Scanning probe - atomic force microscopy: new developments and applications

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Abstract. Recent developments in scanning probe microscopy (SPM) have widened the spectrum of possible investigations that can be performed at a nanometric level at the surfaces of materials. They range, for instance, from physical properties such as surface potential and electric field topological determination to chemical, nanomechanical, catalytic and spectroscopic analysis in air in liquid or in a gaseous environment. After a brief introduction to new SPM technological developments, we present recent achievements in the characterisation and application of nanomorphology, electrostatic surface potential, dielectric properties, shear force microscopy and radiofrequency measurements, scanning microwave microscopy, nanomechanical properties mapping, frequency modulation SPM in air, liquid, vacuum, and finally high-speed SPM.

1. Introduction

Scanning probe microscopy (SPM) is facing a continuous and rapid development of methodologies thanks to the technological advancements of basic components, innovative detection systems and completely new approaches.

The newly available methodologies allow performing a variety of nano-confined measurements of materials physico-chemical properties. Structure and morphology, electrostatic potential and radiofrequency measurements at sample surfaces, viscoelasticity, adhesion and elastic modulus, and charge density mapping are some of the properties nowadays measurable with nanoscale spatial resolution. Furthermore, true atomic resolution can now be achieved in air, vacuum and liquid environment by implementing frequency modulation atomic force microscopy (FM-AFM).

In this paper we will firstly introduce Kelvin probe force microscopy (KPFM) for the study of surface potential, scanning polarisation force microscopy (SPFM) for liquid films imaging and dielectric mapping, and scanning microwave microscopy (SMM) for the study of carrier behaviour in semiconductor devices. Then, shear force microscopy (ShFM) together with a new radiofrequency (RF) methodology for the simultaneous measurements of viscoelastic/shear properties and impedance characterisation in the RF range of sample surfaces will be presented. Some SPM-based methodologies for nanomechanical property mapping will be reviewed. Finally, a section dedicated to atomic resolution AFM capability in air, liquid and vacuum, and the recent efforts to the development of high speed AFM will be presented and discussed.
2. Electrostatic surface potential, dielectric properties measurements and scanning microwave microscopy

2.1. Kelvin probe force microscopy
Kelvin probe force microscopy (KPFM) is a methodology that enables nanometre-scale analysis of surface electrical property of metallic, semiconductor and insulating materials [1]. For instance, KPFM was employed recently to investigate the surface potential of phyllosilicate minerals and the interactions between mineral surfaces and fundamental biomolecules such as DNA, RNA and nucleotides [2-7]. It was found that the nanoscale surface potential distribution of phyllosilicates drive the adsorption, condensation and conformation of the biomolecules.

The “classical” KPFM is a dual pass mode, i.e., during the first pass the surface topography is recorded, then during the second pass the electrostatic interaction is measured by raising the tip above the sample surface following the recorded topographic profile. By this method only the long-range electrostatic interactions are measured and the cross-talk between topography and surface potential measurements is eliminated. However, increasing the tip-sample separation decreases the lateral resolution. The single pass methods allow performing AFM and KPFM imaging simultaneously, decreasing the tip-sample separation and thus increasing the spatial resolution. Furthermore, topographical errors caused by electrostatic forces are minimized because of the active suppression of the electrostatic forces during the simultaneous AFM and KPFM measurement.

In KPFM a conductive tip is biased directly by \( V_{tip} = V_{dc} + V_{ac} \sin(\omega t) \). In the case of a metallic sample, the first harmonic of the force, \( F_{\omega} \), is related to the difference between the constant component of the tip bias \( V_{dc} \) and the surface potential \( V_s \) by

\[
F_{\omega} = \frac{\partial C(z)}{\partial z} (V_{dc} - V_s) V_{ac} \sin(\omega t)
\]

where \( C(z) \) is the tip-surface capacitance, which is dependent on the tip size and geometry, surface topography and tip-surface separation \( z \). By adjusting \( V_{dc} \) to nullify the force, we obtain point by point a surface potential map of \( V_s \).

