



X-Rays and Materials

**Edited by
Philippe Goudeau
René Guinebretière**

ISTE

 **WILEY**

Table of Contents

Preface	xi
Chapter 1. Synchrotron Radiation: Instrumentation in Condensed Matter	1
Jean-Paul ITIE, François BAUDELET, Valérie BRIOIS, Eric ELKAÏM, Amor NADJI and Dominique THIAUDIÈRE	
1.1. Introduction	1
1.2. Light sources in the storage ring	2
1.2.1. Bending magnets	2
1.2.2. Insertion devices	4
1.2.2.1. Wigglers	4
1.2.2.2. Undulators	5
1.3. Emittance and brilliance of a source.	6
1.4. X-ray diffraction with synchrotron radiation.	8
1.4.1. Angle-dispersive diffraction	8
1.4.2. Energy dispersive diffraction	9
1.5. X-ray absorption spectroscopy using synchrotron radiation.	13
1.5.1. X-ray absorption spectroscopy	14
1.5.2. Energy-scanned X-ray absorption spectroscopy	17
1.5.3. Energy dispersive X-ray absorption spectroscopy	18
1.6. SAMBA: the X-ray absorption spectroscopy beam line of SOLEIL for 4–40 keV.	20
1.7. The DIFFABS beam line.	27
1.7.1. Description of the beam line.	27

1.7.2. Examples of use of the DIFFABS beam line	31
1.8. CRISTAL beam line	34
1.8.1. Beam line optics	35
1.8.2. Diffractometers	35
1.8.3. Sample environments	36
1.9. The SOLEIL ODE line for dispersive EXAFS	38
1.9.1. Optics of the ODE line	38
1.9.2. Magnetic circular dichroism	39
1.9.3. X-ray absorption spectroscopy under extreme pressure and/or temperature conditions	41
1.10. Conclusion	43
1.11. Bibliography	44

Chapter 2. Nanoparticle Characterization using Central X-ray Diffraction 49

Olivier SPALLA

2.1. Introduction	49
2.2. Definition of scattered intensity	50
2.3. Invariance principle	52
2.3.1. General case	52
2.3.2. Isotropic systems	53
2.3.3. Multi-level systems	54
2.4. Behavior for large q : the Porod regime	55
2.5. Particle-based systems	59
2.5.1. Definition of form factor	59
2.5.2. Introduction to the structure factor	61
2.5.3. Intensity behavior at small q : the Guinier regime	64
2.5.4. Volume measurements	65
2.5.5. Some well-known form factors	66
2.5.6. Polyhedral particles	70
2.5.6.1. Form factor of a polyhedron	70
2.5.6.2. Comparison between different polyhedra with cylindrical and spherical forms	73
2.6. An absolute scale for measuring particle numbers	75
2.7. Conclusion	78
2.8. Bibliography	79

Chapter 3. X-ray Diffraction for Structural Studies of Carbon Nanotubes and their Insertion Compounds	81
Julien CAMBEDOUZOU and Pascale LAUNOIS	
3.1. Introduction	81
3.1.1. Introduction to carbon nanotubes	82
3.1.2. Uses of X-ray scattering for studies of carbon nanotubes	84
3.2. Single-walled carbon nanotubes.	85
3.2.1. Calculation of a powder diffraction diagram.	86
3.2.1.1. Individual nanotubes	86
3.2.1.2. Bundle structure.	89
3.2.1.3. Inclusion of a distribution of nanotube diameters	91
3.2.1.4. Effects of nanotube length	93
3.2.2. Analysis of experimental scattering diagrams	94
3.3. Multi-walled carbon nanotubes	96
3.3.1. Calculation of powder diffraction diagrams for a powder of individual multi-walled nanotubes	97
3.3.2. Analysis of an experimental diffraction diagram	101
3.4. Hybrid nanotubes.	102
3.4.1. Peapods	102
3.4.2. Ion insertion into nanotubes.	108
3.5. Textured powder samples	110
3.5.1. Quantification of nanotube orientation	112
3.5.2. Separation of diffraction components in hybrid nanotubes.	116
3.6. Conclusion.	121
3.7. Bibliography	122
Chapter 4. Dielectric Relaxation and Morphotropic Phases in Nanomaterials	129
Jean-Michel KIAT	
4.1. Introduction	129
4.2. Dielectric relaxation and morphotropic region: definition and mechanism.	130
4.2.1. Definition of a relaxor compound	130
4.2.2. Microscopic mechanism associated with the occurrence of dielectric relaxation.	134

4.2.2.1. Microscopic mechanism: high temperatures (regimes where $T > T_B$ and then $T^* < T < T_B$).	135
4.2.2.2. Microscopic mechanism: intermediate temperatures (regimes where $T_G < T < T^*$).	141
4.2.2.3. Microscopic mechanism (regime where $T < T_G$)	143
4.2.2.4. Microscopic mechanism: importance of local chemistry.	145
4.2.3. Generalization of mechanism: ferroelectric relaxors	148
4.2.4. Definition of a compound and a morphotropic region.	152
4.3. Relaxation, morphotropic region and size reduction	163
4.3.1. Size reduction in relaxor ceramics.	163
4.3.2. Size reduction in MPB ceramics	168
4.3.3. Thin layers and super-lattices	170
4.4. Conclusion.	174
4.5. Acknowledgements.	175
4.6. Bibliography	175

**Chapter 5. Evolution of Solid-state Microstructures
in Polycrystalline Materials: Application
of High-energy X-ray Diffraction to Kinetic
and Phase Evolution Studies** 181

Elisabeth AEBY-GAUTIER, Guillaume GEANDIER,
Moukrane DEHMAS, Fabien BRUNESSEUX, Adeline BENETEAU,
Patrick WEISBECKER, Benoît APPOLAIRE and Sabine DENIS

5.1. Introduction	181
5.2. Experimental methods	183
5.2.1. Apparatus for thermo-mechanical studies	185
5.2.2. Choice of detector	188
5.2.3. Device calibration	191
5.2.4. Data analysis	192
5.3. Results	195
5.3.1. Phase transformation in titanium alloy Ti-17.	195
5.3.2. Martensitic transformation of a maraging steel	208
5.4. Conclusion.	213

5.5. Acknowledgements.	214
5.6. Bibliography	214
List of Authors	221
Index	223