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Greetings!

NanoScience’s impact on the world of science and engineering has had an enormous influence on future technologies. With the global technology market expected to exceed $125 Billion by 2024, nanosized particles are changing virtually every sector of our society.

In this issue of NanoScientific, we have two articles from presenters at the NanoScientific Symposium in Europe. “Insights into Submicron Scales - Nanobubbles and Their Effect on AFM Measurements and Engineering Processes” by Lisa Ditscherlein, Technical University Freiberg and an interview with Dr. John A Marohn, Professor & Director of Undergraduate Studies, Department of Chemistry and Chemical Biology, Cornell University on “Advances in Electric Field of Materials Science & Engineering”.

We also have a feature interview with Dr. Elliot Bloom from Stanford about the search for dark matter and other wonders of the universe for the last ten years using the Large Area Telescope on the Fermi Gamma-ray space observatory at Stanford.

Keibock Lee Editor-in-Chief

The NanoScientific Symposium on SPM will be held Sept. 19-20 at the Park Nanoscience Center at SUNY Poly’s Albany NanoTech Complex, a fully-integrated research, development, prototyping, and educational facility and home to the College of Nanoscale Sciences and the College of Nanoscale Engineering and Technology Innovation.

Park Systems, Sponsor of NanoScientific Symposium on SPM at SUNY Polytechnic is offering Complimentary Registration thru Sept. 10.

We hope you can join us at one of these or future upcoming NanoScientific Symposia and as always, send us your feedback and story ideas.

Keibock Lee Editor in Chief
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“As SUNY Polytechnic Institute provides cutting-edge educational and research and development opportunities, it is exciting that Park Systems established operations at our Albany campus,” said Dr. Alain Diebold, SUNY Poly Interim Dean of the College of Nanoscale Sciences, Empire Innovation Professor of Nanoscale Science; and Executive Director, Center for Nanoscale Metrology. “Our scientists and engineers look forward to working closely with Park Systems to enhance next generation technologies that will lead to improved metrology capabilities for researchers and members of industry around the world.”

The special session on nanobubbles will cover the influence of nanobubbles in engineering processes like melt filtration (CRC 920, a flagship project of TU Freiberg) and flotation (SPP2045, TU Freiberg and Helmholtz Institute Freiberg for Resource Technology). The special sessions on nanobubbles will cover the influence of nanobubbles in engineering processes like melt filtration (CRC 920, a flagship project of TU Freiberg) and flotation (SPP2045, TU Freiberg and Helmholtz Institute Freiberg for Resource Technology).

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INSIGHTS INTO SUBMICRON-SCALES
NANOBUBBLES AND THEIR EFFECT ON AFM MEASUREMENTS AND ENGINEERING PROCESSES

Since the invention of devices like the surface force apparatus in 1969[1] and the atomic force microscope (AFM) in 1986[2], surface forces have been studied extensively by many scientists[3] as properties of the interfacial region are of great interest for a variety of engineering processes. Interactions between two surfaces are often quantified by classical DLVO-theory, but this theory fails if two hydrophobic surfaces approach each other in an aqueous solution. Other non-DLVO forces come into play that are of different nature and enhance attractive interactions[4]. The origin of such “hydrophobic forces” is still unknown and it is still not clear if it should be considered as a solvation force or some kind of long-ranged electrostatic or van der Waals interaction. Currently “hydrophobic forces” are considered to consist of two parts: a short-ranged true hydrophobic and a long-ranged force, induced by capillary interactions. In 1994, Parker et al. were the first who proclaimed that small bridging bubbles are responsible for the long-ranged, large interaction between hydrophobic surfaces[5]. They observed steps in force-distance curves and interpreted them as coalescing nanobubbles. Some years later, in 2000, Liu et al. and Ishida et al. developed independently a reproducible method to generate nanobubbles on surfaces. Since then, many investigations were done on nanobubble generation[6], stability[7], size and geometry[10-13], detection methods[14], sample roughness[15] and also their impact on engineering processes and other applications.

In most publications, nanobubbles are generated by the so-called solvent exchange method: Here, a liquid (in most cases ethanol) generated by the so-called solvent exchange method: Here, a liquid (in most cases ethanol) stays pinned on the three phase contact line, the Laplace pressure diverges to zero and no large inner pressure or concentration gradient between nanobubble and surrounding environment occurs. Pinning takes place due to chemical and morphological inhomogeneities, even at very smooth and clean surfaces.

With phase contrast imaging it is possible to distinguish between nanobubbles and contaminations.

