3 Experiments</td>
</tr>
<tr>
<td>13.4 Example: ASAXS on Catalyst Nanoparticles</td>
</tr>
<tr>
<td>13.5 Summary and Outlook</td>
</tr>
<tr>
<td>References</td>
</tr>
<tr>
<td>14 Imaging</td>
</tr>
<tr>
<td>Wolfgang Treimer</td>
</tr>
<tr>
<td>14.1 Radiography</td>
</tr>
<tr>
<td>14.1.1 Fundamentals</td>
</tr>
<tr>
<td>14.1.2 Interactions of Neutrons with Matter</td>
</tr>
<tr>
<td>14.1.3 Geometries</td>
</tr>
<tr>
<td>14.1.4 Resolution Functions</td>
</tr>
<tr>
<td>14.1.5 Image Degradation</td>
</tr>
<tr>
<td>14.1.6 Other Imaging Techniques</td>
</tr>
<tr>
<td>14.1.6.1 Energy Dispersive Radiography</td>
</tr>
<tr>
<td>14.1.6.2 Real-Time Radiography</td>
</tr>
<tr>
<td>14.1.6.3 Phase Contrast Radiography</td>
</tr>
<tr>
<td>14.2 Tomography</td>
</tr>
<tr>
<td>14.2.1 Mathematical Introduction</td>
</tr>
<tr>
<td>14.2.2 Slice Theorem, Shannon Theorem</td>
</tr>
<tr>
<td>14.2.3 Image Reconstruction</td>
</tr>
<tr>
<td>14.3 New Developments in Neutron Tomography</td>
</tr>
<tr>
<td>14.3.1 Refraction</td>
</tr>
<tr>
<td>14.3.2 Ultra-Small Angle Neutron Tomography</td>
</tr>
<tr>
<td>14.3.3 Radiography and Tomography with Polarized Neutrons</td>
</tr>
<tr>
<td>References</td>
</tr>
<tr>
<td>15 Neutron and Synchrotron-Radiation-Based Imaging for Applications in Materials Science – From Macro- to Nanotomography</td>
</tr>
<tr>
<td>Felix Beckmann</td>
</tr>
<tr>
<td>15.1 Introduction</td>
</tr>
<tr>
<td>15.1.1 Attenuation-Contrast Projections</td>
</tr>
<tr>
<td>15.1.2 Phase-Contrast Projections</td>
</tr>
<tr>
<td>15.1.3 Phase-Enhanced Projections</td>
</tr>
<tr>
<td>15.1.4 Direct Phase-Contrast Projections</td>
</tr>
<tr>
<td>15.1.5 Indirect Phase-Contrast Projections</td>
</tr>
<tr>
<td>15.2 Parallel-Beam Tomography</td>
</tr>
<tr>
<td>15.2.1 Measurement and Reconstruction</td>
</tr>
<tr>
<td>15.2.2 Density Resolution and Detector Quality</td>
</tr>
<tr>
<td>15.2.3 Data Evaluation and Visualization</td>
</tr>
<tr>
<td>15.3 Macrotopography Using Neutrons</td>
</tr>
<tr>
<td>15.3.1 Experimental Setup</td>
</tr>
<tr>
<td>15.3.2 Measurements and Results</td>
</tr>
</tbody>
</table>
15.4 Microtomography Using Synchrotron Radiation 264
15.4.1 Beamline Optics 265
15.4.2 Experimental Setup 267
15.5 Summary and Outlook 271
References 271

16 μ-Tomography of Engineering Materials 275
Astrid Haibel and Julia Herzen
16.1 Introduction 275
16.2 Advantage of Synchrotron Tomography 275
16.3 Applications and 3D Image Analysis 276
16.3.1 Discharging Processes in Alkaline Cells 276
16.3.2 Microstructural Investigations of Nb₃Sn Multi-filamentary Superconductor Wires 278
16.3.3 Influence of the Foaming Agent on Metallic Foam Structures 280
16.3.4 Ex vivo Grating-Based Phase Contrast Imaging of Human Carotid Arteries 281
16.4 Image Artifacts 282
16.4.1 Ring Artifacts 282
16.4.2 Image Noise 284
16.4.3 Edge Artifacts 284
16.4.4 Motion Artifacts 285
16.4.5 Centering Errors of the Rotation Axis 286
16.5 Summary 286
References 286

