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Acoustic Scanning Probe Microscopy

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Acoustic Scanning Probe Microscopy

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Ingenuity without poetry is like poetry without inspiration

F. Marinello

To Silvia, Angela and Angelica

Foreword

Mechanical properties of materials such as dislocation generation, fatigue, creep, crack propagation, or electrical migration in strip conductors are to a large extent determined by their microstructure. Therefore, the details of the microstructures have a strong impact on the life expectancy of a material in a given component. Materials microstructures are examined by optical microscopy, by scanning electron microscopy, and by transmission electron microscopy, often when loaded in situ mechanically or chemically.

Ultrasonic imaging as used in non-destructive testing is applied for defect detection in a component. Non-destructive materials characterization by ultrasonic imaging can be used to study the microstructure of optically nontransparent solids, in particular, metals employing scattering. In both cases, the acoustic waves penetrate into the materials, enabling one to study the microstructure of materials within the volume, to detect small defects, to study adhesive interfaces, and also to gain information about elasticity as well as absorption (also called internal friction). Ultrasonic waves of frequencies from approximately 20 kHz–2 GHz are used for acoustical imaging and mechanical spectroscopy. In acoustic imaging technologies, the contrast in reflection and transmission provides a map of the spatial distribution of elasticity, density, ultrasonic absorption and scattering, and the occurrence and distribution of defects. These parameters in turn may be used to obtain information on the mechanical properties as defined above, although often only by calibration with test components of known properties because the interrelatedness of the various parameters is often too complex, so that an appropriate analytical formula does not exist. There are many books, handbooks, and review articles providing a detailed account of acoustical imaging for medical, material science, and non-destructive testing applications.

Acoustical imaging modes can be classified into near-field imaging techniques, focusing techniques, and holographic techniques. Examples of near-field imaging techniques are contact oscillators like the Fokker bond test system for monitoring adhesive bonds in an airplane wing. They are operated in a frequency range covering some kHz to some 100 kHz. Their spatial resolution depends on the

antenna size, i.e., the probe size and not on the frequency and hence on the wavelength employed.

Due to the smaller scale of components, in particular, in microelectronics, there was always the demand to obtain higher and higher spatial and temporal resolutions in acoustical imaging systems. This became possible with (a) the ever-increasing capabilities of computers allowing one to store the huge amount of data which followed; (b) the use of operating frequencies beyond 20 MHz for obtaining higher spatial resolution based on focusing probes, and (c) the increase of the bandwidth of the electronic receiving system to increase the temporal resolution of the imaging system. This led to the development of scanning acoustic microscopy (SAM), sometimes also called high-frequency C-scan imaging. Whereas, the physical principle of SAM was known for a long time, it took some efforts in the 1980s to engineer reliable systems. At room temperature, the highest frequency attainable in SAM is approximately 2 GHz, because the attenuation in the liquid water used as couplant necessary to transmit the ultrasonic signals from the acoustic lens to the material to be examined becomes so high that more than 99 % of the ultrasonic power gets absorbed. Even if one uses liquid metals like gallium or mercury as a couplant serving also for impedance matching, the situation does not improve much. Wavelengths at GHz frequencies are some micrometers, depending on the sound velocity. Hence, in an acoustical imaging system using a focusing transducer or an acoustical lens, the spatial resolution is at most 1 μm. Having this technological barrier in mind, it was logical to exploit the principle of near-field imaging, where the resolution is given by the size of the antenna and less by the frequency. This comes at the cost of being able to image only the surface of a component or a material. Such efforts have been undertaken by various groups parallel to the development of SAM.

A further step toward higher resolution based on the near-field principle became possible with the advent of scanning tunneling microscopy (STM) and later of atomic force microscopy (AFM). There were early attempts to construct a near-field ultrasonic microscope based on an STM which, however, was not much pursued because it could only be used in high vacuum and on metals. The situation changed with the invention of the AFM. In atomic force microscopy, a micro-fabricated elastic beam with a sensor tip at its end is scanned over the sample surface and generates high-resolution images of surfaces. The tip radius is typically from a few nm to 100 nm. The contact radius at the surface is much smaller and even atomic resolution is possible with an AFM. It can be operated in ambient conditions for many applications. Thus, it was natural to combine AFM with ultrasonics in order to exploit its high, resolution capacity for acoustical imaging.

