Contents

COPYRIGHTED MATERIAL

Preface **xi**

1 A Brief Historical Overview **1**
1.1 Scanning Electron Microscopy 1
1.1.1 The Beginnings 1
1.1.2 The Need for Added Capabilities 3
1.2 The Development of Imaging in a Gas Environment 6
1.2.1 Overcoming the Limits of Conventional SEM 6
1.2.2 Leaps and Bounds 9

2 Principles of SEM **17**
2.1 Introduction 17
2.1.1 Why Use An Electron Beam? 17
2.1.2 The SEM Column 19
2.1.3 Why Do We Need a Vacuum System? 19
2.2 Electron Sources 20
2.2.1 Thermionic Emission Sources 21
2.2.2 Field Emission Sources 22
2.3 Electron Optics 23
2.3.1 Lenses 23
2.3.2 Lens Aberrations 26
2.4 Signals and Detection 31
2.4.1 Primary Electrons and the Interaction Volume 33
2.4.2 Backscattered Electrons 39
2.4.3 Secondary Electrons 41
2.4.4 X-ray Radiation 45
2.4.5 Cathodoluminescence 47
2.5 Practical Aspects of Electron Beam Irradiation 48
2.5.1 Radiation Damage 48

2.5.2 Minimising Specimen Charging – Low-Voltage SEM 50
2.5.3 Increasing Surface and Bulk Conductivities 51
2.6 SEM in Operation 53
2.6.1 Building Up an Image 53
2.6.2 Magnification 53
2.6.3 Signal-to-Noise Ratio 54
2.6.4 Contrast 55
2.6.5 Adjusting the Contrast 56
2.6.6 Resolution 57
2.6.7 Depth of Field 59
2.6.8 Image Capture 60

3 General Principles of VP-ESEM: Utilising a Gas 63
3.1 Introduction 63
3.2 VP-ESEM Instrumentation 64
3.2.1 Typical Features 64
3.2.2 Primary Electron Scattering in VP-ESEM – the General Case 65
3.2.3 Units of Pressure 68
3.3 Signal Generation in a Gas 68
3.3.1 Introduction 68
3.3.2 Direct Collection of Electrons and Ions 69
3.3.3 Collection of Photons – the Gas Luminescence Signal 74
3.3.4 Detecting Indirect Electron and Ion Currents 78
3.4 Imaging with Water Vapour 81
3.4.1 Introduction 81
3.4.2 Thermodynamic Equilibria 81
3.4.3 Nonequilibrium Conditions 86
3.4.4 Practicalities of Stabilising Hydrated Specimens 87

4 Imaging and Analysis in VP-ESEM: The Influence of a Gas 93
4.1 Introduction 93
4.2 Background to Theoretical Calculations 94
4.2.1 Calculating the Mean Free Paths of Primary Electrons 94
4.2.2 Calculating Pressure-Dependent Variables 94
4.2.3 Estimating the 'Useful' Primary Electron Current 95
4.3 Which Gas? 98
4.3.1 Introduction 98

4.3.2 Usefulness of the Gas – Experimental Conditions 98
4.3.3 Ionisation and Excitation for Different Gases 99
4.3.4 Scattering of the Primary Electron Beam in Different Gases 101
4.4 Exploring the Gas Path Length 106
4.4.1 Introduction 106
4.4.2 Influence of GPL on the Skirt Radius 107
4.4.3 Gas Path Length and Useful Primary Electron Beam Current 108
4.4.4 Constraints on Reducing the Gas Path Length 109
4.4.5 Separating Gas Path Length from Working Distance 111
4.5 How Much Gas? 112
4.5.1 Introduction 112
4.5.2 Scattering of Primary Electrons as a Function of Pressure 114
4.6 X-ray Microanalysis in VP-ESEM 121
4.6.1 Introduction 121
4.6.2 Effects of Chamber Gas on X-ray Signals 121
4.6.3 Considerations for Minimising the Effects of the Gas 123
4.6.4 Post-Acquisition Methods to Correct for Scattering 126

5 Imaging Uncoated Specimens in the VP-ESEM 131
5.1 Introduction 131
5.2 Electronic Structure 132
5.2.1 The Energy Level Diagram 132
5.2.2 Conductors, Semiconductors and Insulators 133
5.3 Factors Affecting Secondary Electron Emission 135
5.3.1 Transport of Excited Electrons 135
5.3.2 Escape of Excited Electrons 137
5.4 The Influence of the Specimen on the System 137
5.4.1 The Effect of Charging – the General Case 137
5.4.2 Measuring Surface Potential 141
5.4.3 Conductive, Electrically Grounded Bulk Materials 141
5.4.4 Conductive, Electrically Isolated Materials 142
5.4.5 Nonconductive, Uncoated Materials 143
5.5 Time- and Temperature-Dependent Effects 145
5.5.1 Introduction 145
5.5.2 Conductivity and Some Time-Dependent Effects 145

5.5.3 Charge Traps and Thermal Effects 150
5.6 Imaging Soft Materials 151
5.6.1 Introduction 151
5.6.2 Choosing an Appropriate Primary Beam Energy 152
5.6.3 Radiation Damage 156
5.7 Effects of Ions on Imaging 158
5.7.1 Introduction 158
5.7.2 Consideration of the Concentration of Positive Ions 158
5.7.3 Ion Mobility Effects 159
5.7.4 An Additional Surface Potential 161
5.7.5 Electron–Ion Recombination and Signal Scavenging 162
5.7.6 Combating Excess Ions 163
5.8 Imaging with a Gas: Summary 164

6 A Lab in a Chamber – *in situ* Methods in VP-ESEM and Other Applications 169
6.1 Introduction 169
6.2 Nanocharacterisation of Insulating Materials 170
6.2.1 High-Resolution Imaging 170
6.2.2 Anti-Contamination in the VP-ESEM 171
6.2.3 Nanometrology 172
6.2.4 Utilising Novel Contrast Mechanisms 173
6.2.5 Transmitted Electron Signals – STEM and Wet STEM 178
6.3 *In situ* Experiments 181
6.3.1 Deformation and Failure 181
6.3.2 Low-Temperature Experiments 182
6.3.3 High-Temperature Experiments 186
6.3.4 Condensation and Evaporation of Water 189
6.3.5 Processes Using Electron Beam Gas Chemistry in the VP-ESEM 194
6.3.6 High-Pressure Experiments 196
6.4 Other Applications 198
6.4.1 Introduction 198
6.4.2 Biological Specimens 198
6.4.3 Liquids and Soft Materials 202
6.4.4 Hard/Soft Composites and Hard Materials 204

Index 215