# PeakForce Scanning Electrochemical Microscopy with Nanoelectrode Probes

Zhuangqun Huang,<sup>1\*</sup> Peter De Wolf,<sup>1</sup> Rakesh Poddar,<sup>1</sup> Chunzeng Li,<sup>1</sup> Andreas Mark,<sup>2</sup> Michael R. Nellist,<sup>3</sup> Yikai Chen,<sup>4</sup> Jingjing Jiang,<sup>4</sup> Georg Papastavrou,<sup>2</sup> Shannon W. Boettcher,<sup>3</sup> Chengxiang Xiang,<sup>4</sup> and Bruce S. Brunschwig<sup>4</sup>

<sup>1</sup>Bruker Nano Surfaces, 112 Robin Hill Road, Goleta, CA 93117

Abstract: This article describes new batch-fabricated, robust, and easy-to-use scanning electrochemical microscopy (SECM) nanoelectrode probes with a characteristic dimension of about 50 nm. The resulting microscopy method, PeakForce SECM™, provides electrochemical images with sub-100-nm resolution, as well as simultaneously acquired topographical, electrical, and mechanical maps on the nanometer-scale. Using Bruker's existing high-bandwidth electronics, PeakForce SECM also provides the capability for high-quality nanoelectrical imaging in liquid. This article describes several applications of this new PeakForce SECM capability that enable various types of multidisciplinary research.

#### Introduction

Electrochemistry deals with the interplay between electrical energy and chemical energy. Many electrochemistry applications impact daily life, from chemical manufacturing to frontier R&D activities in energy research, biological systems, materials development, and surface protection. Macroscopic electrochemical behavior is an average of the heterogeneous reactivity over an electrode surface. This may include different active sites, crystal-facet-dependent properties, or surface defects. Reactivity variation is a result of the heterogeneity in structural, mechanical, electrical, and/or electrochemical properties over the electrode surface. Driven by the recent nanostructural engineering of electrode materials, in situ and localized techniques are needed to map surface reactivity on the nanoscale [1]. To accommodate today's highly multidisciplinary research, approaches capable of simultaneously capturing correlated microscopic multidimensional information in addition to electrochemistry are needed.

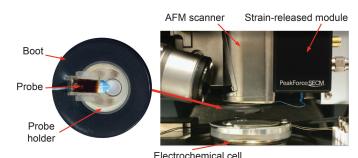
Local electrochemical studies require an electrochemical probe with dimensions smaller than the required spatial resolution, along with stable and precise control of the probe position. Ultramicroelectrodes (UME) were first adopted in scanning electrochemical microscopy (SECM) for spatially resolved measurements. In classic SECM, a UME positioned in close proximity to the sample surface is scanned across the sample. The nature and properties of the sample area underneath the probe perturb the electrochemical processes at the electrode tip. By capturing the electrochemical response of the tip during scanning, the variation of electrochemical properties is imaged. Classic SECM generally has limitations in spatial resolution on the micron scale due to probe size, inefficient control of tip position, the convolution between topography and electrochemistry, and obtainable information only limited to electrochemistry and topography [2]. To address these

limitations, atomic force microscopy (AFM) based SECM has been developed. This is to take advantage of AFM for its sharp cantilevered probe, its high-spatial-resolution imaging capability, and the ability to probe multidimensional material properties beyond electrochemistry on a fine scale. However, it remains an unmet challenge to batch-fabricate stable, reliable, and cost-effective nanoelectrode SECM probes.

Recently we have developed batch-fabricated, high-quality, robust PeakForce SECM probes with a characteristic tip dimension of approximately 50 nm. With these probes, PeakForce SECM provides electrochemical images with sub-100-nm resolution along with simultaneous acquisition of topographical, electrical, and quantitative mechanical maps on the nanometer-scale. In addition, this method also provides unique capabilities for high-quality nanoelectrical imaging in liquid. This article describes PeakForce SECM and provides some illustrative examples of applications.

