

Contents

Part I Fundamentals of Processing for Morphology-Controlled Materials

1 Morphology Control in Size Reduction Processes

<i>F. Saito, M. Baron, J. Dodds</i>	3
1.1 Introduction	3
1.2 Shape of Particles	4
1.2.1 Shapes of Fragments when Grinding a Single Brittle Particle	4
1.2.2 Shape Control by Grinding	7
1.2.3 Shape Control by Sieving	7
1.2.4 Shape Change by Grinding and Its Separation	8
1.3 Morphology and Structure of Materials During Grinding	10
1.3.1 Mechanical Activation by Grinding	10
1.3.2 Polymorphic Transformation of Materials by Dry Grinding	11
1.3.3 Synthesis of Inorganic Materials from Their Constituents by Dry Grinding	13
1.3.4 Synthesis of Inorganic Materials in Wet Grinding	17
1.3.5 Synthesis of Inorganic Compounds from Inorganic and Organic Materials by Dry Grinding	17
1.3.6 Mechanochemical-Soft Solution Processes	17
1.3.7 Morphology Control in Pharmaceutical Products	20
References	22

2 Liquid-Phase Processing

<i>A. Muramatsu</i>	25
2.1 Introduction	25
2.2 Shape Control	25
2.2.1 Basic Principles	25
2.2.2 Gel-Sol Method	30
2.3 Control of Crystallinity	40
2.4 Size and Size Distribution Control	48
References	60

Part II Novel Method of Morphology Control

3 Stratified Materials Synthesized in Liquid-Phase

<i>T. Arai, Y. Sato, K. Shinoda, B. Jeyadevan, K. Tohji</i>	65
3.1 The Stratified Photocatalyst for Hydrogen Evolution	65
3.2 Synthesis and Characterization of the Stratified-ZnS Photocatalyst	66
3.3 Formation Scheme of the Stratified-ZnS Photocatalyst	67
3.4 Hydrogen Evolution by Means of the Stratified-ZnS Photocatalyst	69
3.5 Photocatalytic Reaction Mechanisms of Stratified-ZnS Photocatalyst	70
3.6 Synthesis and Characterization of the Stratified CdS Photocatalyst	71
3.7 Efficiency of the Stratified CdS Photocatalyst for Hydrogen Production	72
3.8 The Role of Stratified Structure	74
3.9 Hydrogen Evolution Under Sunlight by Means of the Stratified-CdS Photocatalyst	75
3.10 Nano-Structure and Photocatalytic Property of Stratified ZnS Thin Films	76
3.11 Synthesis of ZnS Thin Film by Chemical Bath Deposition	76
3.12 Characterization and Measurement of Photocatalytic Activity of ZnS Thin Film	77
3.13 Morphology of CBD ZnS Film	77
3.14 Photocatalytic Property of the CBD ZnS Thin Film	78
3.15 Nano-structure in CBD ZnS Thin Film	78
3.16 Enhancement of the Photocatalytic Activity on Carbon Nanotubes	80
3.16.1 Synthesis of Carbon Nanotubes	80
3.16.2 Stratified ZnS Photocatalyst on Carbon Nanotubes ...	81
3.16.3 Local Analyses of Stratified ZnS on Carbon Nanotubes	82
References	84

4 Well-Dispersed Bimetallic Nanoparticles

<i>T. Yonezawa</i>	85
4.1 Introduction	85
4.2 Principal Synthesis Procedures of Metal Nanoparticles in Liquid Phase	87
4.2.1 Alcohol Reduction of Metal Salts	88
4.2.2 Photolysis	88
4.2.3 Reduction of Metal Ions by Hydrogen	89
4.2.4 Sonochemical Reduction of Metal Ions	89

