Review

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Recent development of PeakForce Tapping mode atomic force microscopy and its applications on nanoscience

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Abstract: Nanoscience is a booming field incorporating some of the most fundamental questions concerning structure, function, and applications. The cutting-edge research in nanoscience requires access to advanced techniques and instrumentation capable of approaching these unanswered questions. Over the past few decades, atomic force microscopy (AFM) has been developed as a powerful platform, which enables in situ characterization of topological structures, local physical properties, and even manipulating samples at nanometer scale. Currently, an imaging mode called PeakForce Tapping (PFT) has attracted more and more attention due to its advantages of nondestructive characterization, high-resolution imaging, and concurrent quantitative property mapping. In this review, the origin, principle, and advantages of PFT on nanoscience are introduced in detail. Three typical applications of this technique, including high-resolution imaging of soft samples in liquid environment, quantitative nanomechanical property mapping, and electrical/electrochemical property measurement will be reviewed comprehensively. The future trends of PFT technique development will be discussed as well.

Keywords: force control; force curve; nanomechanical properties mapping; nanoscience; PeakForce Tapping mode.

1 Introduction

Nanoscience is the study and application of structures and materials at the nanoscale in all science fields, such as physics, materials science, and chemistry. The rapid expansion of this field dramatically fueled the development of new advanced techniques and rigid methodologies to reveal the intrinsic but unknown issues in this field. Since its invention in 1986 by Bing et al., the atomic force microscope (AFM) has been a critical instrument in the development of nanoscience [1]. At the very beginning, contact mode was often applied, and the AFM tip is always in contact with the sample surface during the scanning process [2, 3], thus, inducing a large lateral force, which often causes damages to the tip or the samples. Consequently, soft samples, especially biological samples, are difficult to be characterized. To address this issue, the tapping mode (TM) AFM was proposed [4–6]. In the TM-AFM, the cantilever is driven to oscillate close to its resonant frequency; however, the amplitude of the cantilever changes with the tip-sample interaction force exerted on the tip. This amplitude is always used as the input parameter of the feedback loop to obtain the topographic information of the sample. Meanwhile, the phase of the cantilever oscillation also changes during the scanning process, which reflects some properties of the sample [7–9]. However, the mechanical properties including elasticity, viscoelasticity, and adhesion, all contribute to the phase shift, so the explanation of the phase image remains elusive [10].

Except for TM, the non-contact mode [11] and multiferquency mode [12] were also developed. Considering that the cantilever oscillates close to its resonance
frequency [11–13], all these three modes can be collectively called resonance mode. In the resonance mode, the motion of the cantilever is dynamical and highly nonlinear. In addition, the operator should tune the cantilever to find the resonance peak before imaging and optimize the various parameters including driving frequency, free vibration amplitude, setpoint, and feedback gains during imaging. Furthermore, the change in amplitude, phase, or frequency reflects the average force over multiple periods.

On the contrary, the off-resonance mode, in which the cantilever works at a frequency much lower than its resonant frequency, can be approximated to be a quasi-static process. Up to now, several off-resonance modes have been introduced, including force volume (FV) mode [14–16], digital pulse-force (DFT) mode [17, 18], Hybrid mode [19], QI mode [20], and PeakForce Tapping mode (PFT). In the off-resonance mode, the tip at the end of the cantilever moves toward the sample and then away from the sample. As the tip interacts with the sample, the deflection of the cantilever reflects the interaction force. Through controlling the maximum interaction force (peak force) in each pixel on the sample, the topography of the sample can be obtained. Because of the quasi-static process of the cantilever, the tip-sample interaction force can be measured and controlled precisely in each pixel at real time, and the operation is also simplified without the cantilever tuning process. Most importantly, the force-distance (FD) curve, which reflects the multiple nanomechanical properties of the sample, can be acquired as well. Thus, the off-resonance modes are widely used in biology [21, 22], materials science [23], and other fields recently [24].