#### Materials and Methods

SECM probes and application specimens. The PeakForce SECM probes used here are commercially available from Bruker AFM Probes (Camarillo, CA). A patterned silicon nitride on Au electrode was prepared using a conventional MEMS approach. A nanoelectrode array sample (125 nm diameter by 30 nm height) with a 300 nm electrode spacing on a silicon oxide substrate was prepared using electron beam lithographic approach. A sample of self-assembled monolayers of  $CH_3$ -thiols was transferred by micro-contact printing to Au substrates. Hexaammineruthenium(III) chloride ([Ru(NH<sub>3</sub>)<sub>6</sub>]Cl<sub>3</sub>, 98%, Aldrich / ABCR), potassium chloride (BioXtra,  $\geq$ 99.0%, Aldrich),



**Figure 1:** Pictorial illustration of PeakForce SECM accessories with a pre-mounted PeakForce SECM probe loaded.

<sup>&</sup>lt;sup>2</sup>Physical Chemistry, University of Bayreuth, Universitätsstrasse, 95440 Bayreuth, Germany

<sup>&</sup>lt;sup>3</sup>Department of Chemistry and Biochemistry, 1253 University of Oregon, Eugene, OR 97403

<sup>&</sup>lt;sup>4</sup>Joint Center for Artificial Photosynthesis, California Institute of Technology, Pasadena, CA 91106

<sup>\*</sup>teddy.huang@bruker.com



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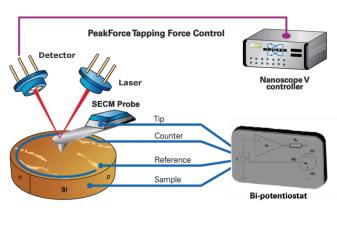












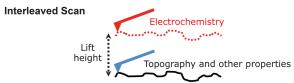


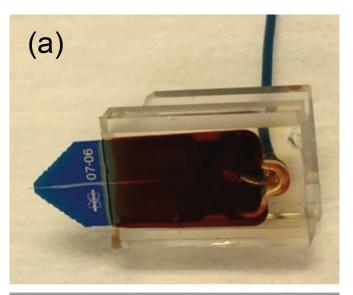
Figure 2: Schematic illustration of a PeakForce SECM system showing the major mechanical and electronic components. The interleaved scanning mode is also illustrated (below).

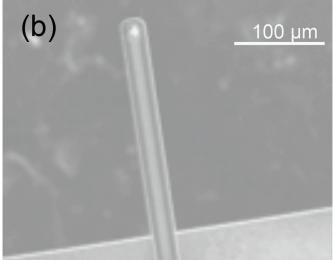
and potassium nitrate (PURATREM, 99.999%, ABCR) were used as electrolyte.

**SECM probe testing.** Prior to the PeakForce SECM measurement, all probes were tested by performing a few cyclic voltammograms (CV, 20 mV/s, 0 to -0.5 V vs. Ag/AgCl) in a standard three-electrode electrochemical cell with a Pt counter electrode and a standard Ag/AgCl (3M NaCl) reference electrode (CH Instruments, Inc.). The solution was 5 mM [Ru(NH<sub>3</sub>)<sub>6</sub>]Cl<sub>3</sub> with 0.1 M KNO<sub>3</sub>. A bi-potentiostat (CHI760D, CH Instrument) was used. In addition to this regular test, 3 probes from each batch of about 80 were randomly selected for three-cycle stability tests. Each cycle includes, sequentially, 100 CV scans, rinse and dry, and 3 hours of amperometric measurement (-0.4 V vs. Ag/AgCl).

SECM setup. All the PeakForce SECM measurements were performed on Bruker Dimension Icon® and FastScan® AFMs equipped with standard PeakForce SECM accessories. These include a PeakForce SECM probe holder, a protective Kel-Rez boot, and a strain-release module with resistance selector (0, 1, and 10 M $\Omega$ ) to limit the maximum current flow, as shown in Figure 1. The module avoids direct electrical connection to the probe, which generally causes mechanical noises in the AFM images. Unless otherwise noted, a 10 M $\Omega$ current-limiting resistor was selected. Although this 10 M $\Omega$ resistor limited the current to generally <100 nA, the SECM probe can handle current of >1 µA in PeakForce Tapping® mode, and >600 nA when in constant contact with a conductive substrate. Other accessories are similar to the Bruker standard ECAFM kit, except for a modified glass cover, a modified chuck plate, and a low-noise cable.