Very early in the development of atomic force microscopy, dynamic modes such as force modulation where the cantilever or the sample surface is vibrated, belonged to the standard equipment of most commercial instruments, allowing one to image the surface of a material, where the contrast depends on the elasticity, the friction, and the adhesion of the tip–sample contact, in particular on compliant materials. The quantitative determination of the Young's modulus of a sample surface with an AFM was a challenge however. Especially when stiff materials

such as metals or ceramics were encountered, the image contrast due to elasticity was very low in force modulation, because the spring constants of common AFM cantilevers, ranging from 0.01 to 70 N/m, are then much lower than the tip–sample contact stiffness. This barrier can be overcome by using the atomic force acoustic microscopy (AFAM) technique, or by ultrasonic atomic force microscopy (UAFM), or similar schemes. One measures the resonances of atomic force cantilevers with the tip contacting the specimen surface, hence often the term contact resonances is used for this class of dynamic atomic force microscopies. From such measurements, one can derive the local contact stiffness k^* and by using a suitable mechanical model for the contact stiffness, one can invert k^* data to measure the local indentation modulus M. The indentation modulus is an elastic constant which accounts for the compressive and the shear deformations in the contact zone between isotropic or anisotropic materials. Similarly, one can gain information on the anelastic part of the indentation modulus, which entails information on the local friction and adhesion within the contact zone and on the material's internal friction within the contact volume. In AFAM, the cantilever with its tip plays the role of the horn in impedance spectroscopy or of the contact oscillators in the Fokker bond tester and the tip–sample contact serves to probe the local mechanical impedance. Due to the small tip radii, the spatial resolution at the surface of the material examined is, however, much smaller and of nanoscale, and resolution much below 10 nm can be obtained if measurement parameters are set right. As it turned out, there is a multitude of factors determining the obtainable spatial resolution, the physical background of the contrast, and the oscillatory behavior of the cantilever when using an AFM tip as acoustical near-field antenna. It stems from the richness of the forces between tip and surface which can be adhesive, elastic, electrical, and magnetic in a linear and nonlinear fashion and because an AFM cantilever can be excited to many vibrational modes.

The authors contributing to this book, perfectly edited by F. Marinello, D. Passeri, and E. Savio, give a first-hand account on the status of the various AFM contact-resonance techniques, the theory of their operation, and the tip–sample contact mechanics. The authors provide many examples of applications and therefore serve the AFM as well as acoustical imaging communities and also those who want to apply these techniques for studying elastic, anelastic, and mechanical properties on the scale of some nanometers, and finally those who want to further develop the techniques.

What might lie ahead? I think that an improved spatial resolution can be achieved by using tips with radii much below 50 nm loaded with static forces of some nN to some 10 nN. This would allow one to examine compliant materials and hence may open the door to image biological samples and to obtain quantitative data as discussed in a chapter of the book. Such improved contact-resonance techniques should allow one to image the nanostructure of materials as well and to shed more light on the local phenomena which are behind adhesion, hardness, yield stress, elastic stresses, closing the circle to conventional acoustical imaging. Then, there is the urgent need to increase the depth sensitivity of the contact-resonance techniques for defect detection which can be achieved by an opposite

approach, using very stiff cantilevers or exploiting the higher cantilever modes with their effective higher stiffness and larger contact radii. This calls for wear-resistant tips. Finally, by using modulated propagating waves in the GHz range demodulated by the nonlinear tip–sample contact, one should be able to exploit ultrasonic scattering to study detailed features of the microstructure, for example, of materials employed in microelectronics, defects buried in wafers deeper than the Hertzian contact stress-field or in biological cells.