The FV mode is the early-developed off-resonance mode [25, 26] and is still used nowadays [14]. However, in the FV mode, a sharp turnaround of the triangular driving signal makes the piezoelectric tube of the scanner oscillate, which can be visible in the image. In order to suppress the resonance of the piezoelectric tube, the vibration frequency of the scanner should be small (i.e. <100 Hz) [24]. Thus, it causes a slow scanning speed, which is usually several hours per frame [20]. Furthermore, when the setpoint (trigger force) is reached, the piezo changes its motion to drag the tip away from the sample; however, at this time, the force higher than the setpoint (overshoot) will be applied to the sample. The overloading causes the difficulty of maintaining low setpoints or imaging forces at higher frequencies. Using a sinusoidal wave as the driving signal, the subsequent pulsed-force mode relieves the low speed and the overloading [27–29]. With no sharp turnaround in the sinusoidal signal, the DFT mode is allowed to drive at significantly higher modulation frequency (about 1.5 kHz), permitting the improvement of the scan speed limitation [28, 30, 31]. However, there are presently still several issues to be tackled in the DFT mode. First, the tip completely detaches from the sample, and then, the tip will freely oscillate above the sample surface. Meanwhile the deflection signal looks like a ringing signal. The trigger force in the DFT should be set to large (a few nanonewtons or more [32]) to distinguish the vertical maximum interaction force signal from the ringing signal. Otherwise, the parasitic motion (i.e. ringing of the tip) of the cantilever will dominate and cause feedback instability. In addition, the DFT AFM needs to wait for the ringing signal to be attenuated to get the baseline of deflection in each cycle, thus, bringing in the limit of modulation frequency.

The PFT mode, introduced by Su et al., effectively solves the above-mentioned problems [33, 34]. Figure 1 illustrates the schematic of the PFT AFM. There are many advantages of the PFT mode. First, similar to the DFT mode, the PFT also uses the sinusoidal wave as the driving signal, but the synchronization algorithm was employed. With the synchronization algorithm, the synchronization window is set at about half of the period to extract the actual vertical maximum interaction force (PeakForce); then, the vertical maximum interaction force signal is distinguished from the ringing signal, and thus, the setpoint can be set to small enough. Second, the background subtraction algorithm is adopted in the PFT mode to eliminate the effect of parasitic deflection signal, which occurs due to environmental damping or rough sample topography [35]. The parasitic deflection signal can be obtained as background signal when the tip lifts up to a certain height (several tens of nanometers) before each scan. Recently, a real-time curve fitting software method was also utilized, which does not require the previous lifting procedure and suits for parasitic deflection signal caused by the interaction forces between the cantilever and rough samples [35]. Finally, the synchronization algorithm separates the tip-sample interaction and non-interaction region effectively. Then, the data obtained from the non-interaction region is averaged to get a horizontal line as the baseline, which efficiently overcomes the impact of the ringing signal on the baseline fluctuations. This allows the PFT mode to start the next cycle without waiting for the ringing to be totally attenuated, thus, enabling a high scanning speed of the AFM. The performance comparison of the PFT with the other modes is summarized in Table 1. It can be seen that the PFT enables quantitative nanomechanical properties of the samples and faster scan rate with the lowest minimum peak force setting as well as the high-resolution mapping of the sample surface.

There were many reviews describing various imaging modes of AFM and their applications in the past two
decades [51–55]. In this review, we will only focus on the state-of-the-art applications and development of the PFT mode. Next, we will discuss the development of the PFT into a powerful tool in three applications. Then, the future development in this technique will also be discussed in detail.

2 High-resolution imaging of soft samples in liquid environment

The AFM was used to image various soft materials, especially delicate biological samples, such as DNA [56], virus [57], proteins [58], and fixed or live cells [59]. However, though a wide range of use, the AFM is not an ideal tool for imaging biological samples because it inevitably exerts vertical and lateral forces on samples that causes soft sample damage while scanning [60]. To avoid the deformations of the biological sample, the force is suggested to be less than 100 pN [55]. When the probe moves in the liquid environment, a hydrodynamic force will be generated and leads to a complex background signal and the distortion of the interaction force and, thus, affecting the image quality [38]. It is reported that the resolution of a live cell in the liquid environment is less than 50 nm [61]. The tapping mode is the most widely used imaging mode; however, it is not easy to operate it in liquid because there is always no single well-defined resonance but, instead, forest of
peaks, and the cantilever resonance is highly damped due to the addition of inertia and damping of the surrounding fluid. On the contrary, the PFT offers a simpler way to operate and also a subtle force control. There are several improvements of the PFT mode to operate in liquid. First, the PFT uses the background subtraction algorithm, extracts the actual peak force in a complex liquid environment, and lets the setpoint value settled to be small enough. Such small imaging force will avoid deformation and damage of the biological samples. Second, commercialized tailored probes (PeakForce QNM-Live Cell), which has a 45-μm-long cantilever with a 17-μm-long tip, can ensure that the cantilever is far from the sample surface and reduces the hydrodynamic force and background signal during scanning [38].