SECM measurements. PeakForce SECM integrates PeakForce QNM® (quantitative nanomechanics) mode with AFM-SECM functions, as shown in Figure 2. Both the probe and the sample are working electrodes sharing the same reference and counter electrodes. The probe and the





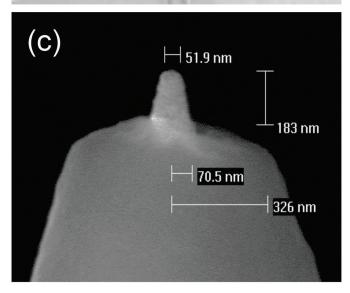
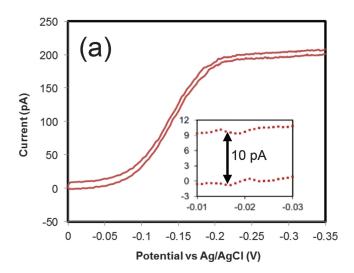
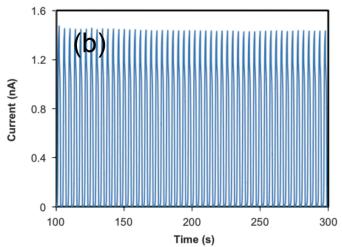


Figure 3: (a) A pre-mounted SECM nanoelectrode probe with the cantilever on the left. (b) SEM image of the probe cantilever showing the Pt conductive path with a width of 15  $\mu$ m. (c) An exposed Pt-coated tip apex with ~50 nm end-tip diameter and ~200 nm tip height (other than the tip apex, the probe is fully insulated with SiO<sub>2</sub> and other dielectric sub-layers).

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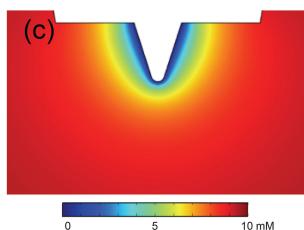


Figure 4: (a) A typical sigmoidal CV in 2.5 mM  $[Ru(NH_3)_6]^{3+}$  and 0.1 M KNO<sub>3</sub> at a scan rate of 20 mV/s (the inset picture shows negligible capacitive and leakage currents at this scan rate). (b) 50 CVs plotted into a current-time fashion in 10 mM  $[Ru(NH_3)_6]^{3+}$  and 0.1 M KCl at a scan rate of 300 mV/s. (c) COMSOL simulation shows a highly localized diffusion layer with a thickness of ~100 nm in 10 mM  $[Ru(NH_3)_6]^{3+}$  solution.

sample are generally biased at different potentials, relative to the reference electrode, to enable different chemical reactions. In this work, the probe reduces the [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> to [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>2+</sup> at -350 to -500 mV versus a pseudo Ag/AgCl

reference electrode, while the sample is biased at 0 to -100 mV for [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> regeneration. For imaging, the PeakForce SECM scan was performed using an interleaved scan mode with a lift height typically 40 to 150 nm (Figure 2). On each line scan during the main scan, the probe scans over the sample surface using the normal PeakForce QNM mode. During the PeakForce QNM scan, both the topological data and the current at contact are captured from the voltage difference between the probe and the sample. This averaged contact current is processed into an AFM conductivity map in real time by NanoScope® software. In this scenario, the contact current signal is limited by low bandwidth and electronic delays, particularly at the sensitivity of nA/V. The topographic line profile from the PeakForce Tapping main scan line is stored for use in the lift scan (Figure 2). During the lift scan, the probe follows the stored topographic profile from the previous scan line maintaining a constant tip-sample distance defined by the lift height. While the probe is lifted, electrochemical information is captured.

Charge transport calculations. The engineering physics model set up in this work solves for charge transport and conservation in the liquid electrolyte as well as the kinetics of the electrochemical reaction. Finite-element solver, COMSOL Multiphysics, has been applied to solve the coupled governing equations and the corresponding boundary conditions. Quadratic element discretization and standard solvers were chosen. A relative tolerance of the corresponding variable of  $10^{-3}$  was used as convergence criteria [3].