In the past years, most nanobubble investigations were done using atomic force microscopy because it is a powerful tool to analyse microscopic surfaces. In principle, one can use AFM to scan the surface for bubbles directly via different scanning modes[25, 26] or indirectly via force spectroscopy[27]. Contact mode is unsuitable due to the lateral force applied to the nanobubbles which lets them coalesce or even detach from the surface. This mode has only been used to prove the soft character of the cap-shaped domes[27]. Commonly, intermittent “tapping” mode is applied to detect the bubbles since lateral forces are minimized as a consequence of cantilever oscillation. Amplitude ratio (set point amplitude to free amplitude) should lie above 0.7 as otherwise deformation of the bubbles becomes strong[28, 29] and distorts the result. Often, phase contrast imaging is done simultaneously to facilitate the distinction between bubble and surface. A more seldom-used method is frequency modulation true non-contact mode. Here, the cantilever oscillates at its resonant frequency close to the nanobubble-covered sample with a small amplitude without touching it. This modus, deformation of nanobubbles is minimized. In recent years, the PeakForce® mode has been used for nanobubble detection as well. This mode works at frequencies below resonance and tracks the maximum force load on the tip in real time—which is advantageous compared to tapping mode, where it is difficult to quantify this force. An influencing factor is the chosen imaging force: the larger the force, the higher the deformation of the bubbles. In order to detect nanobubbles it is obvious that a soft and sharp cantilever is needed due to the soft matter character of the bubbles. Normally, cantilevers with spring constants between 0.2 and 4.8 N/m are used. It is to say that, besides AFM mode, the cantilever tip influences the scanning results[26] so that a recalibration of the geometrical size is necessary, if possible. Although the AFM provides three-dimensional information there are some drawbacks: a perturbation of the nanobubbles by the tip is inevitable. Another problem is the inability of the AFM to distinguish between gaseous domains and contamination. It is important to work under clean conditions, e.g. using only glass syringes and clean cantilevers[31] Therefore, alternative observation techniques are utilized like rapid cryofixation[32], infrared spectroscopy to determine the presence of nanobubbles[33], interference-enhanced reflection microscopy[34] to study bubble growth, attenuated total internal reflection microscopy in combination with other spectroscopic methods that investigate the formation of nanobubbles[35], or confocal microscopy to name but a few.

Since then, many investigations were done on nanobubble generation[6], stability[7], size and geometry[10-13], detection methods[14], sample roughness[15] and also their impact on engineering processes and other applications.

In most publications, nanobubbles are generated by the so-called solvent exchange method: Here, a liquid (in most cases ethanol) stays pinned on the three phase contact line, the Laplace pressure diverges to zero and no large inner pressure or concentration gradient between nanobubble and surrounding environment occurs. Pinning takes place due to chemical and morphological inhomogeneities, even at very smooth and clean surfaces.
Interestingly, nearly all examined surfaces like silicon, Si wafers, mica, HDPE or PP are relatively smooth with rms < 5 nm. Such samples are quite interesting in order to acquire fundamental understanding about the formation and stability of nanobubbles as well as the impact of the chosen measurement device on the results. In nearly all real world engineering processes one has to deal with technical rough (rms ≈ 100 nm) and often inhomogeneous surfaces like crushed particles, sintered or abraded surfaces. Measurement on rough surfaces is more complicated, since peaks and valleys influence results if parameters are improperly set and it is more time consuming. For such surfaces there is a need of statistically valid results because of the highly variable interaction area that changes from point to point. This can only be accomplished by a comprehensive sampling and a high number of measurements. Consequently, for force distance spectroscopy hundreds or thousands of curves and an evaluation routine becomes necessary. Previous works on rough surfaces show the importance of wettability and roughness on nanobubble count, stability and therefore adhesive interactions (36, 38) whilst the probability of capillary interactions is low on hydrophobic surfaces (B ≈ 80°), it is more than doubled on hydrophilic samples (B ≈ 104°). This leads to much stronger adhesion between the two surfaces. Contrary to results for hydrophobic systems, adhesion is increased with higher roughness. The explanation for this behaviour is a larger amount of bubbles nested in a stable position inside the pores and asperities. On the other hand, access for the second interacting surface to the nanobubble has to be ensured as otherwise no capillary bridge is formed. Another, very interesting aspect seen on slightly rough, but also on technical rough surfaces, is the increased stability of nanobubbles. Only small contact forces (below 5 N) are necessary to move bubbles on smooth surfaces. In contrast, on rough surfaces they are only deformed but keep pinned to the asperities and pores. Contact forces tenfold stronger are needed to move them.

Engineering processes like flotation, agglomeration or filtration benefit from the microscopic findings about nanobubbles and can be optimized. For mineral flotation, an engineering process that separates hydrophobic from hydrophilic materials, the coexistence of nanobubbles promotes agglomeration between hydrophobic particles due to capillary interactions between them, a phenomenon that holds true for fine as well as coarse particles (44). In (45), an enhancement of agglomeration can be seen in agglomeration size and particle-particle interactions. Furthermore, agglomeration that contain nanobubbles are more stable at higher shear rates (46). In deep bed filtration experiments, an improvement of filtration efficiency can be observed, too. Here, nanobubbles are sitting on poorly wetted foam filters and keep inclusion particles trapped (37). The amount of nanobubbles determines the degree of efficiency. These results are not only interesting for conventional deep bed filtration processes but also for the purification of metal melts (47, 48).

