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Feature Article: “Smart on-demand Self Defensive Coatings of Biomedical Devices - How Self Activating Anti Biotics Work” Feature Interview with Dr. Svetaiis Kulkshvili, Professor, Department of Materials Science and Engineering Texas A&M University

Application Note: Using Piezorespons Force Microscopy to Observe Local Electromechanical Responses at Nanometer: PFM, also termed dynamic-contact electrostatic force microscopy (DC-EMFM) by Park Systems, is performed in an atomic force microscope (AFM) operating in contact mode with an electrically-biased conductive tip to probe local nanoscale displacements in response to electronic stimuli

Feature Story: Developing Energy from Natural Wood Without Corrosive Chemicals High surface area mesoporous carbon is successfully manufactured from natural wood via this green technique under development at Intelligent Composites Laboratory, The University of Akron – Feature Interview with Jiahua Jack Zhu, Assistant Professor, Chemical & Biomedical Engineering

Feature Interview: Park Systems AFM Luncheon at Semicon West Draws Standing Room Only Crowd Presentations by Dr. Sang-il Park, CEO and Founder of Park Systems & Prof. Krishna Saraswat Department of Electrical Engineering Stanford University

Application Note: Using SKPM with Park AFM to analyze electrical properties of metal, semiconductor device surfaces, organic and biological materials

In the News: Park Recognizes AFM Scholarship 2017 Awardees

The global market for nanotechnology products is expected to reach about $64.2 billion by 2019 and nanotechnology now impacts almost every industry sector. From changing the way medicine and diagnostic procedures are given to developing new and efficient ways to generate electricity, nanotechnology continues to transform the world in many ways to further benefit society.

How do scientists see what’s going on in the extremely small world of nanotechnology? Better microscopes are instrumental in nanotechnology developments and today’s nano scientists use high-powered microscopes with unique methods to see surface features at the atomic scale including microscopes that use thermal, magnetic, capacitive and electrochemical properties.

In this issue, we showcase one of the new revolutionary medical devices poised to improve medical technology in a feature interview with Prof. Svetaiis Kulkshvili from Texas A&M University. Her research in stimuli-responsive polymers has uncovered “Smart on-demand Self Defensive Coatings”, highly effective for release of anti biotics in the human body.

We also feature an example of a newly patented way to produce clean energy from Natural Wood without corrosive chemicals in a feature story highlighting the research at the Intelligent Composites Laboratory at The University of Akron.

Semiconductor devices continue to advance along the technology progression with newer materials and device structures. Nanoscale microscopy plays a key role in advancing this new technology. This issue also features interviews with Dr. Sang-il Park, CEO and founder of Park Systems about the role of AFM and from Dr. Krishna Saraswat from Stanford University about some of the potential future innovations in interconnects.

We also recognize two of the latest awardees of the Park AFM Scholarship which also provides access to a Park AFM to support their research. Park AFM scholarships are giving financial incentive to pioneering new researchers in Nanotechnology at leading academic institutions world-wide.

We also feature article articles on the latest ways to use AFM that includes Piezoresponsive Force Microscopy in multi layered ceramic capacitor and using SKPM with Park AFM to analyse electrical properties.

Our mission at Nano-Scientific is to publish informative articles about the many advances in nano Science and the new microscopy methods that are enabling the evolution of new nano technology across so many sectors of the economy.

We are in our third year of production and we invite you to subscribe to receive our future issues and please submit your story ideas; we would love to publish information about YOUR research!

Photo Caption: Researchers at The Ohio State University Wexner Medical Center have developed a device that can switch cell function to rescue failing body functions with a single touch. The technology, known as Tissue Nanotransfection (TNi), injects genetic code into skin cells, turning those skin cells into other types of cells required for treating diseased conditions. In laboratory tests, this process was able to heal the badly injured legs of mice in just three weeks with a single touch of this chip. The technology works by converting normal skin cells into vascular cells, which helped heal the wounds.
**feature interview**

Smart on-Demand Self Defense Coatings of Biomedical Devices - How Self Activating Antibiotics Work

*Professor Svetlana Sukhishvili is currently a Professor at Texas A&M University Department of Materials Science and Engineering where her research group focuses on stimuli-responsive all-polymer and polymer-nanocomposite assemblies for sensing, separation and biomedical applications.*

**Picture**: Prof. Svetlana A. Sukhishvili (front row) with her research group at the Department of Materials Science and Engineering Texas A&M University, where her research focuses on many areas of polymer science including stimuli-responsive all-polymer and polymer-nanocomposite assemblies for sensing, separation and biomedical applications.