SECM probe operation. The premounted PeakForce SECM probe shown in Figure 3a is encapsulated in glass sealed with a chemical-resistant epoxy compatible with a wide range of electrochemical environments. The probe encapsulation was designed to have a relatively large size for ease of handling. A 15  $\mu$ m width Pt conductive path runs between the tip, and the base is embedded for electrical conductivity (Figure 3b). Figure 3c shows the tip whose apex has a Pt coated area of ~50 nm in diameter and with ~200 nm tip height. Other than this electrochemically active region, the probe is fully insulated by mainly a coating of SiO<sub>2</sub>.

Figure 4a shows the CV curve of this nanoelectrode, which has a sigmoid shape and a diffusion-limited current of ~200 pA, in 2.5 mM [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> and 0.1 M KNO<sub>3</sub> consistent with the COMSOL simulation (220 pA). The inset in Figure 4a demonstrates negligible leakage current and ramp-rate dependent capacitive charging currents. A ~10 pA current at 20 mV/s indicates a compact and pinhole-free dielectric coating. Figure 4b shows an example of 50 CVs plotted as a currenttime profile measured in  $10\,\text{mM}$  [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> and  $0.1\,\text{M}$  KCl at a scan rate of 300 mV/s. It shows a constantly repeatable CV scan on each cycle. Figure 4c is taken from the COMSOL simulation of the [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> concentration profile, when the tip is biased for  $[Ru(NH_3)_6]^{3+}$  reduction, with a bulk concentration of 10 mM. This profile shows expected convergent hemispherical radial diffusion for a typical nanoelectrode, which results in the sigmoidal response in the CV scan (Figure 4A). At the electrode surface, the [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> is fully depleted because of the facile interfacial charge transfer process, while at ~50 nm from the electrode surface, the concentration of Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> has recovered by over 60%. This highly localized diffusion layer is critical for high spatial resolution of electrochemical imaging.

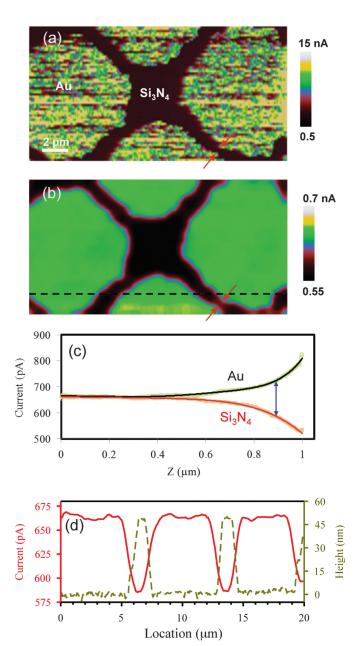
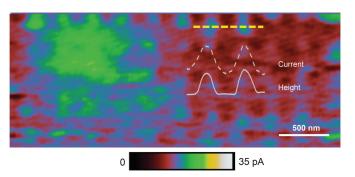


Figure 5: PeakForce SECM measurement of an electrode with a 50 nm thick patterned silicon nitride layer deposited on an Au substrate. (a) Map of current response from the PeakForce Tapping scan. Regions of exposed Au substrate and the nitride layer are labeled. A 500 nm feature is marked by the red arrows. (b) Electrochemical current map at a lift height of 100 nm. The dashed line indicates the location for cross-sectional analysis. (c) Approach curves on Au (green dots) and nitride (yellow dots) regions plotted with respect to the probe movement, respectively. The solid lines are for visible guidance and have no physical meaning. The blue arrow shows a ~100 pA current difference when the tip is ~100 nm away from the sample surface. (d) Line profiles of tip current during the lift scan (solid red: left y axis) and the surface topography (dashed green: right y axis). Solution, 10 mM [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> and 0.1 M KCI.

### Results

 $Si_3Ni_4$  on Au. A sample with a 50 nm thick patterned silicon nitride layer deposited onto an Au substrate was tested using PeakForce SECM. The tip voltage was -0.4 V vs. Ag/AgCl for reducing  $[Ru(NH_3)_6]^{3+}$  while the sample potential was -0.1 V for the regeneration of  $[Ru(NH_3)_6]^{3+}$ . As shown in Figure 5a, when the tip is in contact with the Au surface during



**Figure 6:** High-resolution electrochemical image of a nanoelectrode array sample, 125 nm (diameter) × 300 nm (period). The yellow dashed line indicates the scan line for cross-section analysis. The line profiles of current and topography are the dashed and solid white curves with full scales of 8 pA and 30 nm, respectively. A lift height of 50 nm was used with a solution of 10 mM [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> and 0.1M KCI.