There are many more promising fields for the application of nanobubbles like wastewater treatment (49), micro- and nanofluidics (50) or ore processing (51). The research topic “nanobubbles” provides interesting opportunities in the next few years. It remains exciting.
Force measurements have attracted great attention due to the needs for high resolution mechanical characterization of many newly-developed materials. Lateral force microscopy (LFM), which is particularly useful for identifying and mapping heterogeneities in surface frictional characteristics, has found a steadily increasing number of applications, such as identification of different components within polymer blends and composites, mechanical testing of micro- and nanoelectrochemical systems (MEMS/NEMS) and delineation of surface coverages of deposited materials on thin films.2-7 Herein, we demonstrate the utility of LFM with Park atomic force microscopy (AFM) to identify surface compositional differences of two samples. First, as a proof-of-concept experiment, a sample that contained a polymer deposited onto a glass substrate was imaged with LFM. A clear difference in frictional characteristics was seen from the LFM measurements, albeit the two different frictional domains seen in the LFM images were obscured in the sole topography image. Next, graphene grown on Si was examined with LFM and scanning thermal microscopy (SThM) to examine both the frictional as well as the thermal properties of the sample. Interested readers can refer to Park AFM webpage on SThM for more detailed information regarding SThM mode by Park AFM, we first imaged Sample 1 as a proof-of-principle experiment, with the topography image, LFM images and the representative line profiles shown in Figure 3. Images were acquired at a pixel size of 256 × 256 and a scan size of 20 µm × 20 µm. From the LFM topography image (Figure 3a), we observed circular features with diameters ranging from 1 to 2 µm and heights of 20 to 200 nm within the quadrant detector is used (Equation 1), frequently referred to as an “A-B” signal. Topographic information = (A+C) - (B+D) (Equation 1)

On the other hand, to get the surface frictional characteristics, the LFM signal is obtained from the difference between the right cells (A+B) and the left cells (C+D) (Equation 2).

Frictional information = (A+B) - (C+D) (Equation 2)

To provide a more direct illustration of how LFM works, the schematic illustration of cantilever deflection while the cantilever is scanning over a surface with different frictional domains is depicted in Figure 2. The surface structures used herein contains a raised step in the center and smooth areas on either side, with a region of higher frictional coefficient located at the flat part on the left (Figure 2a). The deflection of the cantilever as it traverses over the surface and encounters topographical features as well as heterogeneous frictional domains is shown in Figure 2b. A representative line profile of AFM topographical signal can be found in Figure 2c. Although the topographical information is the same between point 1 and point 2, a clear difference can be seen from the LFM signal (Figure 2d and 2e). The cantilever will tilt to the right as it scans from left to right over this region due to an increase in relative friction, which leads to an increase in the LFM signal. Whereas the cantilever will tilt to the left when the scan direction is reversed, and a decrease in LFM signal will be observed. The power of LFM lies in its ability to identify different components within a sample based on frictional characteristics while the surface is relatively flat, which allows the user to gain additional information about the sample. Procedure of AFM imaging Two samples were selected and imaged with a Park NX100 AFM at ambient conditions. The first sample consists of a polymer deposited on a glass substrate; hereafter refer to as Sample 1. The second sample contains graphene grown on Si; hereafter refer to as Sample 2. Images for Sample 3 were acquired in LFM Mode using a scan rate of 1.0 Hz. Images for Sample 2 were acquired in both LFM Mode and SThM Mode with a scan rate of 1 Hz. In LFM imaging, the NSC36-C cantilever (nominal spring constant k = 1 N/m) was used. In SThM imaging, a NanoThermal-10 (nominal spring constant k = 0.25 N/m) was used.

Results or Discussion Polymer on Glass

To demonstrate the performance of the LFM mode by Park AFM, we first imaged Sample 1 as a proof-of-principle experiment, with the topography image, LFM images and the representative line profiles shown in Figure 3. Images were acquired at a pixel size of 256 × 256 and a scan size of 20 µm × 20 µm. From the LFM topography image (Figure 3a), we observed circular features with diameters ranging from 1 to 2 µm and heights of 20 to 200 nm within the area under investigation, i.e., AZ = 292 nm between the two triangular cursors as indicated in Figure 3a. LFM forward (Figure 3b) and backward (Figure 3c) images were captured simultaneously along with the topography image, through which the frictional characteristic of the sample can be obtained. The LFM images clearly revealed the compositional heterogeneity within the sample, and two domains with distinct frictional coefficients were observed. To provide a more direct comparison of the signal, line profiles along the solid lines drawn in the topography image (red line in Figure 3a), the LFM forward image (green line in Figure 3b), and the LFM backward image (blue line in Figure 3d) were generated with the GEI software and the three traces are displayed in Figure 3c. From the line profiles obtained from the LFM forward scan (green line in Figure 3d) and backward scan (blue line in Figure 3c), we can have a qualitative idea regarding the frictional characteristic of the sample. In brief, a downshift in the LFM signal was observed during the forward scan (green line in Figure 3d), indicating that the movement of the cantilever was hindered by the underlying substrate due to frictional force. The cantilever was dragged by the surface and eventually a backward torsion occurred, as it scanned from left to right, which was then observed as a negative shift in the lateral...
Figure 3. (a) Topography image, (b) LFM forward scan image; (c) SThM backward scan image; (d) line profiles plotted along the red line seen in 3a, green line seen in 3b and blue line seen in 3c, respectively.

Another interesting finding here is, although drastic changes were observed in both the green trace (LFM forward) and the blue trace (LFM backward), only minor height variations were seen in the red trace (topography). Results here showcased the strength of LFM to identify different components within a sample based on the frictional properties even when no significant difference can be seen from the topographical data.

Oppositely, the LFM signal shifted upwardly during the backward scan (blue line in Figure 3d), which, again, is a result of the cantilever being dragged by the surface because of the larger frictional interaction between the cantilever and the surface. As a result, we can conclude that the frictional coefficient for the central area is higher as compared to the surrounding areas.

Another interesting finding here is, although drastic changes were observed in both the green trace (LFM forward) and the blue trace (LFM backward), only minor height variations were seen in the red trace (topography). Results here showcased the strength of LFM to identify different components within a sample based on the frictional properties even when no significant difference can be seen from the topographical data.