Saarbrücken and Göttingen W. Arnold

Foreword

Advancements in virtually all areas of science and technology demand materials with improved performance. In the past decades, we have witnessed new materials being continually introduced for commercial use in diverse areas like electronics, construction, transportation, textiles, and in medical devices and implants. Key to these new developments is the ability to engineer materials on the nanoscale by incorporating a multitude of components and geometric features. The resulting heterogeneity and complexity of materials call for novel characterization technologies with nanoscale spatial resolution.

Scanning probe microscopes, in particular, atomic force microscopes have played an important role in visualizing materials with nanoscale features. Owing to their mechanical operation principles, there is now a significant potential for the use of atomic force microscopes in measuring and mapping mechanical properties of nanoscale materials. A variety of techniques has already been introduced and their accuracy and range of applicability are continuously improving with an accelerating pace. Consequently, a vast literature on this subject has emerged. In that regard, Francesco Marinello, Daniele Passeri, and Enrico Savio have put together a great sourcebook on scanning probe microscopy-based nanomechanical characterization. This timely book provides a good introduction to newcomers and a thorough source of references and reviews for those already in the field.

Despite the popularity of atomic force microscopes in imaging nanoscale materials, generating quantitative information about material properties has proven difficult. As contributing author Donna C. Hurley puts it; developments in this field have been successful in generating "pretty pictures" from the nanoscale world, with qualitative contrast mechanisms. Characterization of advanced materials, however, requires reliable quantitative measurements of mechanical properties. Inaccuracies can be introduced to the measurements at various stages of information transduction. The book investigates two of the most critical stages in great depth: the contact mechanics that govern tip–sample interactions and the dynamics of the vibrating cantilever. Both intuitive and rigorous treatments of these subjects merge in the book, allowing readers from various backgrounds to benefit from the material.

Once equipped with the basic understanding of the underlying theories of contact mechanics and cantilever dynamics, the reader finds contributed chapters from leading experts in acoustic AFM and related experimental techniques, reviewing what is possible in the current state of the art. The authors share valuable tips in getting reliable measurements. I find it especially helpful that the book devotes a chapter for an in-depth comparison of the quantitative measurements obtained by scanning probe microscopy with more established techniques like instrumented indentation and surface acoustic wave spectroscopy.

The book includes contributions beyond the more established methods. The rise in demand for research in developing advanced nanomaterials is fueling the expansion of the nanomechanical characterization toolbox. Tools geared toward "soft matter" and tools providing contrast from below the surface of materials are rapidly advancing. By incorporating several examples of new techniques, including the applications of acoustic characterization techniques in biological problems, the book provides a breadth of topics that makes it a valuable sourcebook for anyone interested in nanomechanical analysis.

Columbia University, New York, USA Ozgur Sahin

Preface

The rapid progress of nanotechnologies poses significant challenges in manufacturing and characterization. Scanning Probe Microscopy (SPM) techniques have significantly contributed to such development, allowing characterization of a number of properties at the microscale and nanoscale. Having been invented for the morphological investigation of surfaces, SPM has represented the basis for the development of techniques where the tip is used for probing physical properties and the SPM position control system is used for imaging such properties on the samples surface, simultaneously to their topography.

The combination of scanning probe microscopy, and in particular of Atomic Force Microscopy (AFM) with ultrasound techniques, led to the development of acoustic AFM (A-AFM) and acoustic SPM (A-SPM) opening up to a number of measuring techniques which allow surface mechanical properties imaging.

In A-AFM, piezoelectric transducers are used to set the sample surface or the AFM cantilever into vibration at ultrasonic frequencies that are well above the cutoff frequency of the electronics, so that the oscillations are not compensated by the feedback. As a consequence such oscillation does not influence the standard topographical reconstruction, and on the other hand, the ac component of the deflection signal is not suppressed and thus can be subsequently analyzed. The particular way in which ultrasonics and SPM are combined is different for each specific technique and allows collection of different information.

Readers working in different fields of nanotechnology, material science, and biology will find in this book a comprehensive overview of such A-SPM techniques, presented by evidencing similarities and peculiarities. We proudly say that the most widely recognized scientists and researchers have contributed to the 17 chapters of the present volume, discussing acoustic SPM techniques both from the theoretical and from the practical points of view. The volume is divided into three parts.