the PeakForce Tapping cycle, the 300 mV difference between the tip and the sample produces a current when the tip and sample are in contact that is much larger than the noncontact electrochemical current. During the lift scan, the electrochemical current clearly differentiates the Au and nitride regions, and it resolves sub-micron features, as shown in Figure 5b. Approach curves, or current versus tip-sample distance plots, were captured on both the Au and nitride regions and compared in Figure 5c. These two curves are plotted with respect to the probe movement, Z, as it approached the sample surface. Higher Z values indicate a smaller tip-sample distance. The maximum force during the ramping was 5 nN. With a 1.5 N/m spring constant, the Z movement for cantilever bending to achieve this force is only a few nanometers. Including the jump-in-contact movement due to surface attraction, the probe is in contact with the sample surface only for about the last 10 nm, which is shown by the abrupt increase in tip current on the Au curve at Z ~990 nm in Figure 5c. When the probe approaches the nitride surface, the diffusion of [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> toward the probe is increasingly blocked, resulting in current reduction. Conversely, for the Au surface, the regeneration of  $[Ru(NH_3)_6]^{3+}$  by the Au on the surface increases the current, outperforming the blocking effect. When the tip is ~100 nm away from the sample surface, the current difference between the nitride and the Au is ~100 pA, corresponding to the current contrast in Figure 5b. To correlate the electrochemical activity with the surface topography, we compare the line profiles at the same location for the topographical height and the electrochemical current in Figure 5d. The variation in the electrochemical current tracks the surface features with a sub-micron transition. The current over the nitride is ~90 pA lower than that on the Au surface, although the nitride surface is topographically 50 nm higher than the Au substrate. This indicates that the electrochemical information is well decoupled from the topographic features. The ~90-pA difference is also consistent with that shown on the approach curves in Figure 5c.

**Au nanodot array.** To illustrate the sub-100-nm spatial resolution for electrochemical mapping, we used an Au nanodot electrode array sample with 125 nm (diameter) Au dots spaced

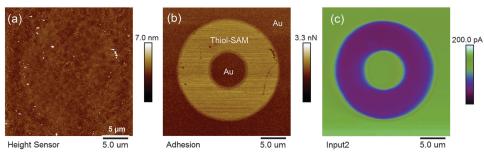


Figure 7: PeakForce SECM images of micro-contact printed CH<sub>3</sub>-thiol self-assembled monolayer (SAM) on an Au substrate: (a) topography, (b) adhesion, and (c) electrochemical activity. Solution, 5 mM [Ru(NH<sub>3</sub>)<sub>6</sub>]<sup>3+</sup> and 0.1 M KNO<sub>2</sub>.

with a 300 nm period. Figure 6 shows the high-resolution electrochemical image from the PeakForce SECM measurement. This sample shows inhomogeneous electrochemical reactivity across the surface, as some regions are highly active while others have minimal response. This may result from the imperfection of sample fabrication. Along the yellow dotted line in Figure 6, individual nanoelectrodes are resolved by the electrochemical response. The cross section analysis shown on the image confirms that the current profile follows the surface topography. The averaged half width at half maximum for the two current peaks is 70 nm, demonstrating sub-100-nm spatial resolution for electrochemical imaging from PeakForce SECM.

Self-assembled monolayers printed on Au. We also investigated an ultra-flat gold electrode chemically patterned by micro-contact printing of CH<sub>3</sub>-thiol self-assembled monolayers (SAMs). This type of sample is generally used as a platform for biological sensors, electrochemical coating, nanoelectronics, microelectromechanical systems (MEMS), and everyday household goods. The topography in Figure 7a shows height variations about 1 nm, which rules out topographic convolution on other imaged properties. However, the donut-shaped structure is clearly observable by the quantitative adhesion force signal obtained through PeakForce QNM, which is part of the functions in PeakForce SECM (compared with Figure 7b). Figure 7b demonstrates ~1 nN difference in adhesion forces between the tip-SAM and tip-Au interactions. In addition, the thiol layer acts as a non-ideal insulator that leads to a reduced interfacial charge transfer, and hence decreased

electrochemical current. Figure 7c illustrates this behavior: on the thiol SAM layer the tip current is reduced by 110 pA compared to that on the bare Au-electrode. For this sample, PeakForce SECM simultaneously captured surface topographic and quantitative mechanical, and electrochemical information in 5 mM  $[Ru(NH_3)_6]^{3+}$  and 0.1 M KNO<sub>3</sub> solution.

