

Contents

List of Figures x

List of Tables xvii

Preface xviii

1 Basics of Material Characterization Techniques 1

- 1.1 Introduction 1
- 1.2 Electromagnetic Spectrum 2
- 1.3 Fundamentals of Crystallography 4
- 1.4 Molecular Vibrations 10
- 1.5 Magnetism in Solids 10
- 1.6 Optical Properties of Solids 11
- References 13

2 X-ray Diffraction 15

- 2.1 Introduction 15
- 2.2 Bragg's Law 17
- 2.3 Von Laue Treatment: Laue's Equation 18
- 2.4 Experimental Techniques 20
 - 2.4.1 Laue Method 20
 - 2.4.2 Rotating Crystal Method 20
 - 2.4.3 Powder Method 21
- 2.5 Geometry and Instrumentation 21
- 2.6 Standard XRD Pattern 26
- 2.7 Applications 27
 - 2.7.1 Orientation of Single Crystals 27
 - 2.7.2 Structure of Polycrystalline Aggregates 29
 - 2.7.3 XRD in the Pharmaceutical Field and Forensic Science 33
 - 2.7.4 XRD in the Geological Field 34
- 2.8 Examples and Illustrations 34
- 2.8.1 XRD Data and Interpretation in the $\text{PrFe}_{1-x}\text{Mn}_x\text{O}_3$ System 34

2.8.2	XRD Data and Interpretation in the $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ System	37
2.8.3	XRD Analysis of $\text{EuFe}_{1-x}\text{Mn}_x\text{O}_3$ ($x = 0.0, 0.3, 0.5$)	38
	References	40
3	Raman Spectroscopy	43
3.1	Introduction	43
3.2	Infrared and Raman Spectroscopy	44
3.3	Raman Spectra: Origin	45
3.4	Classical Theory of Raman Scattering	47
3.5	Quantum Theory of Raman Spectroscopy	48
3.6	Raman Spectrometer	50
3.6.1	Excitation Source	50
3.6.2	Sample Illumination	51
3.6.3	Wavelength Selector	51
3.6.4	Detection and Control System	52
3.7	Resonance Raman Spectroscopy	53
3.8	Special Techniques	55
3.8.1	High-pressure Raman Spectroscopy	55
3.8.2	Raman Microscopy	57
3.8.3	Surface-enhanced Raman Spectroscopy	59
3.8.4	Raman Spectroelectrochemistry	60
3.9	Applications and Illustrations	62
3.9.1	Raman Spectra of the $\text{PrFe}_{1-x}\text{Mn}_x\text{O}_3$ System at Different Concentrations of Mn Doped in Place of Fe	62
3.9.2	Raman Spectra and Measurements of the $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ System ($x = 0.0, 0.3, 0.5$, and 0.7)	64
3.9.3	Temperature-dependent Raman Study of $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ ($x = 0.0$ and 0.3)	66
	References	71
4	X-ray Spectroscopic Techniques	77
4.1	X-ray Absorption Spectroscopy	77
4.1.1	Introduction	77
4.1.2	Basic Principle of XAS	78
4.1.3	Experimental Aspects	83
4.1.3.1	Synchrotron Radiation	83
4.1.3.2	Experimental Setup	84
4.1.3.3	Transmission Mode	84
4.1.3.4	Fluorescence Mode	85
4.1.3.5	Electron Yield Mode	85
4.1.4	Examples and Analysis	86
4.1.4.1	X-ray Absorption Spectra of $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ ($x = 0.0, 0.3, 0.5, 0.7$) Samples	86