Part III New and Emerging Methods 291

17 3D X-ray Diffraction Microscope 293
Henning F. Poulsen, Wolfgang Ludwig, and Søren Schmidt
17.1 Basic Setup and Strategy 294
17.1.1 The 3DXRD Microscope 296
17.2 Indexing and Characterization of Average Properties of Each Grain 296
17.2.1 Application I: Nucleation and Growth Studies 297
17.2.2 Application II: Plastic Deformation 298
17.2.3 Application III: Studies of Subgrains and Nanocrystalline Materials 299
17.3 Mapping of Grains and Orientations 300
17.3.1 Mode III: Mapping Grains in Undeformed Specimens 300
17.3.2 Mode IV: Mapping Orientations in Deformed Specimens 301
17.3.3 Application I: Recrystallization 302
17.3.4 Application II: Grain Growth 303
17.4 Combining 3DXRD and Tomography 304
17.4.1 Grain Mapping by Tomography 304
17.5 Outlook 305
References 306
18  3D Micron-Resolution Laue Diffraction 309
   Gene E. Ice
18.1  Introduction 309
18.2  The Need for Polychromatic Microdiffraction 309
18.3  Theoretical Basis for Advanced Polychromatic Microdiffraction 311
18.3.1  Modified Ewald’s Sphere Description of Laue Diffraction 311
18.3.2  Qualitative Information: Phase, Texture, Elastic Strain, Dislocation Density 312
18.3.2.1  Phase 312
18.3.2.2  Texture 312
18.3.2.3  Dislocation Tensor 313
18.3.2.4  Elastic Strain Tensor 313
18.4  Technical Developments for an Automated 3D Probe 313
18.4.1  Source 313
18.4.2  Microbeam Monochromator 315
18.4.3  Nondispersive Focusing Optics 316
18.4.4  Area Detector 317
18.4.5  Differential Aperture 317
18.4.6  Software 317
18.5  Research Examples 318
18.5.1  3D Grain Boundary Networks 319
18.5.2  Deformation Behavior and Grain Boundaries 319
18.5.3  Deformation in Single Crystals 321
18.5.4  Grain Growth on Surfaces and in Three Dimensions 321
18.5.5  Anomalous Grain Growth 321
18.6  Future Prospects and Opportunities 324
   Acknowledgment 324
   References 325

Part IV  Applications 327

19  The Use of Neutron and Synchrotron Research for Aerospace and Automotive Materials and Components 329
   Wolfgang Kaysser, Jörg Eßlinger, Volker Abetz, Norbert Huber, Karl U. Kainer, Thomas Klassen, Florian Pyczak, Andreas Schreyer, and Peter Staron
19.1  Introduction 329
19.2  Commercial Passenger Aircraft 331
19.2.1  Reduction of Airframe Weight of Commercial Passenger Aircrafts 332
19.2.1.1  Welding Commercial Passenger Aircraft Frames: Reactions, Microstructure Development, and Mechanical Properties 332
19.2.1.2  Welding Commercial Passenger Aircraft: Residual Stresses and Stress Modification 333
19.2.1.3  Welding of Commercial Passenger Aircraft: Fatigue Crack Growth 335
19.2.1.4 Weight Reduction of Aircraft by Polymers and Polymer Matrix-Based Composites 335
19.2.2 Aero-Engines 337
19.2.2.1 Metallic Materials to Improve the Thrust-to-Weight Ratio of Jet Aero-Engines 338
19.2.2.2 Thermal Barrier Coatings to Enhance the Thrust-to-Weight Increase of the Aero-Engine 340
19.3 The Light-Duty Automotive Vehicle 341
19.3.1 The Optimized Light-Duty Car Body 343
19.3.1.1 Lightweight Metallic Materials for Lightweight Car Bodies 343
19.3.1.2 Optimized Joining Processes for Automotive Applications 345
19.3.2 The Automotive Power Train and the Propulsion System of Light-Duty Cars 346
19.3.2.1 Residual Stresses in Components 347
19.3.2.2 Wear and Lubrication 348
19.3.2.3 Polymeric Membranes for Fuel Cells (PEMFCs) 349
19.3.2.4 Nanocrystalline Metal Hydrides for Hydrogen Storage 350
19.4 Other Transport Systems 352
References 353