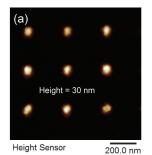
Nanoelectricity in liquid. The PeakForce SECM probe with the same electronics used for PeakForce-

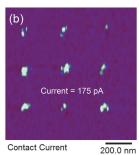
based electrical measurements in air can also be used for nanoelectrical studies in liquid. PeakForce tunneling AFM (PeakForce TUNA<sup>TM</sup>) employs high-bandwidth (15 kHz) electronics to reduce electronic delays for fast data acquisition and has a low noise current amplification sensor close to the tip to reduce stray capacitance. Figure 8 is an application example of PeakForce TUNA using a PeakForce SECM probe in deionized H<sub>2</sub>O. The sample is an Au nanoelectrode array on a semiconductor substrate, as shown in Figure 8a. When a +0.3 V bias is applied, nanoelectrodes have current responses clearly different from the surrounding oxide. To better understand the junction behaviors and sources of the current signals, we compared the I-V characteristics of these nanoelectrodes to the oxide areas, as shown in Figure 8c. When the tip is landed on an oxide region in liquid, only background capacitive charging current (gray curves) was detected at this 400 mV/s ramping rate. While on the Au dot, the I-V curves show non-linear behaviors deviating from either an ohmic contact or a diode junction (green-red curves). Interestingly, the junction behavior in liquid is markedly different from that in air where it shows typical rectifying characteristics to indicate a diode junction (blue curves). This example illustrates the influence of liquid on the interfacial energetics and is related to catalysis in various energy conversion devices.

# Discussion

Previous to this work, AFM-SECM generally relied on either traditional contact or tapping mode scanning. Contact mode requires high imaging forces and is not suitable for

use with soft or fragile samples. Furthermore, in contact mode the constant electrical short between the tip and the sample increases the risk of tip damage. Tapping mode relies on the inherent mechanical resonance of the probe cantilever and is often unstable in liquid. These issues are addressed by Bruker's PeakForce Tapping mode, where the probe is modulated sinusoidally at an offresonance, low frequency (about 1 to 2 kHz) with an amplitude of approximately 100 nm. The feedback signal is the maximum force between the





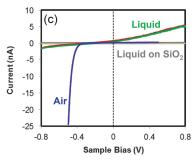


Figure 8: High-resolution PeakForce TUNA in de-ionized  $H_2O$  measurement for conductivity mapping of an Au nanoelectrode array sample: (a) topography with particle height of 30 nm, (b) contact current map at +0.3 V with current on the Au nanoelectrode of 175 pA, and (c) comparison of I-V characteristics of the semiconductor/metal junction in air (blue) and liquid (green-red). The background current in liquid is captured by landing the probe at the oxide region (gray). Voltage ramp rates of 400 mV/s were employed.

tip and the sample. Thus, PeakForce Tapping performs a triggered force curve at every tapping cycle and allows the quantitative imaging of mechanical properties at a normal AFM scan rate. This is known as the PeakForce quantitative nanomechanical (PeakForce QNM) mode. In addition, the sinusoidal modulation results in near zero velocity of the tip when approaching the surface, such that PeakForce Tapping allows direct control for stable, ultralow imaging forces (for example, <50 pN) and automatic image optimization, regardless of the surrounding medium (air, liquid, vacuum, or with varied temperature). PeakForce Tapping also enables AFM electrical and spectroscopic modes for samples that were previously impossible to analyze in this manner [4-6]. Recently, PeakForce QNM has been integrated with AFM-SECM on a Bruker BioScope Catalyst™ AFM, where the probes had a 500 nm to 800 nm long thorn-shaped tip surrounded by a frame electrode approximately 1 µm in diameter [7]. Instead of this microelectrode, in this work we used our developed nanoelectrode to achieve sub-100-nm resolution electrochemical information (Figure 6) while for the first time simultaneously mapping topographic, conductivity (Figure 5), and quantitative nanomechanic properties (Figure 7).

