

Contents

List of Contributor *XVII*

Preface to Second Edition *XXIII*

Part I General *I*

- 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
	<i>Heinz G. Brokmeier and Sangbong Yi</i>	
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
	<i>Andreas Schreyer</i>	
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
5.1	Generation and Properties of Neutrons	83
	<i>Ina Lommatzsch, Wolfgang Knop, Philipp K. Pranzas, and Peter Schreiner</i>	
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
	<i>Rolf Treusch</i>	
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
	<i>Peter Staron</i>	
6.1	Introduction	107
6.2	Diffractionmeter for Residual Stress Analysis	108
6.2.1	Setup of a Diffractionmeter 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
	<i>Javier Santisteban</i>	
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
	<i>René V. Martins</i>	
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
	<i>Christoph Genzel and Manuela Klaus</i>	
9.1	Introduction	161
9.2	Fundamentals of Energy-Dispersive X-ray Diffraction Stress Analysis	162
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 <i>In situ</i> Stress Analysis by Means of Energy-Dispersive Diffraction	171
9.5	Final Remarks	173
	References	175

10	Texture Analyses by Synchrotron X-rays and Neutrons	179
	<i>Sangbong Yi, Weimin Gan, and Heinz G. Brokmeier</i>	
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	<i>In situ</i> Texture Measurement of Steel at Elevated Temperature	191
10.2.3.4	<i>In situ</i> Texture Measurement under Loading	192
10.3	Conclusion	193
	References	194
11	Basics of Small-Angle Scattering Methods	197
	<i>Philipp K. Pranzas</i>	
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
	<i>Philipp K. Pranzas and André Heinemann</i>	
12.1	Introduction	207
12.2	Nanocrystalline Magnesium Hydride for the Reversible Storage of Hydrogen	208
12.3	Precipitates in Steel	210
12.4	SiO ₂ 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
	<i>Ulla Vainio</i>	
13.1	Introduction	217
13.2	Theory	218
13.2.1	Scattering Power of Elements	218
13.2.2	Contrast Variation	219

- 13.2.3 Partial Structure Factor Formalism 219
- 13.2.4 Model-Dependent ASAXS 221
- 13.2.5 Subtraction Method 221
- 13.3 Experiments 223
- 13.4 Example: ASAXS on Catalyst Nanoparticles 223
- 13.5 Summary and Outlook 223
 - References 224

- 14 Imaging 227**
Wolfgang Treimer
 - 14.1 Radiography 227
 - 14.1.1 Fundamentals 227
 - 14.1.2 Interactions of Neutrons with Matter 228
 - 14.1.3 Geometries 230
 - 14.1.4 Resolution Functions 232
 - 14.1.5 Image Degradation 235
 - 14.1.6 Other Imaging Techniques 236
 - 14.1.6.1 Energy Dispersive Radiography 236
 - 14.1.6.2 Real-Time Radiography 238
 - 14.1.6.3 Phase Contrast Radiography 239
 - 14.2 Tomography 240
 - 14.2.1 Mathematical Introduction 240
 - 14.2.2 Slice Theorem, Shannon Theorem 241
 - 14.2.3 Image Reconstruction 243
 - 14.3 New Developments in Neutron Tomography 244
 - 14.3.1 Refraction 246
 - 14.3.2 Ultra-Small Angle Neutron Tomography 247
 - 14.3.3 Radiography and Tomography with Polarized Neutrons 249
 - References 250

- 15 Neutron and Synchrotron-Radiation-Based Imaging for Applications in Materials Science – From Macro- to Nanotomography 253**
Felix Beckmann
 - 15.1 Introduction 253
 - 15.1.1 Attenuation-Contrast Projections 253
 - 15.1.2 Phase-Contrast Projections 254
 - 15.1.3 Phase-Enhanced Projections 255
 - 15.1.4 Direct Phase-Contrast Projections 255
 - 15.1.5 Indirect Phase-Contrast Projections 255
 - 15.2 Parallel-Beam Tomography 256
 - 15.2.1 Measurement and Reconstruction 256
 - 15.2.2 Density Resolution and Detector Quality 258
 - 15.2.3 Data Evaluation and Visualization 258
 - 15.3 Macrotomography Using Neutrons 258
 - 15.3.1 Experimental Setup 258
 - 15.3.2 Measurements and Results 260

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
	<i>Astrid Haibel and Julia Herzen</i>	
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 Nb ₃ Sn Multi-filamentary Superconductor Wires	278
16.3.3	Influence of the Foaming Agent on Metallic Foam Structures	280
16.3.4	<i>Ex vivo</i> 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
	<i>Henning F. Poulsen, Wolfgang Ludwig, and Søren Schmidt</i>	
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
	<i>Gene E. Ice</i>	
18.1	Introduction	309
18.2	The Need for <i>Polychromatic</i> 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
	<i>Wolfgang Kaysser, Jörg EBlinger, Volker Abetz, Norbert Huber, Karl U. Kainer, Thomas Klassen, Florian Pyczak, Andreas Schreyer, and Peter Staron</i>	
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* μ Laue: 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 μ Laue 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: <i>In situ</i> 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
	<i>Manuela Klaus and Christoph Genzel</i>	
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