In the case of a semiconductor sample, \( F_{\omega} \) is derived as

\[
F_{\omega} = \frac{Q_s}{\varepsilon_0} \frac{C_i C_D}{C_i + C_D} V_{ac} \sin(\omega t)
\]

where \( Q_s \) is total charge near the semiconductor surface due to the surface potential of the semiconductor, \( \varepsilon_0 \) is the dielectric constant, \( C_i \) is the capacitance associated with \( V_{ac} \) and the air gap between tip and sample, and \( C_D \) is the capacitance associated with \( V_{ac} \) and the space-charge-layer in the semiconductor [8].

In the configuration of a sample with fixed charges at the surface, placed between a conductive plane substrate and the tip, \( F_{\omega} \) can be written as:

\[
F_{\omega} = \left[ \frac{\partial C(z)}{\partial z} (V_{dc} - V_s) - \frac{Q_s C(z)}{4\pi\varepsilon_r\varepsilon_0 z^2} \right] V_{ac} \sin(\omega t)
\]

where \( C(z) \) is the tip-substrate capacitance, \( Q_s \) is the static charge, \( \varepsilon_0 \) is the permittivity of free space, and \( \varepsilon_r \) describes the dielectric constant of the materials between the conductive plane substrate and the tip [9, 10].
KPFM have been applied to the characterisation of the electrical properties of metallic nanostructures [1], to the study of surface charges of semiconductor [11], to probe semiconductor devices [1], and study the surface potential of mineral surfaces [12, 13].

A considerable resolution improvement has been achieved in KPFM in the last decade, accompanied by significant advancements in the quantitative understanding of the observed contrast, with the aid of analytical and numerical studies [14, 15]. The electrostatic force $F_\omega$ can be detected by employing an amplitude modulation (AM) or a frequency modulation (FM) device [1]. Generally, in single pass KPFM, when electrostatic forces are applied to the tip, the additional oscillating components of the electrical force will be superimposed to the mechanical oscillation. In single pass AM-KPFM, the topography is measured by the oscillation at the first resonance frequency (mechanical excitation), and the surface potential by the amplitude of the oscillation at the second resonance frequency (electrical excitation) to minimize the cross-talk with the topographical signal. In FM-KPFM, $F_\omega$ is detected by the frequency shift at $\omega$, and $V_{DC}$ is applied to the AFM tip to nullify the frequency shift. $V_{AC}$ induces a modulation of the electrostatic force detected by the superimposed oscillation at the frequency variation of the mechanical oscillation.

Generally, in FM-KPFM the spatial resolution is mainly related to the dimension of the tip apex, whereas in AM-KPFM the spatial resolution is reduced by an averaging effect between the tip and cantilever. Atomic resolution single pass FM-KPFM of semiconductor surfaces was demonstrated in UHV [16]. Figure 1a shows the topography of a Pb/Si (111)-(7x7) surface with substitutional Pb adatoms as higher protrusions and figure 1b the corresponding FM-KPFM image.

![Figure 1. a) Topography and b) simultaneously measured FM-KPFM image of an area of the Si (111)-(7x7) surface with substitutional Pb adatoms (brighter protrusions in a)). (Modified from Sadewasser et al. [16]).](image)

Recently, a multiscale approach for simulations of Kelvin probe force microscopy with atomic resolution has been presented [17] in order to obtain a better theoretical understanding of the distance dependence and atomic-scale contrast, recently observed in KPFM measurements. The electrostatics of the tip-cantilever-sample system was treated by a finite-difference method, and the resulting electric field under the tip apex was applied to atomistic wavelet-based density functional theory calculations. The attention was focused on insulating samples and on the interaction of a silicon tip with a NaCl(001) surface. Furthermore, analytical formulae were obtained for the AM- and FM-KPFM signals, taking into account for macroscopic and microscopic bias-dependent forces.