The SECM probe allows for electrical measurement in liquids (Figure 8) largely because of the design of the tip (Figure 3). The tip is fully insulated except for its apex. This greatly reduces stray capacitance because of the small electrically exposed area and the small stray current associated with electrochemical reactions from chemical impurities. In addition, in PeakForce Tapping, the tip intermittently contacts the surface for ~100 µs so that electronics with a bandwidth of 10 to 20 kHz is fast enough to capture signals during the tip-sample contact. PeakForce TUNA, unlike the conductivity measurement by the bi-potentiostat in PeakForce SECM, combines a high bandwidth of 15 kHz with a low-noise current amplifier employing advanced software features that permit sensitive nanoelectrical imaging in liquid with pA resolution while simultaneously providing correlated PeakForce QNM maps. The use of PeakForce TUNA with the SECM probe allows one to measure ultra-low contact currents, as shown in Figure 8.

#### Conclusion

This article describes batch-fabricated, robust, and easy-to-use PeakForce SECM probes with an exposed Pt-coated tip apex having a ~200 nm height and an end tip diameter of ~50 nm. Special coating techniques eliminate pinholes resulting in negligible leakage and capacitive currents. These probes were adopted for the recently developed PeakForce SECM mode, which integrates PeakForce QNM and conductive AFM with AFM-SECM measurements. In this mode topographical, electrical, and mechanical maps can be produced at nanometer-scale and electrochemical images can be produced at sub-100-nm resolution. PeakForce SECM also provides capabilities for high-quality nanoelectrical imaging in liquid environments. All these features were developed to meet the needs of today's highly multidisciplinary research fields, as illustrated by the application examples shown.

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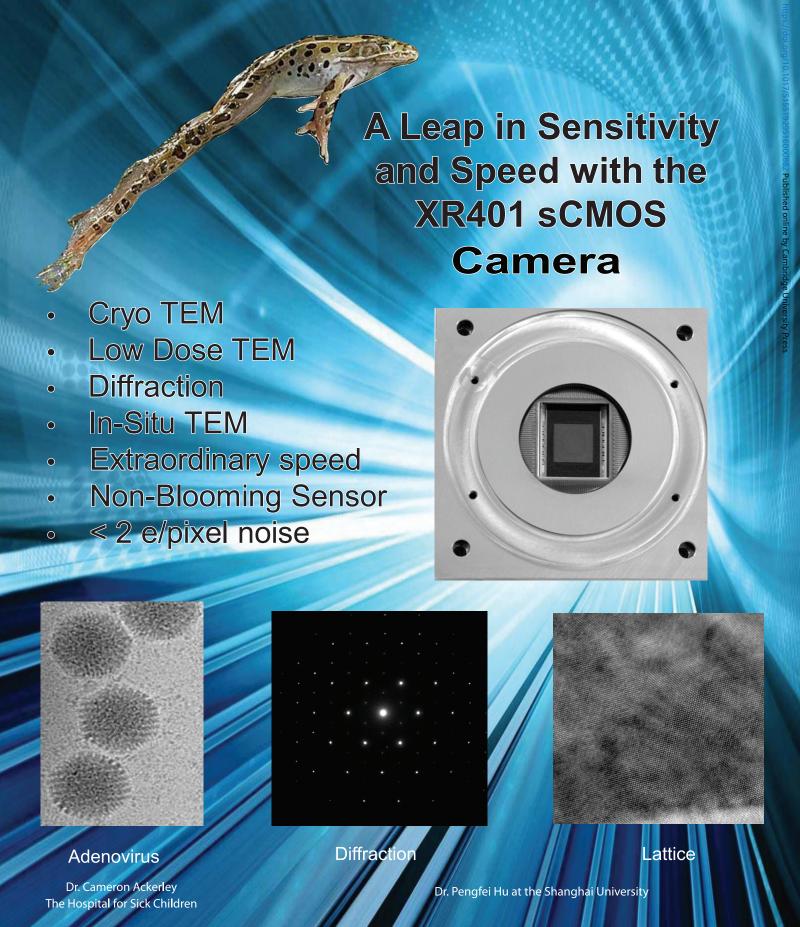


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