4.2.5	Reduction of Metal Ions by Citrates and Hydrazines ..	90
4.2.6	Reduction of Metal Ions by Hydroborates	91
4.2.7	Decomposition of Organometallic Compounds	92
4.2.8	Electrochemical Reduction of Metal Ions	92
4.3	Preparation of Bimetallic Nanoparticles in Liquid Phase	93
4.3.1	Simultaneous Reduction of Metal Ions	94
4.3.2	Successive Reduction of Metal Ions	98
4.3.3	Reduction from Bimetallic Complexes	99
4.3.4	Electrochemical Preparation of Bimetallic Nanoparticles	99
4.3.5	Addition of Metal Ions to the Particles	100
4.4	Characterization of Bimetallic Nanoparticles	100
4.4.1	UV-Vis Spectroscopy	100
4.4.2	Electron Microscopic Observation	100
4.4.3	X-ray Methods	102
4.4.4	Analysis by Chemical Probes	105
4.5	Conclusion	108
	References	108
5 Porous Materials Controlled in Shape		
	<i>T. Okubo, M. Matsukata</i>	113
5.1	Classification of Porous Materials	113
5.2	Structure of Zeolite and Related Materials	115
5.3	Synthesis of Zeolites	116
5.3.1	Hydrothermal Synthesis Method	116
5.3.2	Dry Gel Conversion Methods	118
5.4	Morphology Control of Zeolites	120
5.4.1	Zeolite Synthesis by the Vapor Phase Transport Method	120
5.4.2	Zeolite Synthesis by the Steam-Assisted Crystallization (SAC) Method ..	121
5.4.3	Synthesis of Metallosilicate by the Dry Gel Conversion Method	124
5.4.4	Fabrication of Zeolite Membrane by the Dry Gel Conversion Method	124
5.4.5	Role of Water in the Course of Crystallization in the Dry Gel Conversion Method	125
5.5	Conclusion	126
	References	126
6 Surface Control		
	<i>A. Muramatsu, Y. Waseda</i>	129
6.1	Preparation of Nanoparticles	129
6.2	Selective Deposition of Nanoparticles	137

X	Contents	
6.2.1	Selective Deposition of Gold Nanoparticles on Well-Defined Materials	139
6.2.2	Selective Deposition of Pt Nanoparticles on Well-Defined Materials	143
6.3	Reductive Deposition of Nanoparticles on Monodispersed Particles	146
	References	147

Part III Characterization

7 Fundamentals of Characterization

	<i>D. Shindo, Y. Murakami</i>	153
7.1	Introduction	153
7.2	X-ray Diffraction	154
7.2.1	Principles of X-ray Diffraction	154
7.2.2	Application of Powder X-ray Diffraction	158
7.3	Electron Microscopy	162
7.3.1	Principles of Electron Microscopy	162
7.3.2	Application of Scanning Electron Microscopy to Morphology Analysis	166
7.3.3	Application of Transmission Electron Microscopy	167
7.4	Analytical Electron Microscopy	172
7.4.1	Principles of Electron Energy-Loss Spectroscopy and Energy Dispersive X-ray Spectroscopy	173
7.4.2	Application of Electron Energy-loss Spectroscopy	174
7.4.3	Application of Energy Dispersive X-ray Spectroscopy ..	179
	References	180

8 Photocatalytic Properties:

Effect of Size, Shape and Surface Structures of Fine Particles

	<i>Y. Wada, H. Yin, S. Yanagida</i>	183
8.1	General Aspects	183
8.1.1	Methods for Preparing Semiconductor Nanoparticles ..	183
8.1.2	Crystal Structures	184
8.1.3	Change of Electronic Structures Induced in Nanoparticles	185
8.1.4	Surface Structures of Nanosized Semiconductors	186
8.1.5	Photocatalysis of Semiconductor Nanoparticles	186
8.2	Metal Sulfides	187
8.2.1	Size Effect on Photocatalysis	187
8.2.2	Importance of Surface Structure in Photocatalysis	188
8.2.3	Importance of Adsorptive Activation of Substrates in Photocatalysis	189

8.3	Metal Oxides	190
8.3.1	Preparation of Nanosized TiO ₂	190
8.3.2	Crystallinity and Photocatalysis of TiO ₂	194
8.3.3	Surface Morphology of TiO ₂ Affecting on its Photocatalysis	195
8.4	Future Aspects	197
	References	198
9 Surface Characteristics		
	<i>A. Fukuoka, M. Ichikawa</i>	201
9.1	Overview of Nanoparticles and Nanowires	201
9.2	Template Synthesis of Metal Nanowires in Porous Materials	202
9.2.1	Metal Nanowires in Mesoporous Silicas MCM-41 and SBA-15	202
9.2.2	Template Synthesis and Catalysis of Metal Nanowires in Mesoporous Silicas FSM-16 and HMM-1	204
9.2.3	Metal Nanowires in Anodic Alumina Membrane	213
9.2.4	Metal Nanowires in Carbon Nanotubes	215
9.3	Various Syntheses of Metal Nanowires	216
9.4	Conclusion	218
	References	219
10 Structural Characterization of Surface and Morphology of Materials Using X-ray Scattering		
	<i>Y. Waseda, M. Saito, S. Suzuki</i>	223
10.1	Introduction	223
10.2	Fundamentals of the GIXS Method	224
10.3	Structure of Thin Oxide Films Grown on an Iron-Base Alloy Surface	229
10.4	Characterization for Morphology of Thin Deposited Films on a Silicon Wafer	236
10.5	Structure of Surface Layers in Crystalline Metals	242
10.6	Structure of Surface Layers of Nanometer-Sized Crystalline Particles	246
10.7	Conclusion	253
	References	254
	Index	257