2.2. Scanning polarisation force microscopy

Another interesting approach which involves the polarisation force between an electrically charged AFM tip and a substrate is scanning polarisation force microscopy (SPFM) [18, 19]. SPFM is a non-contact mode particularly useful to study liquid films and droplets, providing information on
topography, dielectric properties and contact potential variation. A conductive probe is brought 10 - 20 nm above the sample surface and electrically biased by $V_{tip} = V_{dc} + V_{ac} \sin(\omega t)$. Two lock-in amplifiers are used to detect the forces at the first and second harmonics experimented by the probe. The oscillation amplitude of the second harmonic component is maintained constant by controlling the probe-to-sample distance. Topography and sample polarisability are mixed in images based on detecting the second harmonic term of the electrostatic force. A second feedback loop measures the probe-surface contact potential difference as in KPFM, based on the first harmonic term detection. As a sake of example, SPFM was successfully used to directly image the evolution of the structures of thin water films on BaF$_2$(111) as a function of relative humidity, RH [20], to study the adsorption of water on alkali halides (KBr, KCl, KF, NaCl) nanocrystals on SiO$_2$ and their deliquescence as a function of RH [21], and to measure the mobility of muscovite mica surface ions at different humidity levels [22].

2.3. Scanning microwave microscopy

Very recently scanning microwave microscopy (SMM) was developed to investigate carrier behaviour in semiconductor devices. A continuous microwave signal is applied from a vector network analyser to an AFM conductive cantilever with a tall tip, and the reflected portion is analysed [23]. The amplitude of the reflection coefficient was calibrated for quantitative measurements of the capacitance at the tip sample interface, enabling two dimensional mapping of the carrier density across different regions of a semiconductor. SMM is applied for the characterisation and performance modelling of semiconductor devices and failure analysis. SMM was used for calibrated capacitance spectroscopy and dopant profiling measurements in the frequency range from 1 GHz to 20 GHz. As an example, figure 2 shows a topographic and $dC/dV$ image of a bipolar SRAM (static random access memory) integrated circuit device. Several p-n defect structures in the n-doped channels can be identified in the dopant profiling image. A sketch depicts p- and n-doped areas in figure 2a, which corresponds well to the various doping types and concentrations in the $dC/dV$ image.

![Figure 2.](image)

Figure 2. a) Sketch of the p- and n-doped areas of a static random access memory (SRAM). b) AFM topographical image of a SRAM sample. c) $dC/dV$ phase image of the same area as in b) with defect structures (arrowed) in the n-doped channels (modified from Huber et al. [23]).
3. Shear force microscopy, radiofrequency measurements and nanomechanical properties mapping

3.1. Shear force microscopy and radiofrequency measurements
Shear force microscopy (ShFM) [24, 25] is a non-contact imaging methodology that uses a cantilever oriented in a vertical configuration with respect to the sample surface, i.e., with the long axis of the cantilever perpendicular to the sample surface. The main advantage of this geometry is the absence of the jump-to-contact. This phenomenon arises in the horizontal configuration when the gradient of the surface attractive force overcomes the cantilever spring constant. As a consequence the lateral stiffness of the cantilevers can be reduced by more than two orders of magnitude with respect to the softest commercially available cantilevers [26]. Furthermore, the probe at the free-end of the cantilever (figure 3), which is oscillated in a direction parallel to the sample surface by a piezoelectric element, is exclusively sensitive to horizontal forces (shear forces) and achieves a very low probe to sample interaction force. This force is considered to be mediated by the water confined between the sample surface and the cantilever apex. When the probe is in the non-contact shear force regime, few nanometres from the surface, the monotonic relation between the cantilever oscillation amplitude and the probe-sample distance is used to image the sample surface by scanning the surface at a constant oscillation amplitude [25, 27]. Thanks to this features ShFM was used, for instance, to investigate molecular layering and layer viscoelastic properties in confined liquid films [25, 28], the effect of surface-adsorbed cations on the viscoelastic response of water to shear in sub-nanometre confinement [29], mechanical properties of biomolecular motors [30], and also as a feedback system in scanning near-field optical microscopy [31, 32].

Figure 3. Scanning electron microscopy micrograph of a commercial AFM cantilever oriented in a vertical configuration. The sensing element is the apex at the free-end of the cantilever (arrowed A).