The first part includes three chapters on subjects that form the basis of all A-SPM techniques, namely, the contact mechanics describing the tip–sample interaction, the analytical models for the dynamics of the cantilevers interacting

with the sample in the different A-SPM modalities, and numerical methods for their simulation.

The second section describes the most important A-SPM techniques emphasizing recent advances: Atomic Force Acoustic Microscopy (AFAM), Ultrasonic Atomic Force Microscopy (UAFM), Scanning Microdeformation Microscopy (SMM), Ultrasonic Force Microscopies (UFM) and related techniques, Scanning Near-Field Ultrasound Holography (SNFUH), and Torsional Harmonic Atomic Force Microscopy (TH-AFM). Two chapters are dedicated to quantitative data extrapolation, presenting strategies for enhancing the sensitivity of such techniques allowing exploitation of measuring performance and discussing the main points of data post processing, providing hints and strategies for repeatable analysis of surface data sets. The presentation of A-SPM techniques is completed with a comparison between quantitative elastic measurements by A-SPMs and conventional techniques (i.e., nanoindentation and surface acoustic wave spectroscopy).

The third section reviews applications of A-SPM. Two chapters are devoted to quantitative aspects in the characterization of friction and internal friction and in subsurface imaging. Finally, the last two chapters describe some recent results in the quantitative mechanical characterization of polymers and of biological samples.

We gratefully acknowledge the support of all authors. We also wish to thank Springer, and in particular Mr. Claus Ascheron, for his initiative to setup this volume and his organizational work. We sincerely hope that readers will find this volume scientifically stimulating and rewarding.

Padua, Rome

Francesco Marinello
Daniele Passeri
Enrico Savio

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Symbols

α_0	
A	Cantilever cross-section area
a_c	Contact radius
b	Cantilever thickness
E	Young modulus of the cantilever
E^*	Reduced modulus of the contact
E'	Storage modulus
E''	Loss modulus
E_t, E_s	Young's moduli of the tip and of the sample
f	Frequency
f_0	Free resonance frequency
$F_\perp$	Is the static load, i.e. the normal component of instantaneous force interacting between the tip and the surface, in the coordinate system of the sample surface
$F_{//}$	Lateral load, i.e. the normal component of force interacting between the tip and the surface in the coordinate system of the sample surface
F_m	Time-averaged force between tip and sample
φ	Tilt of the cantilever relatively to the surface
G^*	Reduced shear modulus of the contact
γ	Adhesion energy
Φ	Phase
γ	Normal damping constant
γ_{lat}	Lateral damping constant
h	Tip height
h_1	Initial sample indentation
h_{eq}	Equilibrium indentation
k^*	Normal contact stiffness
k^*_{lat}	Lateral contact stiffness
k_c	Static spring constant of the cantilever
k_n $\{n = 1, 2, \ldots\}$	Wave numbers of the nth eigenmode

k_s	Linear contact stiffness
L	Total length of the cantilever
L_1	Actual distance between the tip and the chip
L_{eff}	Effective length of the cantilever
λ	Wavelength
χ_s	Quadratic contact stiffness
M	Indentation modulus
m^*	Effective mass of the cantilver
m_c	Additional or concentrated mass
ns	Nanosecond (10^{-9}) s
R	Tip radius of curvature
r_c	Radius of contact area
S_{eff}	Differential contact stiffness
ρ	Density of the cantilever
t	Film thickness
τ	Relaxation time
v_t, v_s	The Poisson's ratios of the tip and of the sample
w	Cantilever width
z_{eq}	Equilibrium cantilever deflection