Fakhrullina et al. used the PFT AFM to characterize the Caenorhabditis elegans epicuticle cell at different processes of growth [62]. These epicuticle cells needed to be kept alive in liquid environment. As shown in Figure 2A, the width of the annuli increases with the age growth of the C. elegans (from L1 to L4), but the packing density of the annular rings per area unit decreases. Schillers et al. employed the PFT AFM to characterize the microvilli structure of the Madin-Darby canine kidney cells [38]. In the past, the microvilli structure of the living cells were too fragile to be imaged with the AFM and requires forceless imaging tools, for example, the scanning ion conductance microscope(SICM) [64–68]. As shown in Figure 2B, with a fine trigger force setpoint value of 100–130 pN, the individual microvillus starts to appear as cylindrical

structures. Some artifacts such as the wave structures indicate that there is still deformation and displacement of the microvillus. However, when the setpoint value decrease to 80–100 pN, those artifacts disappear, and the structures seems almost upright cylindrical, as displayed in Figure 2C.

As for the other biological samples in liquid, high-resolution images with the PFT mode are also obtained. As shown in Figure 2D, a sub-10-nm resolution image of the human IgM molecules adsorbed on mica depicts a homogeneous distribution. As shown in Figure 2E, with the fine force control (down to 50 pN) in the PFT mode, high-resolution image of the DNA plasmid molecule can be recognized with the lateral resolution down to several nanometers. The recent application of the PFT mode in soft sample imaging is summarized in Table 2.

### 3 Quantitative nanomechanical measurement

As one kind of the off-resonance mode of the AFM, the PFT mode can also probe biophysical properties through recording a single FD curve when the AFM tip is approached to and retracted from the sample. As illustrated in Figure 3A, the approach FD curves (red line) and retraction FD curves (blue line) can quantify the height,

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**Table 2:** Imaging soft samples with PFT mode in liquid.

<table>
<thead>
<tr>
<th>Category</th>
<th>Sample</th>
<th>Setpoint</th>
<th>Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Living cells</td>
<td>Fibroblasts [69]</td>
<td>150 pN</td>
<td>Pixel size of 15 nm</td>
</tr>
<tr>
<td></td>
<td>Microvilli structure of Madin-Darby</td>
<td>80–100 pN</td>
<td>Pixel size of 26 nm</td>
</tr>
<tr>
<td></td>
<td>canine kidney [38]</td>
<td>80–100 pN</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Breast cancer cells [70]</td>
<td>700 pN–1 nN</td>
<td>20 nm</td>
</tr>
<tr>
<td>Molecular bio-imaging, including DNA, proteins, and membranes in liquid</td>
<td>M immunoglobulin antibodies [63]</td>
<td>300 pN</td>
<td>Sub-10-nm resolution</td>
</tr>
<tr>
<td></td>
<td>DNA plasmid [71]</td>
<td>50 pN</td>
<td>Single-molecule resolution</td>
</tr>
<tr>
<td></td>
<td>DNA and DNA-protein complexes [72]</td>
<td>100 pN</td>
<td>Sub-10-nm resolution</td>
</tr>
</tbody>
</table>

**Figure 3:** (A) Schematic illustration of the FD curve. The red line indicates the process of the tip approaching the sample, while the blue line indicates the process of the tip retracting from the sample. (B) The mechanical properties of the human IgM antibodies obtained using PFT QNM in a liquid environment. (B) Voss A, Dietz C, Stocker A, Stark RW. Quantitative measurement of the mechanical properties of human antibodies with sub-10-nm resolution in a liquid environment. *Nano Res.* 2015, 8, 1987–1996. Reproduced with permission from Ref. [63] by Springer Nature.
surface forces, and mechanical deformation of the sample, or derive its elastic modulus and energy dissipation, and also allow adhesion forces to be measured. The details are provided as follows.

**Deformation:** The maximum deformation can be gained by subtracting the position of the tip where the force is zero from the maximum normal interaction force (peak force point) position along the approach FD curve. This value reflects the deformability property of the sample. Figure 3B shows several property images of immunoglobulin antibodies adsorbed on the mica substrate [63]. It is hard to distinguish the antibodies from the mica substrate from the topography image, but from the deformation image, it is easy to distinguish those two parts and shows that the deformation of the antibodies is much larger than that of the mica substrate under the same applied force.

