

Contents

Preface for the second edition — VII

Preface — IX

Acknowledgment — XI

1 Introduction — 1

2 Molecular structure and electronic structure — 5

- 2.1 X-ray absorption spectroscopy — 5
- 2.1.1 Hard X-rays: XAS and XANES — 5
- 2.1.2 Soft X-ray spectroscopy: NEXAFS — 23
- 2.1.3 Tender X-ray absorption spectroscopy — 24
- 2.1.4 Chlorine — 28
- 2.2 X-ray emission spectroscopy — 31
- 2.2.1 Hard X-ray emission — 31
- 2.2.2 Energy-dispersive X-ray spectroscopy — 36
- 2.2.3 Soft X-ray emission spectroscopy — 37
- 2.3 X-ray photoelectron spectroscopy — 44
- 2.3.1 Core level spectroscopy — 44
- 2.3.2 Valence band spectroscopy — 62

3 Crystal structure and microstructure — 73

- 3.1 X-ray diffraction — 73
- 3.2 X-ray wide-angle scattering — 78
- 3.3 Small-angle X-ray scattering (SAXS) — 82
- 3.3.1 SAXS on GC supercapacitor electrodes — 83
- 3.3.2 Excursion to ultrasmall-angle neutron scattering (USANS) — 91
- 3.3.3 USAXS on aerogel catalysts — 95
- 3.4 Extended X-ray absorption fine structure (EXAFS) spectroscopy — 100
- 3.5 X-ray reflectometry — 104
- 3.5.1 XRR on photoelectrode materials — 104
- 3.5.2 *In situ* and *operando* X-ray and neutron reflectometry on electrochemical systems — 109
- 3.5.3 Neutron reflectometry on electrochemical systems — 110

4 Real space imaging and tomography — 114

- 4.1 X-ray imaging and X-ray microscopy — 115
- 4.1.1 Polymer electrolyte membrane fuel cells — 115

4.1.2	Batteries — 116
4.2	Neutron imaging and radiography — 118
4.2.1	Solid oxide fuel cells — 118
4.3	Complementarity of X-ray and neutron methods — 120
4.4	Tomography — 122
4.4.1	Neutron tomography on SOFC — 123
4.4.2	X-ray tomography on SOFC — 124
4.4.3	Tomography on PEM-FC — 125
4.4.4	Tomography on batteries — 129
4.5	Scanning transmission X-ray microspectroscopy (STXM) — 132
4.5.1	STXM on PEM fuel cells — 134
4.5.2	STXM on lithium ion batteries — 140
4.5.3	STXM on biological samples — 142
4.5.4	STXM on soot from combustion processes — 144
4.5.5	STXM on Liesegang rings — 147
4.6	X-ray ptychography — 152
4.6.1	Concept — 152
4.6.2	Application — 153
5	Resonant methods and chemical contrast variation — 155
5.1	X-ray tube (Kathodenstrahlen) — 155
5.2	Synchrotron storage rings — 158
5.3	The X-ray Free Electron Laser (XFEL) — 159
5.4	Anomalous X-ray diffraction — 161
5.4.1	Distinguishing W and Cu in CuWO ₄ photoanodes — 162
5.4.2	Separation of manganite spinel diffractogram from other battery components — 169
5.4.3	Diffraction on substituted protonated ceramic proton conductors — 172
5.5	Anomalous small-angle X-ray scattering — 176
5.5.1	<i>In situ</i> ASAXS on lithium batteries — 176
5.5.2	<i>In situ</i> ASAXS on Pt-based PEM-FC assemblies — 181
5.5.3	ASAXS on SOFC assemblies — 184
5.6	Resonant photoemission spectroscopy — 194
5.6.1	SOFC cathodes — 194
5.6.2	Mn 2p resonant photoelectron spectroscopy for SOFC chromium poisoning — 196
5.6.3	VB PES on photoelectrodes — 199
5.6.4	Lithium ion battery cathodes — 215
5.6.5	Resonant inelastic X-ray scattering — 224
5.6.6	Nuclear resonance vibrational spectroscopy — 226