4.1.4.2	Electronic Structure of $\text{PrFe}_{1-x}\text{Mn}_x\text{O}_3$ by X-Ray Absorption Spectroscopy	88
4.2	X-ray Photoelectron Spectroscopy (XPS)	92
4.2.1	Introduction	92
4.2.2	Basic Principle	93
4.2.3	Energy Referencing	94
4.2.4	Instrumentation	96
4.2.5	XPS Spectra and Their Features	97
4.3	Auger Electron Spectroscopy	99
4.3.1	Introduction	99
4.3.2	Interactions of Electrons with Matter	100
4.3.3	Competition Between X-ray and Auger Electron Emissions	100
4.3.4	Auger Process	101
4.3.5	Kinetic Energy of the Auger Electron	103
4.3.6	Auger Spectra	104
4.3.7	Instrumentation	105
	References	106
5	Magnetic Measurements	109
5.1	Introduction	109
5.2	Magnetization Measuring Instruments	111
5.2.1	Extraction Technique	112
5.2.2	Vibrating Sample Magnetometer	113
5.2.3	SQUID Magnetometer	114
5.3	Advantages and Disadvantage of a Vibrating Sample Magnetometer	115
5.4	Susceptibility Measurement	116
5.5	Examples and Illustrations	117
5.5.1	Magnetic Behavior Shown by Thin Films of the $\text{PrFe}_{1-x}\text{Mn}_x\text{O}_3$ System Deposited on Substrate Si (100)	117
5.5.2	Magnetic Behavior of the $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ System Where x is the Concentration of Ca as Dopant and Equals 0.0, 0.3, 0.5, or 0.7	120
5.5.3	Magnetic Behavior of $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ Thin Films Deposited on Si(100) with $x = 0.0, 0.3, 0.5$, and 0.7 Being the Concentrations of Ca	124
	References	125
6	Dielectric Measurements	129
6.1	Introduction	129
6.2	Polarization and Dielectric Constant	130
6.2.1	Electronic or Optical Polarization	131
6.2.2	Orientational Polarization	134
6.2.3	Atomic Polarizability	135
6.2.4	Interfacial Polarization	136
6.3	Mechanism for the Colossal Dielectric Response	137

6.4	Frequency Dependence of Polarizability	138
6.5	Classification of Dielectric Materials	138
6.5.1	Nonferroelectric Materials	138
6.5.2	Nonpolar Materials	139
6.5.3	Polar Materials	139
6.5.4	Dipolar Materials	139
6.6	Dielectric Dispersion: A Brief Discussion	140
6.7	Dielectric Loss and Relaxation	141
6.8	Complex Permittivity	141
6.9	Polarization Buildup	142
6.10	Jonscher's Universal Law	145
6.11	Examples and Illustrations	148
6.11.1	Dielectric Behavior of $\text{PrFe}_{1-x}\text{Mn}_x\text{O}_3$ with $x = 0.0, 0.1, 0.3$, and 0.5 Being the Concentration of Mn Doped in a Pristine Compound	148
6.11.1.1	Frequency and Temperature Dependence of Dielectric Properties	148
6.11.2	Dielectric Properties of the $\text{EuFe}_{1-x}\text{Mn}_x\text{O}_3$ System in Which Different Concentrations of Mn Are Doped in EuFeO_3	151
6.11.2.1	Dependence of Dielectric Behavior on Frequency	151
6.11.2.2	Dependence of Dielectric Behavior on Temperature	154
	References	157

7	Electron Microscopy	159
7.1	Introduction	159
7.2	Generation of an Electron Beam	159
7.3	Interaction of an Electron Beam with a Sample	160
7.4	Inelastic Scattering and Absorption	161
7.5	The Family of Electron Microscopes	164
7.5.1	The X-ray Microscope	165
7.5.2	The Transmission Electron Microscope	166
7.5.3	The Scanning Electron Microscope	172
7.5.4	The Scanning Transmission Electron Microscope	174
7.6	Atomic Force Microscopy	175
7.7	Examples and Illustrations	177
7.7.1	AFM Studies of $\text{PrFe}_{1-x}\text{Mn}_x\text{O}_3$ ($x = 0.0, 0.1, 0.3, 0.5$) Thin Films Grown on Si (100)	177
7.7.2	Atomic Force Microscopy in the Case of $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ for (a) $x = 0.0$, (b) $x = 0.3$, (c) $x = 0.5$, and (d) $x = 0.7$	177
7.7.3	Morphological Studies and Elemental Analysis of $\text{EuFe}_{1-x}\text{Mn}_x\text{O}_3$ ($x = 0.0, 0.3$, and 0.5)	178
	References	181

8	Infrared Spectroscopy	183
8.1	Introduction	183
8.2	Instrumentation for FTIR	184
8.3	Fourier Transform	189
8.4	Electromagnetic Radiation	190
8.5	Infrared Absorption	190
8.6	Normal Modes of Vibration	191
8.7	Complicating Factors	192
8.8	Applications of IR Spectroscopy	192
8.8.1	Food Science	192
8.8.2	Chemistry in Clinical Practice	193
8.8.3	Plants	194
8.8.4	Disease Diagnosis	194
8.8.5	Environmental Applications	195
	References	196

Index 199