**Adhesion:** The maximum adhesion force can be gained by extracting the force of the tip as it detaches from the sample (the pull-off point) in the FD curve of the tip away from the sample. Adhesion includes a range of attractive forces such as Van der Waals forces, capillary forces, and electrostatic forces. As observed in Figure 3B, the adhesion of the immunoglobulins is smaller than that of the mica substrate.

**Modulus:** To obtain the Young’s Modulus, the part of the retract FD curve where the sample and tip are in contact is fitted using the contact mechanics models. There are several models that are based on continuum mechanics already developed, including the Hertz model [73], Johnson-Kendall-Roberts (JKR) model [74], and Derjaguin-Muller-Toporov (DMT) model [75]. The Hertz model describes the non-adhesive contact between two elastic spheres. More realistic modes are DMT and JKR models, which both account for adhesion effects. The difference between the two models is that the JKR mode is recommended for a soft sample with high energy of adhesion and a large tip radius. The suitable model can be selected according to a elasticity parameter, which is the ratio of the adhesion to elasticity [76]. With the equations listed in Table 3, the Young’s modulus can be calculated by the FD curve fitting with a knowledge of the tip radius. The maximum adhesive force and deformation can be obtained from the FD curve. Thus, we can calculate the contact radius of the tip and finally get the effective Young’s modulus of the tip sample interface \( E_{tot} \). The relationship \( E_{tot} \) is defined as

\[
E_{tot} = \frac{4}{3} \left( \frac{1-V_{tip}^2}{E_{tip}} + \frac{1-V_{sample}^2}{E_{sample}} \right)^{-1} \tag{1}
\]

where \( V_{tip} \) is the Poisson’s ratio of the tip, \( V_{sample} \) is the Poisson’s ratio of the sample, \( E_{tip} \) is the tip’s elastic modulus, and, \( E_{sample} \) is the sample’s elastic modulus. With the tip modulus \( E_{tip} \) assumed to be infinitely large, and \( V_{sample} \) and \( V_{tip} \) are known, we can calculate the \( E_{sample} \). In addition, for measuring samples quantitatively, the appropriate spring constant \( K \) of the probe should be selected. The \( K \) should neither be too large to get a sufficient deflection signal for providing accurate force measurement, nor too small to achieve enough sample indentation. As shown in Figure 3B, the modulus of the immunoglobulins is much smaller than that of the mica substrate.

**Energy dissipation:** The energy dissipation is calculated by the hysteresis area (yellow area) between the approaching and retracting processes, which characterizes the loss of the energy in one cycle. Usually, the work of adhesion

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**Table 3:** Relevant results predicted by the Hertz, DMT, and JKR contact models [77].

<table>
<thead>
<tr>
<th></th>
<th>Hertz</th>
<th>DMT (Low adhesion; small ( \alpha_{tip} ))</th>
<th>JKR (High adhesion; large ( \alpha_{tip} ))</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Range of use</strong></td>
<td>No adhesion; ( a_{Hertz} &lt; \alpha_{tip} )</td>
<td>Low adhesion; small ( \alpha_{tip} )</td>
<td>High adhesion; large ( \alpha_{tip} )</td>
</tr>
<tr>
<td><strong>Maximum adhesive force</strong> ( F_{ad} )</td>
<td>0</td>
<td>( F_{ad}^{DMT} = 2\pi R_{tip} W_{132} )</td>
<td>( F_{ad}^{JKR} = \frac{3\pi}{2} R_{tip} W_{132} )</td>
</tr>
<tr>
<td><strong>Deformation</strong> ( D )</td>
<td>( D_{Hertz} = \frac{\alpha_{Hertz}^2}{R_{tip}} )</td>
<td>( D_{DMT} = \frac{\alpha_{DMT}^2}{R_{tip}} )</td>
<td>( D_{JKR} = \frac{\alpha_{JKR}^2}{R_{tip}} )</td>
</tr>
<tr>
<td><strong>Contact radius</strong> ( \alpha )</td>
<td>( \alpha_{Hertz} = \left( \frac{R_{tip} F}{E_{tot}} \right)^{\frac{1}{3}} )</td>
<td>( \alpha_{DMT} = \left( \frac{R_{tip} (F + F_{ad}^{DMT})}{E_{tot}} \right)^{\frac{1}{3}} )</td>
<td>( \alpha_{JKR} = \left( \frac{R_{tip} (F + F_{ad}^{JKR})}{E_{tot}} \right)^{\frac{1}{3}} )</td>
</tr>
</tbody>
</table>

\( R_{tip} \): Radius of the tip; \( W_{132} \): work of adhesion; \( F \): applied force (tip-sample interaction force); \( E_{tot} \): effective elastic modulus.
and viscous/plastic deformation contributes to the dissipation. In the FD curve, as shown in Figure 3, the yellow area between the red and blue lines represents the energy dissipation.

