ATOMIC FORCE MICROSCOPY

Understanding Basic Modes and Advanced Applications

GREG HAUGSTAD



CONTENTS

Pref	Preface			
Ackı	owled	lgments	xxi	
1.	Overview of AFM			
	1.1. 1.2. 1.3. 1.4. 1.5. 1.6. 1.7. 1.8.	The Essence of the Technique, 1 Property Sensitive Imaging: Vertical Touching and Sliding Friction, Modifying a Surface with a Tip, 13 Dynamic (or "AC" or "Tapping") Modes: Delicate Imaging with Property Sensitivity, 16 Force Curves Plus Mapping in Liquid, 21 Rate, Temperature, and Humidity-Dependent Characterization, 24 Long-Range Force Imaging Modes, 28 Pedagogy of Chapters, 30 References, 31	6	
2.	Distance-Dependent Interactions			
	2.3.	General Analogies and Types of Forces, 33 Van der Waals and Electrostatic Forces in a Tip–Sample System, 38 2.2.1. Dipole–Dipole Forces, 38 2.2.2. Electrostatic Forces, 41 Contact Forces and Mechanical Compliance, 44 Dynamic Probing of Distance-Dependent Forces, 51		
		2.4.1. Importance of Force Gradient, 51		

viii CONTENTS

	2.5.	 2.4.2. Damped, Driven Oscillator: Concepts and Mathematics, 56 2.4.3. Effect of Tip-Sample Interaction on Oscillator, 60 2.4.4. Energy Dissipation in Tip-Sample Interaction, 64 Other Distance-Dependent Attraction and Repulsion: Electrostatic a Molecular Forces in Air and Liquids, 67 2.5.1. Electrostatic Forces in Liquids: Superimposed on Van der Waals Forces, 67 	
	2.6.	 2.5.2. Molecular-Structure Forces in Liquids, 69 2.5.3. Macromolecular Steric Forces in Liquids, 72 2.5.4. Derjaguin Approximation: Colloid Probe AFM, 76 2.5.5. Macromolecular Extension Forces (Air and Liquid Media), Rate/Time Effects, 83 2.6.1. Viscoelasticity, 84 2.6.2. Stress-Modified Thermal Activation, 85 2.6.3. Relevance to Other Topics of Chapter 2, 86 References, 88 	78
3.	Z-Dej	pendent Force Measurements with AFM	91
	3.1.	Revisit Ideal Concept, 91	
	3.2.	Force-Z Measurement Components:	
		Tip/Cantilever/Laser/Photodetector/Z Scanner, 93	
		3.2.1. Basic Concepts and Interrelationships, 93	
		3.2.2. Tip–Sample Distance, 96	
		3.2.3. Finer Quantitative Issues in Force–Distance Measurements,	99
	3.3.	Physical Hysteresis, 106	
	3.4.	Optical Artifacts, 109	
	3.5.	Z Scanner/Sensor Hardware: Nonidealities, 113	
	3.6.	Additional Force-Curve Analysis Examples, 118	
		3.6.1. Glassy Polymer, Rigid Cantilever, 118	
		3.6.2. Gels, Soft Cantilever, 123	
		3.6.3. Molecular-Chain Bridging Adhesion, 126	
		3.6.4. Bias-Dependent Electrostatic Forces in Air, 129	
		3.6.5. Screened Electrostatic Forces in Aqueous Medium, 131	
	3.7.	Cantilever Spring Constant Calibration, 133	
		References, 135	
4.	Topog	graphic Imaging	137
	4.1.	Idealized Concepts, 138	
	4.2.	The Real World, 143	
		4.2.1. The Basics: Block Descriptions of AFM Hardware, 143	
		4.2.2. The Nature of the Collected Data, 149	
		4.2.3. Choosing Setpoint: Effects on Tip-Sample Interaction and	
		Thereby on Images, 156	
		4.2.4. Finite Response of Feedback Control System, 162	

CON	ΓENTS	ix
		 4.2.5. Realities of Piezoscanners: Use of Closed-Loop Scanning, 167 4.2.6. Shape of Tip and Surface, 180 4.2.7. Other Realities and Operational Difficulties—Variable Background, Drift, Experimental Geometry, 182 References, 186
5.	Prob	ing Material Properties I: Phase Imaging 187
	5.1.	Phase Measurement as a Diagnostic of Interaction Regime and Bistability, 189 5.1.1. Phase (and Height, Amplitude) Imaging as Diagnostics, 189 5.1.2. Comments on Imaging in the Attractive Regime, 200
	5.2.	Complications and Caveats Regarding the Phase Measurement, 202 5.2.1. The Phase Offset, 202 5.2.2. Drift in Resonance Frequency, Phase Offset, Quality Factor,
		and Response Amplitude, 207 5.2.3. Change of Phase and Amplitude During Coarse Approach, 211 5.2.4. Coupling of Topography and Phase, 214 5.2.5. The Phase Electronics and Its Calibration, 221
	5.3.	 5.2.6. Nonideality in the Resonance Spectrum, 230 Energy Dissipation Interpretation of Phase: Quantitative Analysis, 234 5.3.1. Variable A/A₀ Imaging, 235 5.3.2. Fixed A/A₀ Imaging, 240 5.3.3. Variable A/A₀ via Z-Dependent Point Measurements, 243
	5.4. 5.5.	Virial Interpretation of Phase, 247 Caveats and Data Analysis Strategies when Quantitatively Interpreting Phase Data, 248 References, 255
6.		ing Material Properties II: Adhesive Nanomechanics and ping Distance-Dependent Interactions 258
	6.1. 6.2.	 General Concepts and Interrelationships, 259 Adhesive Contact Mechanics Models, 261 6.2.1. Overview and Disclaimers, 261 6.2.2. JKR and DMT Models, 263 6.2.3. Ranging Between JKR and DMT: The Transition Parameter λ, 266 6.2.4. The Maugis–Dugdale Model, 270 6.2.5. Other Formal Relationships Relevant to Adhesive Contact Mechanics, 273 6.2.6. Summary Comments and Caveats on Adhesive Contact