Ulcinas and co-workers [29] measured the viscoelastic response to shear of water in a sub-nanometre confinement by detecting the thermal noise of a vertically oriented microcantilever. This response was related to the effect of surface-adsorbed cations, which act as pinning centres restricting the mobility of the confined water molecules. Measurements were performed in pure water as well as under acidic and high-ionic-strength conditions.
By means of this methodology information about the viscoelastic properties of a fluid can be extracted, being the thermal noise connected to the local mechanical susceptibility. From the fluctuation-dissipation theorem,

\[ |u|^2(\omega) = \frac{k_B T}{\pi \omega} \chi''(\omega) = \frac{A \gamma}{(\omega_0^2 - \omega^2) + \gamma^2 \omega^2} \]

where \( u \) is the displacement, \( \omega \) is the frequency, \( \chi'' \) is the imaginary part of the mechanical susceptibility, \( \omega_0 = (k/m)^{1/2} \) is the eigenfrequency, \( k_B \) is the Boltzmann constant, \( T \) is the absolute temperature, \( k \) is the effective spring constant, \( m \) is the effective mass, \( A = k_B T / \pi m \) is the oscillator strength, \( \gamma = \zeta / m \) is the drag coefficient, and \( \zeta \) is the damping. Following the approach of Roters and Johannsmann [33], the power spectral density of the thermal fluctuations around the fundamental vibrational mode is fitted with a Lorentzian with parameters of \( \omega_0, \gamma, \) and \( A \), and effective parameters \( k \) and \( m \) are calculated using fitted values.

As a practical example, the dependencies of the effective stiffness versus the probe-specimen separation (displacement) from the surface in pure water, in a pH 2.9 solution, and in a 55 mM NaCl solution are shown in figure 4.

![Figure 4](image)

**Figure 4.** Trend of the effective stiffness obtained from fitting the thermal noise power spectral density spectra as a function of displacement, i.e., at different separations between the probe and the surface. (Modified from Ulcinas et al. [29]).

ShFM is usually developed as a prototype at laboratory level. Various microscope setups and detection systems have been proposed based on scattered evanescent electromagnetic waves [34], optical devices [24, 31, 35, 36], piezoelectric tuning forks [37], self-actuated piezo-resistive cantilevers [38], and capacitive sensors [39]. Recently, Valdrè and co-workers [40] developed a simple device that enables to perform ShFM with a commercial AFM head, with no modification of the AFM hardware and software, and using commercial available cantilevers as probes.

Exploiting this favourable probe-to-sample configuration the same research group developed a novel system for simultaneous shear forces/morphology and RF impedance measurements of specimen surfaces [41]. A vector network analyser was connected to the vertically oriented cantilever in order to perform measurements of RF-impedance variation at the nanoscale, minimizing the cantilever parasitic capacitance thanks to the vertical configuration of the cantilever with respect to the sample surface. The complex impedance was obtained by the measurement of the reflected wave amplitude and phase and their comparison to those of the incident wave.

The variation of the phase \( \theta \) and modulus \( |Z| \) of the complex impedance \( Z = |Z| e^{i\theta} \) at the probe-to-sample interface were measured in the RF range from 100 kHz to 8.5 GHz, being the impedance variation dependent on the material local properties and on the probe-to-sample distance at the nanoscale.
Preliminary investigations were conducted acquiring RF impedance spectra variation after depositing a micrometre thick synthetic oil film on the atomically flat surface of a pyrolytic graphite substrate (figure 5). The RF impedance modulus, $|Z|$, and phase, $\theta$, signal variation were measured in the frequency range from 100 kHz to 8.5 GHz. A comparison of the spectra acquired on graphite with and without the oil film is shown in figure 5. The measurements were performed with the cantilever apex at forced-contact with the surface (the contact condition which nullifies the free oscillation amplitude). The modulus of the impedance measured for the oil film deposited on graphite reveals higher values at low frequency ($< 100$ MHz) respect to the same measurement on pure graphite in air (figure 5a). The phase signal shows a pure resistive forced-contact condition at low frequency, $< 100$ MHz (figure 5b). A strong dependence of the RF impedance phase $\theta$ from both the probe-to-sample distance and the material at the interface was observed. In addition, with this device is possible to measure the RF transmitted wave through the specimen to image sub-surface through-specimen defects or inhomogeneities with nanoscale resolution.