Using PFT-QNM AFM, Heu et al. studied the effects of glyphosate on the topography and mechanical properties of HaCaT keratinocytes [78] (Figure 4A). After the cell was treated with glyphosate, the cell membrane becomes flat, and the amount of protrusions decreased as illustrated from the topography image (Figure 4A3), while the cell turns to be stiffer as indicated from the modulus images (Figure 4A3), which explains a defense mechanism of the cell to glyphosate. In addition, Sababi et al. distinguished different components of the paperboard coating by analyzing the material properties using PFT-QNM AFM [42]. As shown in Figure 4B, the paperboard is coated with starch, latex, and clay composition in sequence. The latex particles are packed closely together except in some areas where some of the particles are absent from the starch layer. The clay composition is difficult to be distinguished from the latex part in the topography; however, the modulus image shows the difference due the larger modulus of clay (arrow in Figure 4B). Chen et al. also used the PeakForce QNM to study the topography and mechanical properties of organic matter and clay minerals in the bitumen froth fine solids [79]. From the topography image as shown in Figure 4C1, the height of the two particles seems to be similar. Meanwhile, the adhesion force exhibits a significant difference: the upper particle has a lower adhesion force (10 nN) than the lower particle (75 nN) does, which means that the lower particle (circle region in Figure 4C2) is most likely organic in nature.

4 Electrical and electrochemical property measurement

The PFT can be also combined with the other techniques to characterize more properties of the sample, such as

![Figure 4](image-url)

the electrical properties and electrochemical properties. The application of the PFT mode in electrical and electrochemical property mapping is summarized in Table 4. In addition, this section can be divided into three parts: conductivity measurement, Kelvin probe force microscope (KPFM), and electrochemistry measurement.

Initially, the contact mode was adopted to perform the conductivity measurement of different samples such as biological samples [90], material samples [91, 92], and chemistry samples [93], which is called the conductive AFM (CAFM). However, in the CAFM, the tip is easily contaminated or damaged in the scanning. The TMAFM can protect the tip; however, it needs a current amplifier with a large bandwidth (about dozens of kHz to hundreds of kHz) and high gain setting (up to $10^8$), which is very challenging for the recent technology of electronics. The PFT provides a possible solution, PeakForce Tunneling (PF-TUNA), which allows the tip to intermittently touch the sample to extend the tip service life, but requires a smaller bandwidth (i.e. 10 kHz) of the current amplifier due to the lower modulation frequency. In addition, in the PF-TUNA, the tip oscillates at a fixed voltage toward the sample. The force-distance curve, which is used to extract the topography, modulus, adhesion, and other mechanical properties, is obtained in each tapping cycle with a controlled peak force. At the same time, the peak current (currents at the maximum contact force), contact current (currents averaged over the contact duration), and TUNA current (currents averaged over the whole tapping cycle) are all recorded and used for representing the electrical conductivity of the sample.

With the PF-TUNA, Wang et al. studied the effects of different environmental factors and different components on the conductivity of shale samples [80]. Pyrite and the organic in illite promote the electrical conductivity of a shale sample, while other components such as chlorite, quartz, and carbonate do the opposite. The phenomena that conductivity increases with time were explained by the absorbed water molecules on the surface of illite. In addition, the quantitative nanoproperties of nanostructured hybrid composites (ITO/PEDOT:PSS/ P1:EG-C60) are measured through the PF-TUNA [81]. The increasing current information shows that the add material EG-C60 acts as a receiving material, facilitating the transfer of electrons and improving the conductivity of the sample ITO/PEDOT:PSS/P1. Moreover, the PF-TUNA also obtains the morphology and mechanical properties of the sample while probing the electrical properties [82], as shown in Figure 5A. The adhesion image reveals that the nanoparticles can be divided into two phases due to a different adhesion force, while those produce almost the same current value under applied bias voltages regardless of the sign.