Graphene on Si

Upon demonstration of the LFM operation with Sample 1, we repeated the LFM measurements on Sample 2 to examine the difference in frictional characteristics between graphene and Si. In addition, we also conducted SThM imaging to study the thermal properties of the two materials. Images were acquired with a pixel size of 256 x 256 and a scan size of 35 µm x 15 µm.

In Figure 4a, topography of Sample 2 is shown. The boundary between graphene and Si, as indicated by the white dashed line, is discernible. A representative line profile was plotted along the red line drawn in Figure 4a and is shown in Figure 4d (red trace). A ∆Z of ~5 nm was measured between graphene and Si. From the LFM image in Figure 4b, the two materials were clearly differentiated. From the line profile (green trace in Figure 4d) of the LFM signal, a larger frictional coefficient was observed for Si compared to that of graphene, as evident by the downward shift in the LFM signal over Si as compared to that over graphene. According to previous literature, the nominal friction coefficient for graphene is 0.014 whereas the Nominal friction coefficient for Si is 0.2.9 Our LFM results are consistent with previous literature. Furthermore, to gain insights about the thermal properties of the two materials, SThM was performed, with the resultant SThM error image shown in Figure 4c and a representative line profile (blue trace) shown in Figure 4d. A higher SThM error was observed over Si than graphene, which indicates a higher thermal conductivity of graphene as compared to that of Si.

Conclusions

Here we demonstrate the use of LFM to differentiate surface compositional variations based on the relative differences in frictional properties, implemented with Contact Mode AFM imaging, this technique enables nanoscale characterization of frictional domains within a sample. The strength of LFM is first demonstrated in Sample 1, where the two different materials (i.e., polymer and glass) within the sample were not easily distinguishable from the topography image. However, from the LFM images, the two domains were clearly separated by their difference in frictional coefficient. Next, we applied both LFM and SThM to examine the frictional as well as thermal properties of Sample 2, which is graphene grown on Si. Qualitative results show that graphene has a lower frictional coefficient and a higher thermal conductivity as compared to that of Si. In conclusion, LFM has already found a wide range of applications in nanoscale frictional measurements and will continue to facilitate the development of many exciting technologies.

References

**AN IN DEPTH LOOK AT IMPLANTABLE ORGANIC NANO-ELECTRONICS**

**Dr. Tobias Cramer, Asst. Professor and Researcher at University of Bologna, Italy**

Flexible, biocompatible electronic materials and semiconductors are wanted to create a novel generation of bioelectronic interfaces. They are developing nano-scale charge transport phenomena in condensed matter by theoretical and experimental methods. He is the author of 45 scientific articles and book chapters.

Dr. Tobias Cramer studied chemistry and physics at the University of Freiburg (D) and finished in 2006 with a Ph. D. degree. After a period of post-doctoral fellowships with stays in Germany (Institute for Advanced Studies, Freiburg), Italy (Faculty of Chemistry and Institute for the Study of Nanostructured Materials – ISMANN, both Bologna) and United States (The Scripps Research Institute, San Diego), since 2014 he has worked as a researcher in the Department of Physics and Astronomy at UNIBO, where he is a tenure track professor. In his research he investigates nano-scale charge transport phenomena in condensed matter by theoretical and experimental methods.

**BIOELECTRONIC INTERFACES OFFER MANY POTENTIAL MEDICAL BREAKTHROUGHS INCLUDING RESTORING HEARING, CREATING ARTIFICIAL RETINAS TO RESTORE EYESIGHT AND MANY OTHER NEURAL ADVANCES FOR LOST NERVE FUNCTIONS BY CREATING PARTICULAR DEVICES FOR SPECIALISED REGIONS OF THE NERVOUS SYSTEM, IN ORDER TO COMPENSATE FOR THE LOSS OF FUNCTION.**

**IN OUR GROUP WE EMPLOY PARK NX10 AFM EQUIPPED WITH DIFFERENT MODULES ALLOWING FOR MULTI-MODAL AFM TECHNIQUES.**

**Dr. Tobias Cramer, Asst. Professor and Researcher at University of Bologna, Italy**

**IMPLANTABLE ORGANIC NANOELECTRONICS**

**An Interview with Tobias Cramer, Asst. Professor, Department of Physics and Astronomy, University of Bologna, Italy**

Professor Cramer works in the semiconductor physics group at University of Bologna, Italy, with a focus on flexible and stretchable semiconductors for bioelectronics and photonics applications. He teaches the course “Laboratory of Nanoscience and Technology” in the Master Degree “Materials Physics and Nanoscience (MANO)” He is responsible for the Atomic Force Microscopy lab and development of stretchable sensors for bioelectronics.

**Tobias Cramer studied chemistry and physics at the University of Bologna, Italy, with a focus on flexible and stretchable semiconductors for bioelectronics and photonics applications. He teaches the course “Laboratory of Nanoscience and Technology” in the Master Degree “Materials Physics and Nanoscience (MANO)” He is responsible for the Atomic Force Microscopy lab and development of stretchable sensors for bioelectronics.**

**Can you give a brief overview of Implantable Organic NanoElectronics (IONE) and describe the benefits?**

In recent years materials based on organic small molecules or polymers have been developed that combine important properties of two rather different material classes: On the one hand they show semiconducting electronic properties equivalent to materials employed in modern information and communication technology. On the other hand they also have soft and biocompatible properties as well as stability in water, all typical for biomaterials. IONE employs such materials to develop novel microelectronic implants that interface to important processes in the body, relevant to diagnose or treat disease.