20 In situ Experiments with Synchrotron High-Energy X-rays and Neutrons 365
Peter Staron, Torben Fischer, Thomas Lippmann, Andreas Stark, Shahrokh Daneshpour, Dirk Schnubel, Eckart Uhlmann, Robert Gerstenberger, Bettina Camin, Walter Reimers, Elisabeth Eidenberger-Schober, Helmut Clemens, Norbert Huber, and Andreas Schreyer
20.1 Introduction 365
20.2 In situ Dilatometry 366
20.2.1 Motivation 366
20.2.2 FlexiTherm 366
20.2.3 Results 367
20.3 In situ Study on Single Overload of Fatigue-Cracked Specimens 368
20.3.1 Motivation 368
20.3.2 Experimental 369
20.3.3 Results 369
20.4 In situ Cutting Experiment 370
20.4.1 Motivation 370
20.4.2 Experiment 371
20.4.3 Results 371
20.5 In situ Study of Precipitation Kinetics Using Neutrons 372
20.5.1 Motivation 372
20.5.2 Experimental Details 372
20.5.3 Results 373
20.6 Conclusions 373
References 374
21 Application of Photons and Neutrons for the Characterization and Development of Advanced Steels  377
Elisabeth Eidenberger-Schober, Ronald Schnitzer, Gerald A. Zickler, Michael Eidenberger-Schober, Michael Bischof, Peter Staron, Harald Leitner, Andreas Schreyer, and Helmut Clemens

21.1 Introduction  377
21.2 Characterization Using Synchrotron Radiation  378
   21.2.1 Ex situ and In situ High-Energy X-ray Diffraction (HE-XRD) during Heating  378
   21.2.2 Small-Angle X-ray Scattering (SAXS)  380
   21.2.3 In situ High-Energy X-ray Diffraction under Tensile Loading  381
21.3 Characterization Using Small-Angle Neutron Scattering (SANS)  382
   21.3.1 Use of SANS to Study Precipitates in Steels  382
   21.3.1.1 Analysis of Secondary Hardening Carbides  382
   21.3.1.2 Analysis of Intermetallic Precipitates  383
   21.3.2 In situ SANS during Continuous and Isothermal Aging  386
   21.3.3 SANS with Variable Magnetic Field  388
21.4 Conclusions  388
References  390

22 The Contribution of High-Energy X-rays and Neutrons to Characterization and Development of Intermetallic Titanium Aluminides  395
Thomas Schmoelzer, Klaus-Dieter Liss, Peter Staron, Andreas Stark, Emanuel Schwaighofer, Thomas Lippmann, Helmut Clemens, and Svea Mayer

22.1 Introduction  395
22.2 High-Energy X-rays and Neutrons  396
22.3 In situ Investigation of Phase Evolution  398
   22.3.1 General Aspects  398
   22.3.2 Phase Evolution in β/γ-Alloys  399
   22.3.3 Formation and Identification of a Transition Phase  401
   22.3.4 Formation of Lamellar Microstructure  405
22.4 Atomic Order and Disorder in TiAl Alloys  409
22.5 Recovery and Recrystallization during Deformation of TiAl  412
   22.5.1 General Aspects  412
   22.5.2 Analysis of Diffraction Data  414
   22.5.3 Hot Deformation of a Multi-phase Alloy  415
22.6 Lattice Parameter and Thermal Expansion  418
22.7 Conclusions  419
References  420

23 In situ µLaue: Instrumental Setup for the Deformation of Micron Sized Samples  425
Christoph Kirchlechner, Jozef Keckes, Jean S. Micha, and Gerhard Dehm

23.1 Introduction  425
23.1.1 µLaue Diffraction, a Short Introduction  426
23.2 Experimental Instrumentation  427
23.2.1 The Straining Device  427
23.2.2 The Synchrotron Beamline  428
23.2.3 The Experiment  429
23.2.4 Data Analysis  430
23.2.5 Example: In situ Deformation of a Copper Pillar  430
23.3 Discussion  433
23.3.1 Deformation Behavior of the Pillar  433
23.3.2 Tails of the Primary Beam  433
23.3.3 Sample Movements during Deformation  435
23.3.4 Streaking of Laue Patterns  435
23.4 Conclusion  436
Acknowledgments  436
References  436

24 Residual Stresses in Thin Films and Coated Tools: Challenges and Strategies for Their Nondestructive Analysis by X-ray Diffraction Methods  439
Manuela Klaus and Christoph Genzel

24.1 Introduction  439
24.2 Compilation of Approaches to Meet the Challenges in Thin Film X-ray Stress Analysis (XSA)  441
24.2.1 Stress Analysis under Grazing and Glancing Diffraction Conditions  441
24.2.2 Separation of Residual Stress and Composition Gradients  444
24.3 Final Remarks and Recommendations  447
References  448

Index  451