Acronyms

AFAM	Atomic Force Acoustic Microscopy
AFFM	Acoustic Friction Force Microscopy
AFM	Atomic Force Microscopy
AM-AFM	Amplitude Modulation AFM
CM	Concentrated Mass
DOF	Degree of Freedom
FEA	Finite Elements Analysis
FFM	Friction Force Microscopy
FM-AFM	Friction Modulation AFM
FMM	Force Modulation Microscopy
FRF	Frequency Response Function
HFM	Heterodyne Force Microscopy
NC-AFM	Non Contact AFM
NI	Instrumented (nano-) Indentation
SAFM	Scanning Acoustic Force Microscopy
SAM	Scanning Acoustic Microscopy
SAWS	Surface Acoustic Wave Spectroscopy
SLAM	Scanning Local Acceleration Microscopy
SMM	Scanning Microdeformation Microscopy
TM	Tapping Mode
UAFM	Ultrasonic Atomic Force Microscopy
UFM	Ultrasonic Force Microscopy
W-UFM	Waveguide Ultrasonic Force Microscopy

Chapter 1
Acoustic Scanning Probe Microscopy: An Overview

D. Passeri and F. Marinello

Abstract In this chapter, which serves as an introduction to the entire book, an overview is given of techniques resulting from the synergy between ultrasonic methods and scanning probe microscopy (SPM). Although other acoustic SPMs have been developed, those reviewed in this book are either the earliest proposed techniques, which are most widespread, extensively used, and continuously improved, or have been recently developed, but have been proved to be extremely promising. The techniques are briefly introduced, emphasizing what they have in common, their differences, their capabilities, and limitations.

1.1 Touching Instead of Seeing

The invention in the 1980s of the two main scanning probe microscopy (SPM) techniques, namely atomic force microscopy (AFM) [1] and scanning tunneling microscopy (STM) [2–5], extended the significance of microscopy, giving it a wider acceptation beyond its mere etymological significance. Deriving from the Greek $\mu\iota\kappa\rho\acute{o}\nu$ (transliterated as 'mikron', meaning 'small') and $\sigma\kappa o\pi\acute{\varepsilon}\omega$ (transliterated as 'skopeo', meaning 'I see' or 'I look'), the word 'microscopy' recalls the idea of seeing 'by eyes' and thus by instruments where the visualization of objects is based on the collection of the light diffracted by them by means of suitable lenses. The observability of small objects is thus limited by the wavelength λ of the particular electromagnetic radiation used for illuminating them: the lower the λ the higher the

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resolution, where the latter can be evaluated for instance by the Abbe's criterion as $\lambda/2NA$, where NA is the numerical aperture of the optical lens. To increase the resolution, λ must be reduced. Such a requirement suggested the use of radiation more energetic than visible light, leading to the invention of X-ray and electron microscopy.

SPM techniques are based on a completely different idea. A tip is brought in close proximity to the sample surface and then is moved across it in two directions (namely, the x and y axes). At each point of the surface, which is divided into a homogeneous array of points, a physical parameter is monitored. In the case of STM, such a parameter is the tunneling current flowing between the (conductive) sample and the (conductive) tip. In the case of AFM, such a parameter is the deflection of the cantilever at the extremity of which the tip is mounted. More precisely, the monitored parameter is the cantilever static deflection in AFM contact mode, while it is the amplitude of the oscillating cantilever in AFM semi-contact mode. These parameters can be collected at each point of the scanned area and reported in maps that qualitatively reflect the sample morphology (the open feedback loop mode). In practice, this operation mode exposes the tip to the risk of abrupt damage and thus is scarcely used except in case of very flat sample surfaces (e.g., when atomic resolution is required). Alternatively, the acquired parameter is used as the input signal of a feedback loop that maintains a constant value over the scanned surface by acting on a piezoelectric transducer in the vertical direction, namely, the z axis (the closed feedback loop mode). This modifies the cantilever-to-sample distance by an amount Δz that is equal to the local height variation of the surface. The value of Δz at each point of the scanned area is reported in a map that quantitatively reflects the sample topography. It is worth noting that in the case of AFM operation in contact mode, the closed feedback loop ensures that the surface is scanned at constant value of the cantilever deflection and thus of the force exerted between tip and sample, which is an important requirement in contact mode acoustic AFM techniques, as described below.