**How are implantable organic nano-electronics inspired by nature?**

Information in the nervous system is processed as electronic excitations in nerve cells. The underlying microscopic mechanisms build on the cells’ ability to control tiny ionic currents. This research regards an organic nanomaterial that has been invented by Eric Daniel Glowacki at Linköping University. As Physicists we investigate in detail the microscopic mechanism that occurs when such materials are illuminated by a strong red light source. And it is remarkable. Although it consists of a layer of only a few tenths of nanometers, it leads to the efficient generation of a charged bilayer. The resulting transient ionic current pulse is sufficiently strong to excite neuronal cells. So this material is becoming a very hot candidate for artificial retina implants. With this device no battery support or wiring with cables will be necessary. The light pulse will provide...
Can you describe the potential for use in medical therapies?

Soft bioelectronic devices that interface non-invasively neuronal tissue are expected to open an enormous potential for novel medical therapies.

At first sight one would think that students just have to learn more and more, but I think this is not the case. Focus has to be set on important concepts and one also has to take experimental skills seriously. Regarding the recent developments in equipment it has also become easier to teach nanoscience. For example nowadays it is no problem that students perform their own AFM experiments independently in the laboratory course.

In our group we employ a Park X101 AFM equipped with different modules allowing for multimodal AFM techniques.

In fabrication work we use it a lot in routine and microscopic technique to decipher the behaviour with stable electronic performance. Architectures that combine soft mechanical properties of the tissue where they are embedded (≤1 MPa). Misaligning compliance with the low elastic modulus of tissue leads on the long-term to inflammation, scar formation and passivation. However, bio-electronic implants rely on patterned microelectronic structures made of more rigid electronic materials for signal recording and stimulation. The resulting mismatch in mechanical properties imposed by the requirement of a soft substrate combined with electrical functional rigid elements makes bio-electronic microelectrodes prone to stress induced mechanical failure and does not allow any further miniaturization and reduction in invasiveness.

In order to overcome these limitations our research team has been investigating amorphous oxide semiconductors as novel materials for microelectronic dosimeters. Oxide semiconductors based on Indium Gallium Zinc Oxide (IGZO) have the right structure and electronic properties to achieve fast transport of electrons as a n-type semiconductor even in the case of an amorphous, that is disordered microstructure. This opens the way to deposit the IGZO semiconductor by physical methods on flexible substrates instead of relying on crystalline growth. For our research on microelectronic dosimeters, we combined the IGZO transistor with a high Z oxide dielectric that absorbs the ionizing radiation and builds up a space charge layer.

The combined device structure, that we call radiation sensitive oxide field effect transistor (ROXFET), is shown in Figure 1. By introducing the amorphous oxide based semiconductor, we achieve a microelectronic dosimeter that outperforms silicon based devices by an order of magnitude in sensitivity achieving the detection of radiation doses down to the 100 μSv range. In addition, we demonstrate that the dosimeter can be patterned in arrays on flexible plastic foil. The fast electronic transport properties of IGZO make it also possible to integrate the ROXFET in a passive RFID circuit. With support from the French company Tagsys-RFID we created the first wireless radiation sensor that is operated without a battery. Its detection mechanism is so energy efficient, that a radio frequency signal as generated by an RFID reader provides enough energy to operate the sensor and to send back to the reader the information on the sensor irradiation status. The research has been carried out in collaboration with people at the University of Bologna, the University of Coimbra, the University of Lisbon and the University of Bologna. We also thank the European Union for financial support through the project "Soft Bioelectronic Interfaces that Interface Neuronal Tissue with the Skin" (SoftBioelectronics, grant number 666115-3) of the Horizon 2020 Programme. The project was supported by the European Research Council (ERC) through an Advanced grant (erc-2014-adg 693823).
**Introduction**

Since the inception of scanning tunneling microscopy (STM) [1], electrochemists have sought to take advantage of scanned probe microscopy (SPM) techniques to manipulate the spatial position of a probe with high resolution to facilitate simultaneous high resolution topographical, conductometric, and amperometric/voltammetric imaging of surface and interfaces [2]. Lately, scanning ion conductance microscopy (SICM) [3] has emerged as a versatile non-contact imaging tool and been employed for a variety of applications. SICM has been used to study the surface topography of both synthetic and biological membranes [4, 5], ion transport through porous materials, dynamic properties of living cells [6, 7, 8], and suspended artificial black lipid materials [9]. In addition, integration of complementary techniques with SICM has led to many exciting new applications, including scanning near-field optical microscopy (SNOM) [10] and patch clamping [11, 12]. Powerful as it is, SICM remains insensitive to electrochemical properties, or, in other words, SICM is inherently chemically-biased and has no chemical specificity.

To obtain spatially-resolved electrochemical information, scanning electrochemical microscopy (SECM), also known as the chemical microscope, developed and has been widely employed to examine localized electrochemical properties and reactivity of various materials/interfaces, such as electrode surfaces and interfaces [13, 14, 15], membranes [16, 17, 18], and biological systems [19, 20, 21, 22, 23]. Despite its many applications, SECM, however, lacks reliable probe-sample distance control, and the probe is usually kept at a constant height during conventional SECM scanning. As a result, any variation in surface topography will result in changes in probe-sample distance, and thus leading to corroboration to the measured faradaic current, which will compound the subsequent data interpretation [18].