6	Surface-sensitive and volume-sensitive methods — 229
6.1	X-ray Raman spectroscopy — 236
6.2	Hard X-ray XPS and hard X-ray PES (HAX-PES) — 240
6.3	X-ray standing waves for electrochemical double layer studies — 246
6.3.1	Resonant photoemission spectroscopy on thin thermochromic window VO ₂ films — 249
7	Organic and bioorganic samples — 253
7.1	X-ray studies on polymer electrolyte membranes — 255
7.2	Organic solar cells — 257
7.2.1	Organic dyes for photosensitization — 259
7.3	Proteins, enzymes, biocatalysts, living cells, and biofilms — 262
7.3.1	Protein crystallography — 262
7.3.2	Reflectometry on proteins and cells — 264
7.3.3	Protein spectroscopy — 269
7.3.4	Bioelectrodes, biofilms, and bioelectricity — 271
7.3.5	Photo-electrochemical studies on cyanobacteria during γ -irradiation — 275
8	Complex case studies/electrochemical <i>in situ</i> studies — 286
8.1	Lithium ion batteries — 289
8.1.1	Battery cell assembly and <i>ex situ</i> studies on materials and components — 289
8.1.2	Manufacturing and assembly of <i>in situ</i> battery cells at the synchrotron — 297
8.1.3	<i>Operando</i> XANES, anomalous XRD, EXAFS, and ASAXS on a lithium ion battery — 303
8.1.4	<i>Operando</i> X-ray Raman study on a lithium ion battery — 308
8.1.5	X-ray spectro-electrochemical cells for <i>operando</i> and <i>in situ</i> battery studies — 316
8.1.6	Isosbestic points — 318
8.2	Ceramic fuel cells — 321
8.2.1	Ceramic proton conducting electrolyte membranes — 321
8.2.2	SOFC anode poisoning and sulfur molecular structure — 329
8.3	Photo-electrochemical cells (for solar hydrogen) — 338
8.3.1	<i>Operando</i> NEXAFS spectroscopy during water splitting — 339
8.3.2	<i>Operando</i> photoelectron spectroscopy during water splitting — 348
8.4	Bioelectrochemical systems — 355
8.4.1	Fe 3p resonant VB XPS on phycocyanin adsorbed on iron oxide — 355
8.4.2	<i>Operando</i> NAP-XPS spectroscopy on an illuminated algal biofilm — 362
8.5	Electrochemical double layer capacitors — 365
8.5.1	X-ray diffraction and wide angle scattering on glassy carbon — 365

8.5.2	Small-angle scattering on supercapacitor electrodes with X-rays and with neutrons — 365
8.5.3	NEXAFS spectroscopy on thermally oxidized glassy carbon plates — 367
8.6	“OpMetBat”: A battery metrology project — 369
9	Correlation of electronic structure and conductivity — 374
9.1	Hole conductivity in SOFC cathodes — 377
9.2	SOFC chromium poisoning — 391
9.3	Proton conductivity in IT-SOFC electrolytes — 393
9.4	Lithium ion batteries — 394
9.5	Water splitting photoelectrodes — 396
10	Radiation damages — 402
11	Background subtraction — 409
11.1	Optical absorption spectra of phycocyanin on a photoelectrode — 409
11.2	NEXAFS data on CuWO ₄ from BESSY-II — 410
11.3	Treatment of optical Raman spectra — 415
11.4	Background subtraction for an empty cell — 417
A	Appendix — 421
A.1	X-ray physics Nobel prizes — 421
A.2	Synchrotron centers worldwide — 424
A.3	Electromagnetic spectrum — 426
A.4	K α , β X-ray energies — 427
A.5	Periodic table of elements — 429
A.6	Electronic configuration of the chemical elements — 430
A.7	X-ray data analysis software — 431
Bibliography — 433	
Index — 479	