As discussed in the following, imaging performed by *touching* (in the sense of a tip coming into close interaction with the surface), rather than *seeing*, the surface has its own disadvantages, but on the other hand offers the possibility of going beyond topography by developing unique tools for the qualitative and/or quantitative characterization of several physical properties of the sample surface.

1.1.1 Facing the Limitations...

The reconstruction of a sample morphology by touching and scanning its surface has its own disadvantages. As a direct consequence of touching the surface, collected images are the convolution of both surface features and tip shape, resulting in artifacts that can seriously compromise the quality of the image (e.g., nanoparticles on flat surfaces may lead to images where the apex of the tip is reproduced inverted in correspondence with each nanoparticle) unless a proper deconvolution is performed

[6]. Moreover, in the case of soft samples like polymers or biological specimens, the interaction between tip and sample may contaminate the former and/or damage the latter [7]. These drawbacks can be prevented or reduced by operating in semi- or non-contact mode instead of contact AFM mode.

As a consequence of scanning, movement limitations are introduced by both the z direction (vertical) piezoelectric actuator and the x and y direction (in-plane) scanners. The limitation of the vertical range implies a sufficient flatness of the surface to be analyzed: when such a requirement is not met, only restricted portions of the surface can be imaged, thus reducing the statistical meaning of the SPM investigation. The limitation of the in-plane scanners does not allow the visualization of large areas even for perfectly flat samples, thus not permitting overall visualization of surfaces, fast selection and positioning on specific sample regions, or characterization of features with widely different magnifications, all characteristics that, conversely, allow electron microscopy to collect images that in some cases are admittedly astonishing.

1.1.2 ... and Converting them into Opportunities

Despite such disadvantages, imaging by touching and scanning the sample surface turned out to represent a key feature that determined the success of SPM techniques as the basis for the development of a wide number of tools to image, measure, and map several physical properties simultaneously with samples' topography. Touching surfaces allows one to probe mechanical, electric, and/or magnetic (e.g., by using AFM cantilevers coated with conductive and/or magnetic films) properties. Scanning surfaces allows one to repeat such measurements at each point and thus to map the measured physical properties over the surface simultaneously with the morphological reconstruction. In some cases new techniques have been developed based on standard SPM setups, while in other cases researchers have reproduced at micro- and nanoscales techniques already available at macroscales. For example, the tip is used from time to time as an indenter, as the probe of a multimeter, etc. Such an approach enables measurements with nanometrical lateral resolution and the collection of qualitative maps of properties beyond the topography, although they are generally affected by artifacts induced by topography itself. Gathering accurate quantitative data is nevertheless limited by the nonideal instrumental parameters such as the real shape of the tip. Theoretical models are thus needed to analyze data that are based on, but generally more complex than, those used by more conventional instruments. A comprehensive review of such techniques far exceeds the aims of this book. In the following we limit our attention to some of the techniques that combine ultrasonic methods with AFM tools for the surface and subsurface mechanical characterization of samples.

1.2 Two Points of View

Acoustic or ultrasonic SPM (A-SPM) refers to a class of several different techniques that are characterized by the use of almost standard SPM setups, integrated with some modified electronics and/or mounting specifically functionalized tips. Both AFM and STM setups have been used for developing A-SPM techniques (A-AFM and A-STM, respectively). Nevertheless, in the following we refer only to the AFM-based ones, which are undoubtedly more widespread and versatile. In A-AFMs, piezoelectric transducers are used to set the sample surface and/or the AFM cantilever into vibration at ultrasonic frequencies that are well above the cutoff frequency of the electronics, so that the oscillations are not compensated by the feedback. This ensures that such oscillation does not influence the standard topographical reconstruction, as well as that the ac component of the deflection signal is not suppressed and thus can be subsequently analyzed. These two represent the key points for the simultaneous acquisition of topography and acoustic signal images. The particular way in which ultrasonics and SPM are combined is different for each specific technique and will be described in detail through the chapters of the book. Here, the interest is focused on the common features of these techniques. The enrichment produced by the combination of ultrasonics and SPM can be fully understood by looking at such a combination from two different and complementary points of view. From the first viewpoint, A-SPM techniques can be regarded as nanoscale versions of dynamic indentation tests: the SPM tip replaces standard indenters and the effect of ultrasounds is to modulate the indentation of the sample surface. From the second viewpoint, A-SPMs can be regarded as nanoscale versions of scanning acoustic microscopy techniques: the tip is used for probing the acoustic wave field with high spatial resolution, far beyond the limitation imposed by other methods such as the use of piezoelectric transducers, light wave diffraction, X-ray scattering, or electron reflection. These two points of view are characterized by different approaches, models, and mathematical instruments for rationalizing the results of the experiments. Such grouping can be somewhat limiting, since each technique can be described in terms of each of the two approaches; however, it can be useful to understand the role of ultrasonics in SPM-based techniques.