To address the above-mentioned issues for SICM and SECM, hybrid SICM-SECM techniques have been developed, in which the SICM compartment provides the accurate probe-sample distance control, while the SECM compartment measures that facilitate assessment for electrochemical information collection. Here in this application note, first, the principle of operation for SICM, SECM, and SICM-SECM will be briefly discussed. Next, the probe as well as the sample that are used for SICM-SECM imaging experiments are described. Finally, simultaneous SICM-SECM topography imaging and electrochemical imaging with SmartScan PTR™ using a Park NX12 AFM system is demonstrated.

**Principle of Operation**

**Scanning Ion Conductance Microscopy (SICM)**

In SICM operation, an electrochemical tip (or nanopipette) is used to probe the sample raster scanning an underlying substrate immersed in bath electrolytes (i.e., PBS buffer). A Ag/AgCl electrode is back-inserted in the pipette to serve as the working electrode, while another Ag/AgCl electrode is placed in the bath solution and used as the reference electrode. As a potential is applied between the two electrodes, an ion current is generated. The amplitude of the ion current is probe-substrate distance (Dp) dependent. The ion current decreases as the probe approaches the sample surface. This result of the ion flow being hindered between the probe tip and the underlying surface. This distance relationship can be obtained experimentally with an approach curve, where the ion current is plotted as a function of Dp (Fig. 1).

**Scanning Electrochemical Microscopy (SECM)**

Scanning electrochemical microscopy employs a micro- or nanoelectrode, termed as the tip, to scan in the vicinity of a substrate. The bath solution consists of supporting electrolyte and an electrochemically active species. During SECM experiments, the WE is biased at a sufficient potential such that the electrochemically active species can be oxidized or reduced at the tip. By measuring the faradic current, quantitative electrochemical imaging of the interface region will be obtained. Several imaging schemes have been developed for SECM operation, including negative feedback mode, positive feedback mode, and substrate generative tip collection (SG/TC) mode. Feedback mode exploits the variation in the faradic current as the tip approaches the substrate, with the sign of the variation depending upon the conductivity of the surface. When the probe approaches a conductive surface, redox cycling will lead to an increase in the measured faradaic current, this known as the positive feedback. Whereas if the probe moves close to an insulative surface, the diffusion of the redox molecules to the electrode surface will be hindered, and thus, a decrease in faradaic current will be detected, also known as the negative feedback. In SG/TC mode, the sample and the tip are biased at different potentials, where the sample is biased to generate oxidized or reduced redox species in solution and the tip is biased to collect (reduce or oxidize) the species generated at the substrate.

**Hybrid SICM-SECM**

To overcome the limitations in constant-height imaging, when variations in surface topography and reactivity occur, hybrid SICM-SECM systems have been developed. An alternative approach, theta pipette is used, followed by focus ion beam (FIB) milling to expose an underlying substrate while raster scanning the probe tip. This method was described in Shi et al. [18]. In brief, nanopipettes obtained via laser pulling were first coated with 100 nm Cr adhesion layer, followed by 200 nm Au by thermal evaporation. Next, chemical vapor deposition of parylene C was performed such that the Au-coated nanopipettes were completely covered by parylene C. Finally, a focused ion beam (FIB) technique was used to expose the nanopipette and the Au crescent. A representative scanning electron micrograph of the SICM-SECM probe is shown in Figure 4. The diameter of the nanopipette range from 200 - 250 nm. The Au crescent thickness is ~200 nm.

**Sample**

As shown in Figure 3, the standard sample used for SICM-SECM experiment is a histological section of liver tissue, which consists of support electrolyte and an electrochemically active species. During SECM operation, the WE is biased at a sufficient potential or at which the redox reaction of the electrochemically active species occurs. By scanning the probe at a constant height away from the underlying substrate while raster scanning the faradaic current response, the electrochemical activity of the surface can be observed.

Prior to SICM-SECM imaging, cyclic voltammetry (CV) in a bulk solution consisted of 100 mM KC1 and 10 mM NaH2PO4 was performed. The potential range for CV measurement is from -0.5 to 0.5 V. The purpose of the CV measurement is to 1) characterize the Au crescent electrode performance and 2) to choose the potential at which the Au-crescent electrode will be biased at during SICM-SECM imaging experiments. To carry out the CV measurement, a potentiostat (Model: VMP, CH Instrument, Austin, TX) with a three-electrode electrochemical cell is used. The Au-crescent electrode served as the working electrode (WE) with respect to an Ag/AgCl reference electrode (RE) and a Pt counter electrode (CE) in the bulk solution, as indicated in the cartoon illustration seen in Figure 5a.

**Cyclic Voltammetry**

The CV measurement was performed using an electrochemical cell consisting of a working electrode (WE), a counter electrode (CE) and a reference electrode (RE). The bath solution contains supporting electrolyte and an electrochemically active species. During SECM operation, the WE is biased at a sufficient potential such that the electrochemically active species can be oxidized or reduced at the tip. By measuring the faradaic current, quantitative electrochemical imaging of the interface region will be obtained.
The Au crescent electrode (AuE) is used to acquire electrochemical signal. The potential applied between the PE and RE was 0.1 V, and the potential at the AuE was held at 0.5 V. The ammeter is used for both applying potential to and measuring the faradaic current at the AuE. The measured faradaic current is then fed to one of the auxiliary recording channels (AUX IN) of the NX12 controller and recorded in the software. As a result, simultaneous topographical imaging (from SICM) and electrochemical activity mapping (from SECM) is accomplished.