**Figure 5.** a) RF impedance modulus and b) phase signal variation measured on pyrolytic graphite with synthetic oil film of 1 µm of thickness (dashed line) and without the oil film (solid line). c) Typical ShFM approaching curve in air, showing oscillation amplitude variation as a function of probe-sample distance. d) ShFM approaching curve in oil.
With the same device we have investigated the probe-specimen shear behaviour in different environmental conditions. As an example, we report in figures 5c and d typical ShFM approaching curves in air and oil on graphite, showing the oscillation amplitude variation as a function of probe-sample distance related to different shear forces.

3.2. Nanomechanical properties mapping

A new method developed to measure nanomechanical properties is the peakforce quantitative nanomechanical mapping (PeakForce™ QNM™, Bruker AXS GmbH, Germany). It allows the acquisition of high resolution images and simultaneous measurement of material mechanical properties [42]. The maximum force applied on the tip is the control parameter of the feedback circuit, and can range down to the picoNewton.

The tip to specimen distance is modulated with a frequency of about 2 kHz and the position is measured as a function of time. The sample properties are calculated by transforming the time function curves in separation function ones. From the curve analysis a wide variety of properties can be obtained, such as tip-sample adhesion, energy dissipation, elastic modulus and the maximum deformation on the sample. To determine the elastic modulus the first linear tract of the withdrawal line in the force-distance curve is fitted using the Derjaguin-Muller-Toporov (DMT) model according to the following equation [42]:

\[ F - F_{adh} = \frac{4}{3} E' \sqrt{R(d - d_0)^3} \]

where \( F - F_{adh} \) is the force on the cantilever relative to the adhesion force, \( R \) is the tip radius of curvature, and \( d - d_0 \) is the sample deformation. The DMT contact mechanics model is applied to the withdrawal curve which is not subject to the jump-to-contact and related tip-sample settlements. The reduced modulus \( E' \) is calculated by data fitting and, considering the Poisson ratios of the sample, \( \nu_s \), and of the tip, \( \nu_{tip} \), the elastic modulus \( E_s \) is obtained from \( E' \) by the following relation:

\[ E' = \left[ \frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_{tip}^2}{E_{tip}} \right]^{-1}. \]

By considering the tip elastic modulus \( E_{tip} \gg E_s \), the previous equation can be approximated to:

\[ E' = \left[ \frac{1 - \nu_s^2}{E_s} \right]^{-1}. \]

In order to obtain meaningful Young’s modulus values, care should be taken that the sample is not plastically deformed. The maximum applied force is controlled by the feedback circuit. Generally, a minimum of 2 nm elastic deformation is necessary for accurate measurements, and the sample surface can be subsequently checked for residual plastic deformation. The Poisson’s ratio generally ranges between about 0.2 and 0.5 (perfectly incompressible) giving a difference between the reduced modulus and the sample modulus between 4% and 25%. By PeakForce QNM it is generally possible to determine the elastic modulus in a range from 700 kPa to 70 GPa.

For the sake of example, Schön and co-workers [43] applied PeakForce QNM to investigate segmented polyether polyurethanes with varying stoichiometric ratios of the isocyanate and the hydroxyl groups. The morphology was observed to strongly depend on stoichiometric ratio, providing specific “fingerprint” phase images that allow the identification of the sample. The elastic moduli of
stiff and soft segments of the phase separated polyurethane samples were resolved at nanoscale resolution. While tensile testing provided a bulk average value for the elastic modulus of the elastomers, the AFM-based methodology enabled a quantitative investigation of surface stiffness with nanoscale resolution.

The pulsed force mode (PFM, WITec GmbH, Germany) is a SPM-based methodology that employs a procedure for nanotopographical measurements without shear lateral forces and simultaneous measurement of materials properties such as superficial adhesion, viscoelastic material behaviour and charge distribution [44].