1.2.1 Modulating the Indentation of the Surface

Used for setting into vibration the sample surface and/or the cantilever, acoustic waves produce a modulation in the cantilever-sample distance. In case of infinitely stiff sample and tip, such a modulation is entirely observed as the modulation of the cantilever deflection. In the case of a sample much more compliant than the cantilever, the modulation results partially in the modulation of the cantilever deflection and partially in a variation of the penetration depth of the tip into the sample surface: the softer the sample, the higher the modulation amplitude of the indentation and the

lower that of the cantilever deflection. Therefore, the oscillating component of the cantilever deflection can be acquired at each point of the scanned area, thus obtaining an image which is related to the surface elastic modulus. This idea forms the basis of the force modulation microscopy (FMM) technique [8, 9], which has been proved to allow qualitative elastic imaging of soft samples like polymers. Implementation of FMM on materials with higher elastic modulus is indeed limited by the availability of standard cantilevers with sufficiently high spring constant values. In this sense, the merit of ultrasonics is the stiffening of AFM cantilevers at high frequencies: in other words, the cantilever dynamic spring constant values are far higher than the static ones. Therefore, ultrasonics enables dynamic indentation measurements by AFM on relatively stiff samples, especially when combined with ad hoc designed cantilevers having higher static spring constants [10, 11] and/or tips harder than the standard Si or Si_3N_4 ones [12].

1.2.2 Detecting the Near-Field Acoustic Waves

Widely used for nondestructive testing, ultrasonic waves are employed in the so-called scanning acoustic microscopy (SAM) technique [13–15], which enables the imaging of sample surface elastic properties at submicrometer scale with resolution that highly depends on the ultrasonic wavelength in the investigated material. In a reflection acoustic microscope in the linear regime, the resolution is slightly better than that established by the Rayleigh criterion for a conventional microscope and is $0.51\lambda_0/NA$, where λ_0 is the ultrasonic wavelength and NA the numerical aperture of the acoustic lens [16]. Acoustic microscopy takes advantage of the use of surface acoustic waves (SAWs) (also known as Rayleigh waves), whose amplitude exponentially decays into the material as the distance from the surface increases. In other words, SAW energy is confined in a volume of material underneath the surface down to a depth of a few times the wavelength. Therefore, acoustic microscopy is sensitive to the mechanical properties of the material in a volume included from the sample surface to a depth of a few times the wavelength into its interior. The contrast in SAM images is therefore produced by the variation of elastic modulus, as well as by the presence of subsurface defects, voids, and delamination [15]. The acoustic field diffracted by an object is generally composed of both propagating and evanescent waves [17]. The former can be collected by SAM, while the latter—whose amplitude exponentially decays as a function of the distance from the object—cannot propagate up to the piezoelectric transducer acting as the receiver. As the spatial Fourier transform of the diffracting object is involved, the smaller its dimension the more predominant is the evanescent component with respect to the propagating one [17]. The spectrum emerging from nanosized objects that are easily detectable by AFM is generally only composed of evanescent waves, and thus such objects are invisible to SAM. Nevertheless, if the diffracting features are at the interface or under but in proximity to the surface investigated by AFM, the tip can be used as a mechanical probe to collect the evanescent—but not yet extinguished—diffracted waves. In practice, the unique