Results and Discussion

As shown in Figure 7, the far-faradaic CVs taken with the Au crescent electrode in a bulk solution containing 100 mM KCl and 5 mM Ru(NH₃)₆³⁺.

In Figure 8, representative SICM-SECM images are shown. In Figure 8a, topography image of the Au/Pyrex pattern is shown. Figure 8c (top) shows the line profile of the topographic image. The measured pitch width is 20.06 µm, which matches the actual pitch width (20 µm). The measured height of the Au bar is 303.3 nm, which is, again, agrees with the feature height of 300 nm. In Figure 8b, electrochemical activity map of the same region seen in Figure 8a is shown. The absolute value of the faradaic current over Au is ~4.5 nA, while over Pyrex, the absolute value of the faradaic current is ~3.6 nA. An overall ~981 pA faradaic current difference was observed. For Ru(NH₃)₆³⁺, starting from ~0.25 V, the reduction reaction of Ru(NH₃)₆³⁺ to Ru(NH₃)₆²⁻ takes places, and as the potential is ramped up, at ~0.5 V, the electrochemical reaction is diffusion-controlled and steady-state current is reached. The electrochemical reaction is shown below:

Ru(NH₃)₆³⁺ + e⁻ → Ru(NH₃)₆²⁻

From the CV data, a steady-state current was reached at ~0.5 V, and thus, in the following SICM-SECM imaging experiment, the bias applied to the Au electrode will be kept at ~0.5 V.

In this application note, we demonstrated the use of a Park NX12 AFM in combination with an ammeter for concurrent topography imaging and electrochemical mapping. The SICM-SECM probe utilized here consisted of a Au crescent electrode (AuE) on the top of a nanoparticle. A high resolution probe for SICM-SECM imaging was obtained by the ion current feedback from SICM, while simultaneous electrochemical signal collection achieved via the AuE of SECM. As a proof of concept experiment, a AuE pattern standard sample was imaged with the SICM-SECM technique. The Au bar and the Pyrex substrate were clearly resolved from the SICM topography image, with the bar height and pitch width closely matching the actual values. In terms of the electrochemical property mapping, higher faradaic current was seen when the probe was scanned over Au bar as result of redox cycling, while lower faradaic current was observed when the probe was over Pyrex substrate due to hindered diffusion. This capability of the SICM-SECM technique described here holds promise of many exciting applications in the field of chemical material science and battery research.

**References**


of Energy, along with important contributions from academic institutions and partners in France, Germany, Italy, Japan, Sweden, and the United States. Fermi (formerly known as Gamma-ray Large Area Space Telescope, or GLAST) was launched on June 11, 2008. It is named after Enrico Fermi, an Italian-American scientist who did pioneering work in high-energy physics.

Theories of dark matter, mainly invented by Particle Physicists, propose that it is a new sector of matter very different than normal matter.

In 1993 a group of physicists at Stanford came up with an idea. They wanted to develop a large telescope to see the entire universe and the gamma ray sky in order to find out more about dark matter. To this day, particle physicists don’t agree on what dark matter is, yet it makes up as much as 90% of matter. With this new Gamma Ray Telescope, the largest ever launched in space, this group of physicists wanted to find out more about dark matter and the hi energy universe we cannot see.

After winning an announcement of opportunity from NASA, the GLAST collaboration began, gathering funding from an international team including United States, NASA, DOE and Sweden, Italy, Japan, Germany, & France. Construction took place at the University of CA Santa Cruz and the GLAST (renamed Fermi Gamma Ray Telescope) was launched in 2008. Now after ten years in space, the scientists have some results. And they have done way beyond what they initially thought they could do.

Gamma Rays are electromagnetic short wave lengths with lists of energy at a very high frequency. 1 trillion of an atom or trillion degrees Celsius. Gamma Rays which have billion electron volts are formed by the accelerating hi energy charged particles and are visible with the FERMI LAT.

The hi energy universe was dramatically influenced by FERMI in the last 10 years, thus FERMI have we learned a lot about pulsars, states Dr. Bloom.

Pulsars are rapidly spining neutron stars, super dense objects forged when a massive star collapses and explodes as a supernova. Young neutron stars spin dozens of times a second and gradually slow with age. But if an aging pulsar is paired with a normal star in a binary system, their interaction may ramp up the neutron star’s spin to hundreds of rotations each second, creating what astronomers call a millisecond pulsar (Dzying spin coupled with a super strong magnetic and electric fields make pulsars superb natural particle accelerators, nearly 1,000,000 times more powerful than a 400 watt engine). Originally discovered nearly 50 years ago by their radio emissions, more than 2,000 pulsars have been identified to date. Radio telescopes - and later, satellites with detectors sensitive to X-rays and gamma rays - detect a quick pulse whenever a pulsar’s rotation sweeps its beam of emission across our field of view.

With a big budget increase for particle physics this year (800 million), future scientists searching for answers to the mysterious dark matter can go back to fundamental science says Dr. Bloome. Applied science was doing very well, he says, but science was dying. Fundamental science is the long term. “Since we don’t know what dark matter is, and it does look like some kind of matter - that’s the excitement of science.”