Mechanics Models, 274 Capillarity, Details of Meniscus Force, 277

6.3.2. Basic Elements of Modeling the Meniscus, 280

6.3.1. Framing the Issues, 278

6.3.

CONTENTS X

7.

		CONTENTS			
	6.3.3.	Mathematics of Meniscus Geometry and Force, 283			
	6.3.4.				
	6.3.5.	The state of the s			
	0.5.0.	Opportunities, 293			
6.4.	Approach–Retract Curve Mapping, 296				
	6.4.1.	** *			
	6.4.2.	E .			
	6.4.3.	** *			
	6.4.4.	** * *			
		Complex Thin Films, 313			
6.5.	High-Speed/Full Site Density Force-Curve Mapping and				
	Imagin	g, 315			
		Liquidy Domains in Complex Thin Films, 317			
		PBMA/PLMA Blend at Variable Ultimate Load, 319			
		PBMA/Dexamethasone Mixture at Variable Temperature, 320			
	6.5.4.	Arborescent Styrene–Isobutylene–Styrene Block Copolymer			
		Plus Drug Rapamycin, 322			
	6.5.5.	Comments on "Force Modulation" Mode, 323			
	Referer	nces, 324			
Probi	ng Mat	erial Properties III: Lateral Force Methods 330			
	_	•			
7.1. 7.2.		nents of Lateral Force Signal, 330			
7.3.		ation of Lateral Force Difference, 336 tion of Lateral Force, 343			
7.3. 7.4.		Dependent Friction, 346			
7.7.		Motivations, 346			
		Load Stepping and Ramping Methods, 347			
7.5.		e Rate and Environmental Parameters in AFM Friction			
		ear, 352			
		Motivations, 352			
	7.5.2.	Interplay of Rate, Temperature, Humidity, and Tip Chemistry			
		in Friction, 354			
		Wear Under Variable Rate and Temperature, 359			
	7.5.4.	Musings on the Spectroscopic Nature of Friction and Other			
		Measurements, 362			
7.6.		erse Shear Microscopy (TSM) and Anisotropy of Shear			
	Modulu	•			
7.7.		Modulation Methods, 366			
	7.7.1.	Motivations and Terminology, 366			
	7.7.2. 7.7.3.	Shear Modulation During 1D Lateral Scanning, 368			
	7.7.3. 7.7.4.	Diagnostics of Sliding Under Shear Modulation, 371			
	7.7.5.	Complementarity of Shear Modulation Methods to TSM, 372 Shear Modulation Within Force Curves: Material Creep, 373			
		nces, 375			

CONTENTS xi

8.	Data Post-Processing and Statistical Analysis				
	8.1. 8.2. 8.3.	1D Ro 2D-Do 8.3.1. 8.3.2. 8.3.3. 8.3.4. "Lines	inary Data Processing, 379 ughness Metrics, 383 omain Analysis, 385 Slope and Surface Area Analysis, 385 2D-Domain Fourier Methods for Spatial Analysis, 386 Fourier Methods for Time-Domain Analysis, 391 Grain or Particle Size Analysis, 394 hape" Fitting, 396 nces, 398		
9.	Adva	nced D	ynamic Force Methods	400	
	9.1.	9.1.1. 9.1.2. 9.1.3.	Interleave Methods for Long-Range Force Probing, 405 Interleave-Based EFM/KFM on Different Metals and Silicon, 408		
		9.1.4.	KFM of Organic Semiconductor, Including Cross-Technique Comparisons, 412	ue	
	9.2.	9.2.1.	ds Using Higher Vibrational Modes, 414 Mathematics of Beam Mechanics: The Music of AFM, 414 Probing Tip–Sample Interactions via Multifrequency Dyna AFM, 419		
		9.2.4.			
App	endice	es		437	
	Appendix 1:		Spectral Methods for Measuring the Normal Cantilever Spring Constant <i>K</i> , 437 A1.1 Plan-View/Resonance Frequency Method, 438 A1.2 Sader Method, 441 A1.3 Thermal Method, 442		
	Appendix 2:		Derivation of Van der Waals Force–Distance Expressions,	443	
	Appendix 3:		Derivation of Energy Dissipation Expression, Relationship to Phase, 447	-	
	Appendix 4:		Relationships in Meniscus Geometry, Circular Approximation, 449 References, 450		
Inde	×			453	