In PFM a function generator modulates the SPM tip to sample distance with a sinusoidal signal of amplitude in the range 10 nm - 500 nm and frequency from 100 Hz to 5 kHz. The amplitude is re-settled after each contact between the tip and the surface during one cycle.

The maximum tip-sample force is the feedback control parameter and it is set by the user to limit the maximum tip-sample repulsive force. To measure adhesive forces the maximum negative (attractive) force value is recorded when the tip retracts from the surface. Elastic properties are calculated by the force trends before and after the point of maximum tip-sample force in one approach-withdrawal cycle. The difference between a force value in the repulsive part and the maximum force is related to the sample local stiffness. Furthermore, long range electrostatic forces due to surface potential distribution can be mapped recording the fluctuations of the zero force line.

For the sake of example, Roffat and co-workers [45] applied PFM to investigate the pH sensitivity of Li_{0.30}La_{0.56}TiO_3 ceramics, which depends on their sintering temperature. These ceramics may be employed as pH sensors and reference electrodes in solid-state combined pH electrode devices. It was observed, by adhesive surface property mapping, that sensitive ceramics have a higher hydrophilic surface properties respect to the insensitive ones. The hydrophilic character of the sensitive ceramics was attributed to surface hydroxyl –OH groups that may display a variable basic character depending on the covalency of their O atoms.

Rezende and co-workers [46] used PFM to evaluate the surface mechanical properties of thin film patterns (less than 100 nm in thickness) obtained by drying an aqueous solution of poly (N-isopropylacrylamide) and sodium dodecyl sulphate on mica. AFM images of dry films showed a morphology formed by droplets distributed on the substrate. PFM adhesion and stiffness mapping revealed heterogeneous features inside the droplets, which were ascribed to be phase separation of the polymer – surfactant complex during the drying/dewetting process. Furthermore, a very thin layer containing polymer and surfactant was observed by a comparison of the adhesion forces measured on dewetted and freshly cleaved mica.

4. Frequency modulation AFM – air, liquid, vacuum
Atomic force microscopy can achieve atomic and also sub-atomic resolution probing short-range chemical forces between a sharp tip and a sample. The tip is positioned with sub-angstrom precision above the surface using piezoelectric actuators.

The most significant recent advancements in AFM high-resolution imaging are based on frequency modulation atomic force microscopy (FM-AFM) [47, 48]. Unlike amplitude-modulation AFM [49], in FM-AFM the cantilever is driven at its resonant frequency. The control signal is the frequency shift of the resonant frequency of the cantilever, which is modified by the tip-surface interaction. The cantilever is kept oscillating at its current resonant frequency, for instance by means of a phase-locked loop (PLL), and the frequency shift induced in the cantilever motion by the tip-sample interaction is used as the source of topographical contrast. In FM-AFM sub-angstrom tip oscillation amplitude enhances the sensitivity to short range forces and it is now possible to directly measure with sub-atomic precision the force of a single chemical bond between two atoms. The image resolution can also be improved by controlling the chemical reactivity of the AFM tip apex picking up adatoms or small inorganic molecules [50].
FM mode, firstly developed for in vacuum experiments, was subsequently applied to ambient conditions to achieve atomic resolution [51].

UHV FM-AFM experiments have involved the chemical identification at the atomic level [52, 53], and also the manipulation of individual atoms on semiconductor surfaces [54].

The research group of Giessibl [55] imaged the silicon (111)-(7×7) surface in ultra-high vacuum resolving fine structures within a single atom. This sub-atomic structure was interpreted as arising from orbitals of the atom at the end of the tip interacting with orbitals of the atom on the surface. It is known that AFM images involve a convolution of tip and sample features, thus comprising sub-atomic information on the electronic structure of the probe.