The KPFM is another kind of electrical property-probing tool with a nanometer resolution [94, 95]. The KPFM can be used to map local electrostatic potential at sample surfaces to provide information about work function [96], trapped charges [97], doping level variations [98], and electronic structure [99]. The sensitivity of the KPFM is proportional to the value of the dividing quality factor $Q$ to the spring constant $K$ [100]. Using a cantilever with a large $Q$ and a small $K$ also can improve the KPFM sensitivity, but for the TM, $Q$ cannot be very large, and $k$ cannot be very small. The PFT mode has no $Q$ and $K$ limitation, thus, it has a higher sensitivity than the TM. Apart from this, the probe is scanned twice per line. In the first scan (main scan), the PFT can simultaneously obtain the topography, stiffness, and adhesion properties of the samples. In the second scan (lift scan), the tip lifts at a fixed user-defined distance above the sample surface to avoid mechanical cross-talk caused by the tip-sample interactions, then performs like the conventional frequency modulation KPFM.
to measure the work function difference between the tip and the sample.

Robinson et al. used the PF-KPFM to study the correlation between the layer orientation and graphene layer thickness with surface potential [85]. It is found that the surface potential and G-peak Raman shifts are linearly correlated in different thickness and torsion angle regions, and surface potential increases with the twist.
angle. Therefore, graphene with different thicknesses and twist angles can be distinguished by analyzing the surface potential, as presented in Figure 5B. In addition, Xiao et al. also used the PF-KPFM to distinguish perovskite from the Au region in the surface potential image (as shown in Figure 5C1 and C2), although the topography seems to show that there is no change in those two regions [86]. Similar to the PF-TUNA, the PF-KPFM can also obtain the surface topography and mechanical properties simultaneously. It was verified by the result of the duplex stainless steel, as shown in Figure 5D [87].

Local electrochemistry studies are useful in different areas, including chemical manufacturing [101–103], material development [104–106], energy research, biological systems [107,108], and surface protection. Initially, scanning electrochemical microscopy (SECM) uses ultramicroelectrodes to probe electrochemical processes of the sample area underneath the probe. Classic SECM generally has limitations in poor spatial resolution due to the micrometer size of the probe, convolution between topography and electrochemistry, and inefficient control of the tip position. To address these limitations, AFM based on SECM was developed due to the state-of-the-art reliable batch probe fabrication technology [83]. As shown in Figure 6A, the SECM probe is fully insulated with SiO₂ and other dielectric sub-layers, except that the tip apex is exposed with a Pt coat. Using these probes, it is possible to map the electrical property in a liquid environment with AFM. For instance, Huang et al. used the PF-TUNA to study the conductivity of semiconductor/metal samples in liquid and air environment [83]. It is found that semiconducting/metal junctions exhibit unidirectional conduction characteristics in air, but bidirectional conductivity in liquids. Meanwhile, as shown in Figure 6B, contact current of HOPG in deionized H₂O was measured using PF-SECM.

Similar to the PF-KPFM, the tip performs to scan twice per line in the PF-SECM. In the main scan, the PFT captures most information including topography, mechanics, and electrical conductivity, while electrochemical activities are characterized in the lift scan. As for the electrochemical activity probing, a self-assembled monolayer of CH₃-thiol on the Au substrate is studied. The topography shows that height variations about 1 nm is hardly distinguished. However, a donut-shaped structure is clearly observable by the quantitative adhesion force and current signal obtained through the PF-QNM and PF-SECM, respectively. Nellist et al. also adopted the PF-SECM to characterize high pyrolytic graphite with different environments [84]. Figure 6C shows the characterization of a high pyrolytic graphite sample in 10 mM of [Ru(NH₃)₆]³⁺ and 0.1 M of KCl solution. The topography shows that the surface of the high pyrolytic graphite sample has a circular defect region, which is about 0.4 nm higher than the surrounding region. Meanwhile, the adhesion image shows that the adhesion of the defect area is smaller than the surrounding area. In addition, the electrochemical activity image also shows that the induced current of the tip in the defect area is smaller than that of the surrounding area.