Some Fermi-LAT Collaboration members gathered at the SLAC National Accelerator Laboratory on the 10th anniversary of the Fermi Gamma-ray Space Telescope (FGST) on June 11, 2018. The LAT collaboration includes more than 400 scientists and students at more than 90 universities and laboratories in 12 countries. Above is a picture of some Fermi-LAT Collaboration members, who gathered at the National Accelerator Laboratory in front of the Fermi Gamma-ray Space Telescope (FGST) on June 11, 2018. There are about 400 members of the Fermi-LAT Collaboration worldwide. The group of 37 in the picture is mainly (half red) from SLAC/Stanford University, and includes students and engineers that helped design and build the Fermi-LAT, maintain it in operations, and analyze data transmitted from the telescope by NASA to SLAC. The Fermi-LAT is a one of a kind model of the Fermi-LAT. Dr. Elliott Bloom pictured 2nd row in red shirt. Credit: SLAC National Accelerator Laboratory Communications Department.

Above: Alice Harding studies how gamma-ray pulsars work using observations from NASA's Fermi Gamma-ray Space Telescope.
ALL SKY INTENSITY MAP DERIVED FROM 9 YEARS OF FERMI-LAT DATA SHOWS THE ENTIRE UNIVERSE

This picture is an all-sky intensity map derived from 9 years of Fermi-LAT data. August 4, 2016 (August 4, 2021) time integrated all-sky image (based on Pass 8 Source class, PSF5 event raya-intercalibration correction). The map is also integrated above 1 GeV (that is 1 billion electron volt energy gamma rays and above up to about 1 trillion electron volt energy). The intensity scale is blue color with low intensity black and high intensity white. The map shows the entire universe in astronomical Galactic coordinates (in what is called a type-internal collaboration notation). The map is based on Pass 8 Source class, PSF3 event raya-intercalibration correction (Photos and Captions provided by Dr. Elliott Bloom, Fermi-LAT)

Looking for Dark Matter by Observing Dwarf Galaxies

Dark matter makes up about 28% of the matter in the universe. Regular matter that we are used to makes up about 5% of the matter in the universe. The rest of the energy density of the universe is made up of dark energy. Thus about 80% of the matter in the universe is of a very different kind that we know about all of the elements of the periodic table, and does not shine so we can see it in any kind of EM radiation from radio to gamma rays. Theories of dark matter, mainly invented by Particle Physicists, propose that it is a new sector of matter very different than normal matter. What is it?

The Fermi-LAT community is be searching for emission of gamma-rays from dark matter that has been predicted by theoretical models of what might make up the dark matter (new types of elementary particles).

This last picture shows as a green dot a hypothetical dwarf galaxy located where there are no gamma ray point sources (this is typical in our data, though this sky location is hypothetical). Optical telescopes would measure the presence of a small galaxy with not too many stars and also indicate that the dwarf galaxy was dark matter dominated.

Fermi knows where to look as the optical telescopes precisely establish the location of this dwarf galaxy. When the Fermi - LAT does its observations and analysis of this spot it finds no emission of gamma-rays, and then we can set limits on the gamma ray intensity from this region. These set limits challenge theories of particle dark matter that predict the Fermi-LAT should have observed gamma-rays from this source. There are a number of other ways that the Fermi-LAT can search for dark matter gamma rays, but using dwarf galaxies has proven so far to give the most stringent limits on dark matter gamma ray emission.

New Search Underway for the Dark Matter at SLAC

Scientists know that visible matter in the universe accounts for only about 15% of all matter and the rest is a mysterious substance, called dark matter. Due to its gravitational pull on regular matter, dark matter is a key driver for the evolution of the universe, affecting the formation of galaxies like the Milky Way. Still searching for what dark matter is made of, scientists at SLAC believe it could be composed of dark matter particles, and WIMPs are top contenders. If these particles exist, they would barely interact with their environment and fly right through regular matter untouched. However, every so often, they could collide with an atom of our visible world, and dark matter researchers are looking for these rare interactions.

Construction Begins on One of the World’s Most Sensitive Dark Matter Experiments

The SuperCDMS SNOLAB project, an international effort led by SLAC, is expanding the hunt for dark matter to particles with properties not accessible to any other experiment.

U.S. Department of Energy funded the construction of SuperCDMS SNOLAB experiment, which will begin operations in the early 2020s to hunt for hypothetical dark matter particles called weakly interacting massive particles, or WIMPs. The DOE Office of Science will contribute $29 million to the effort, joining forces with the National Science Foundation ($12 million) and the Canada Foundation for Innovation ($3 million). The DOE's SLAC National Accelerator Laboratory is managing the construction project for the international SuperCDMS collaboration of 111 members from 26 institutions, which is preparing to do research with the experiment.

The experiment will be at least 50 times more sensitive than its predecessor, exploring WIMP properties that can't be probed by other experiments and giving researchers a powerful new tool to understand one of the biggest mysteries of modern physics, what is dark matter?

"Understanding dark matter is one of the hottest research topics – at SLAC and around the world," said Joshua Hewett, head of SLAC's Fundamental Physics Directorate and the lab's chief research officer. "We're excited to lead the project and work with our partners to build this next generation dark matter experiment." For more information go to: https://www.slac.stanford.edu/news/2018-05-07-construction-begins-one-worlds-most-sensitive-dark-matter-experiments.aspx
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