The extension of FM-AFM to high-resolution operation in liquids has been also achieved. For instance, Fukuma developed a wideband low-noise optical beam deflection sensor with photothermal cantilever excitation system for liquid-environment FM-AFM, applicable to relatively stiff cantilevers (> 20 N/m) [56]. Atomic resolution imaging of mica surface in water was demonstrated (figure 6) by using a stiff cantilever, k = 20 N/m, and an oscillation amplitude of about 0.1 nm. The research group of Garcia [57] combined FM-AFM with bimodal excitation to image and measure protein flexibility with molecular resolution. Mapping of the elastic modulus variation in a single antibody pentamer was achieved in liquid with sub-2 nm spatial resolution.

![Figure 6.](image)

**Figure 6.** a) FM-AFM image of mica in phosphate buffer solution at RT, pH 7.4. b) Atomic-scale model of the cleaved mica surface. (Modified from Fukuma [56]).

5. High-speed AFM

One of the most challenging advances in AFM involves the combination of high spatial resolution capabilities with improved temporal resolution. Sub-second acquisition timescale is required both in materials science, in biology, and also for quick high resolution investigations of large sample areas. At present AFM is generally a slow process (tens of seconds of acquisition times).

High-speed atomic force microscopy (HS-AFM), developed at laboratory level, allows at present the direct observation of dynamic processes at a sub-second to sub-100 ms temporal resolution, with about 2 nm of lateral and about 0.1 nm vertical resolution [58]. AFM imaging speed is limited by the bandwidth and dynamic behaviour of actuators and mechanical parts. The efforts towards an improved temporal resolution are mainly addressed to the development of small cantilevers and suitable optical beam deflection systems, fast amplitude detectors, fast scanners and new feedback controllers [59, 60]. Picco and co-workers [61] presented a HS-AFM working in contact mode with a flexure stage arrangement allowing imaging video rates of 30 fps (frame per second) and an ultra-high speed version exceeding 1000 fps by employing a tuning fork and a flexure-stage scanning combination.
Recent interest is focused on the development of high-bandwidth flexure-guided nanopositioning systems [62], investigation of the stability of force-feedback applied in HS-AFM imaging [63], on the effect of higher eigenmode flexural oscillations of the cantilever on the signal-to-noise ratio in the topography images [64], and on positioning error mapping to address dimensional metrology issues [65].

Other research groups proposed novel scan trajectories for HS-AFM. For instance, Tuma and co-workers [66] demonstrated a real-time AFM imaging with a frame rate of 1 fps, in a custom-built AFM, employing a two-dimensional Lissajous pattern generated by actuating the scanner with two single-tone harmonic waveforms of constant amplitude and frequency. Yong and co-workers [67] proposed a smooth cycloid-like scan pattern allowing a scan speed up to 156.25 Hz implemented on a commercial AFM with minimal modifications.