Jiang et al. adopted the PF-SECM to study the electrical and electrochemical properties of Pt/p-Si and Pt/p-Si electrodes [89]. Figure 6D is the PF-SECM image of the Pt particles, which were deposited on a Si substrate doped with p+. The images were obtained using a 700-pN image force in a solution of 10 mM of [Ru(NH₃)₆]³⁺ and 0.1 M of KCl. A low imaging force (700 pN) can minimize the movement of the particles. Some particles were deposited on a substrate to form the topography, while the contact current image shows that the particles in regions 1 and 2 have better conductivity than the particles in regions 3 and 4, and the electrochemical activity current image shows that the particles in regions 1 and 3 have a higher electrochemical activity. Thus, different particles can be distinguished by comparing the contact current and the electrochemical activity current.

5 Development of the PFT technology

A slow imaging speed strongly limits the use of the PFT AFM. However, several developments were introduced in different groups to improve the PFT mode recently. First, the modulation frequency of the scanner has to stay well (1–2 kHz) below the scanner frequency to avoid a distorted motion, which is the main reason for the PFT scan rate limitation. Fantner et al. introduced a fast-drifting mechanism including a two-actuator design and a resonance suppression control to improve the scanning speed [109]. A secondary stack pizeo actuator with higher resonance frequency, which is responsible for the high frequency but small amplitude component of the Z-axis displacement, is placed on top of the long-range standard scanner. Along with the first-order notch filter method, which is applied to suppress both the axial as well as the lateral resonance of the scanner, the authors were able to operate at 31.5-kHz modulation frequency, thus, obtaining large line rates (176 line/s), as shown in Figure 7A. Second, Dokukin et al. came up with a powerful extension of the PFT mode-ringing mode. The ringing mode is based on the ringing signal of deflection, which is usually

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treated as noise and always filtered out in the previous PFT mode, to get more compositional information and faster speed than the PFT mode [110]. Compared with the adhesion image of the PFT (Figure 7B1), the restored adhesion image (Figure 7B2) of the ringing mode is much clearer at the 5-Hz scanning rate. From Figure 3A, the adhesion value in the PFT mode easily produces errors due to the hard identification of the instantaneous pull-off moment. So multiple scanning times are performed for each pixel to reduce the error by averaging the adhesion data. However, the multiple scanning makes the imaging time longer. Different from the PFT mode, the restored

adhesion image of the ringing mode uses the multiple points of the ringing signal to extrapolate several values of the adhesion force and, then, averages these values to decrease the errors. Such ringing mode gets a precise restored adhesion force by only a one-time touch and saves a lot of time.

In addition, some groups focus on the expanding range of the QNM with the PFT mode. For example, Hui Xie's group developed a magnetic drive PFT AFM to broaden the modulus range mapping of the probe [111]. It is recommended that a probe with a spring constant close to the elastic modulus of the sample in the PFT mode should be
chosen. Therefore, the PFT mode is not sufficient to characterize the samples that have elastic modulus values that varied with a range of two or more orders. As presented in Figure 7C, a magnetic bead attached on a soft cantilever is actuated by an alternating magnetic field in the Z direction to drive the cantilever up and down. When scanning a region with a large elastic modulus, the magnetic force of the magnetic beads is increased by adjusting the strength of the magnetic field to drive the tip to produce a sufficiently large effective sample indentation. Thus, using this method, with a soft cantilever, it can measure the elastic modulus ranging up to four orders of magnitude.

6 Summary and outlook

The PFT AFM offers a powerful tool to biology, medicine, materials science, and many other disciplines. It enables high-resolution imaging, fine force control, and concurrent mapping of mechanical, electrical, or other properties. Despite of the wide applications and significant process of the PFT mode in the past years, challenges, including further improvement of imaging speed, reliability of property characterization, and integration with other applications, still remain. Although efforts were made in the improvement of imaging speed, the PFT mode still lags behind the contact mode or TMs. The introduction of faster data acquisition systems, feedback loop probe with a high bandwidth, and advanced control schemes is expected to solve the problem. The PFT offers the ability to record thousands of force curves in each second automatically; however, by evaluating the physical properties from those force curves, it is possible that they will be distorted by artificial factors, such as improper spring constant or uncertain shape of probe, appropriate curve fitting mode, and incorrect calibration. A probe with certain shape and larger wear resistance, and automatic parameter calibration will help solve this problem. Due to the low wear and fine control of PFT, SECM and Scanning Microwave Impedance Microscopy both have already been integrated into PFT. What’s more, there are still other AFM applications such as scanning probe lithography required to be integrated with PFT.

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