References
[1] Melitz W, Shen J, Kummel A C and Lee S 2011 Surf. Sci. Rep. 66 1
[2] Antognozzi M, Wotherspoon A, Hayes J M, Miles M J, Szczelkun M D and Valdrè G 2006 Nanotechnology 17 3897
[3] Valdrè G, Antognozzi M, Wotherspoon A and Miles M J 2004 Phil. Mag. Lett. 84 539
[4] Valdrè G 2007 Eur. J. Mineral. 19 309
[5] Valdrè G, Moro D and Ulian G 2011 Micro Nano Lett. 6 922
[6] Valdrè G, Moro D, Hounsome C M and Antognozzi M 2012 Nanotechnology 23 385301
[7] Valdrè G, Moro D and Ulian G 2012 IOP Conf. Ser.: Mater. Sci. Engng. 32 012027
[8] Hudlet S, Saintjean M, Roulet B, Berger J and Guthmann C 1995 J. Appl. Phys. 77 3308
[9] Nyffenegger R M, Penner R M and Schierle R 1997 Appl. Phys. Lett. 71 1878
[10] Girard P 2001 Nanotechnology 12 485
[11] Valdrè G, Moro D, Lee D, Smith C G, Farrer I, Ritchie D A and Green R T 2008 Nanotechnology 19 045304
[12] Valdrè G, Malferrari D and Brigatti M F 2009 Clay Clay Miner. 57 183
[13] Valdrè G, Tosoni S and Moro D 2011 Am. Mineral. 96 1461
[14] Valdrè G and Moro D 2008 Nanotechnology 19 405501
[15] Valdrè G and Moro D 2008 Nanotechnology 19 405502
[16] Sadewasser S, Jelinek P, Fang C-K, Custance O, Yamada Y, Sugimoto Y, Abe M and Morita S 2009 Phys. Rev. Lett. 103 266103
[17] Sadeghi A, Baratoff A, Ghasemi S A, Goedecker S, Glatzel T, Kawai S and Meyer E 2012 Phys. Rev. B 86 075407
[18] Hu J, Xiao X-D, Ogletree D F and Salmeron M 1995 Science 268 267
[19] Hu J, Xiao X-D and Salmeron M 1995 Appl. Phys. Lett. 67 467
[20] Verdaguer A, Cardellach M and Fraxedas J 2008 J. Chem. Phys. 129 174705
[21] Arima K, Jiang P, Lin D-S, Verdaguer A and Salmeron M 2009 J. Phys. Chem. A 113 9715
[22] Xu L and Salmeron M 1998 Langmuir 14 5841
[23] Huber H P, Humer I, Hochleitner H, Fenner M, Moertelmaier M, Rankl C, Imtiaz A, Wallis T M, Tanbakuchi H, Hinterdorfer P, Kabos P, Smoliner J, Kopanski J J and Kienberger F 2012 J. Appl. Phys. 111 014301
[24] Antognozzi M, Haschke H and Miles M J 2000 Rev. Sci. Instrum. 71 1689
[25] Antognozzi M, Humphris A D L and Miles M J 2001 Appl. Phys. Lett. 78 300
[26] Vicari J A, Ulcinas A, Hörber J K H and Antognozzi M 2011 Ultramicroscopy 111 1547
[27] James P J, Antognozzi M, Tamayo J, McMaster T J, Newton J M and Miles M J 2001 Langmuir 17 349
[28] Antognozzi M, Protti A, Miles M J and Valdrè G 2003 GeoActa 2 101
[29] Ulcinas A, Valdrè G, Smitka V, Miles M J, Claesson P M and Antognozzi M 2011 Langmuir 27 10351
[30] Scholz T, Vicary J A, Jeppesen G M, Ulcinas A, Horber J K H and Antognozzi M 2011 *Nanotechnology* **22** 095707

[31] Yang P C, Chen Y and Vaeziravani M 1992 *J. Appl. Phys.* **71** 2499

[32] Betzig E, Finn P L and Weiner J S 1992 *Appl. Phys. Lett.* **60** 2484

[33] Roters A and Johannsmann D J 1996 *J. Phys.: Condens. Matter* **8** 7561

[34] Antognozzi M, Ulcinas A, Picco L, Simpson S H, Heard P J, Szczelkun M D, Brenner B and Miles M J 2008 *Nanotechnology* **19** 384002

[35] Froehlich F F and Milster T D 1994 *Appl. Phys. Lett.* **65** 2254

[36] McMaster T J, Miles M J, Antognozzi M and Humphris A D L 2003 *U.S. Patent US20030160170*

[37] Karrai K and Grober R D 1995 *Appl. Phys. Lett.* **66** 1842

[38] Woszczyna M, Gotszalk T, Zawierucha P, Zielony M, Ivanov T, Ivanova K, Sarov Y, Nikolov N, Mielczarski J, Mieluarska E and Rangelow I W 2009 *Microelectron. Engng.* **86** 1212

[39] Leong J K and Williams C C 1995 *Surf. Sci. Rep.* **23** 197

[40] Sugimoto Y, Namikawa T, Abe M and Morita S 2009 *Nature* **446** 64

[41] Ando T 2013 *Microscopy* **62** 81

[42] Klapetek P, Picco L, Payton O, Yacoout A and Miles M 2013 *Meas. Sci. Technol.* **24** 025006

[43] Tuma T, Lygeros J, Kartik V, Sebastian A and Pantazi A 2012 *Nanotechnology* **23** 185501

[44] Yong Y K, Moheimani S O R and Petersen I R 2010 *Nanotechnology* **21** 365503