**Dr. Svetlana Sukhishvili**

**Professor, Department of Materials Science and Engineering Texas A&M University**

*What are smart self-defense anti-biotic coatings and how were they discovered?*

The practical problem we address is bacterial infection associated with biomaterials, such as orthopedic implants or urinary devices, for example. The traditional treatment of biomaterial-associated infections with systemic antibiotics is often inefficient because of the formation of bacterial biofilms in which bacteria are poorly responsive to treatment. Polymer coatings have been earlier explored to prevent surfaces from colonization with bacteria, but in most cases these coatings continuously elute antibiotics, increasing emergence of antibiotic resistance. Our focus is on another approach, which minimizes the emergence of antibiotic resistance. We are using smart coatings, which contain antibiotic, but do not elute it until they get activated by stimuli. Prior work explored such on-demand activation of polymer coatings when various external stimuli, such as pH, temperature or light are used. In contrast, our group explores a different type of the coatings - self-defense coatings, which are triggered by bacteria rather than external stimuli. Specifically, we are using acids excreted by bacteria as triggers for antimicrobial release from the coating to combat adhering bacteria.

**How do layer-by-layer films enable the self activation process?**

We work with ultra-thin layer-by-layer films, which are built when molecules of at least two types are layered at surfaces in a simple dip-, spray- or spin, aqueous-based deposition. The molecules are held together in the coating by through electrostatic pairing or hydrogen bonding interactions. One type of these molecules can be an antibacterial compound. When these films are assembled, electrostatic interactions are balanced and the film interior is overall electrostatically neutral. Such coatings are stable and do not release antimicrobials at a constant pH, i.e. in absence of bacteria. When a signal – i.e. bacteria-induced acidification – is felt by the coatings, the balance of charges shifts and the coating releases antibiotic to compensate for the bacteria-induced charge imbalance.

**What is the role of 3D Hierarchical Nanostructures for this process?**

Building hierarchical nanostructures at solid surfaces (such as on titanium, often used as an implant material) boosts the efficacy of the self-defense coatings. Here, an antibacterial layer-by-layer (LbL) film and hierarchical nanostripography act synergistically to efficiently reflectometry (NIR). Students from my group then travel to ORNL to perform NR experiments. The collaboration has been extremely productive, resulting in almost a dozen joint publications with SNS ORNL within the last 10 years. We hold weekly conference calls with John Ankner to discuss prior results and plan new experiments. These interactions are mutually beneficial and enjoyable. Graduate students greatly enrich their experiences by working with the national lab experts, while through both personal interactions and through participation in the SNS Users Workshops we have a unique opportunity to provide feedback on instrumental development at ORNL and interact with ORNL’s computational/modelling community.

**What nanotechnology advances do you see in the future for biomedical devices?**

While many of the technologies we are working on still wait to make their way to the market, I envision that the work of the highly inventive scientists in the area of smart coatings will eventually make a tremendous impact on the future of biomedical devices. The coatings, which are nanoscopically thin, can be deposited on various substrates and can be designed to be extremely effective against bacterial adhesion and biofilm formation. The ‘lack’ in their thickness can be compensated by the smart design that assures targeted, on-demand ‘activation’ to deliver antimicrobials. In the future such innovative design will assure that the coatings will be available to completely eliminate chances of infection of biomedical devices, while the side effects and the development of antibiotic resistance associated with the use of these devices will be minimized.
“WE FIND AFM THAT IS PERFORMED IN LIQUID ENVIRONMENT (IN WATER) TO BE INDISPENSABLE TO DIRECTLY OBSERVE EXPANSION AND CONTRACTION OFLBL-ASSEMBLED SPHERICAL MICELLES IN RESPONSE TO VARIOUS STIMULI.”

Texas A&M University with a current enrollment of over 60,000 was founded in 1876 as the state’s first public institution of higher learning and is one of only 17 institutions in the nation to hold the triple designation as a land-grant, sea-grant, and space-grant university. Texas A&M stands today as one of the largest research universities in the United States with faculty-researchers generating more than $866 million in research expenditures, all while enhancing undergraduate and graduate education by providing hands-on research.

The coupling between an electrical and mechanical response in a material property can be found in a variety of applications ranging from sensors and actuators to energy harvesting and biology. This property can be directly measured using piezoresponse force microscopy (PFM) in Park Systems atomic force microscopes (AFMs) to directly probe the response of a material to an electrical bias. Here we demonstrate the utility of PFM for failure analysis of a multilayered ceramic capacitor. The piezoresponse of the dielectric was analyzed by evaluating the response to switching of the electric field. Additionally, discontinuous structures in the device were identified, which likely had a direct effect on device performance.

INTRODUCTION
From renewable energy to electronics and biology, there are varieties of materials that exhibit a coupling of electrical and mechanical behavior. This coupling, known as a piezoelectric effect, is an intrinsic material property where the application of an electrical field induces a mechanical response. This material property is implemented in a multitude of applications ranging from ultrasonic imaging, to actuators and sensors.1

Piezoresponse force microscopy (PFM) is one of the most established non-destructive techniques to observe local electromechanical response at the nanometer length scale. PFM, also termed dynamic-contact electrostatic force microscopy (DC-EFM) by Park Systems, is performed in an atomic force microscope (AFM) operating in contact mode with an electrically-biased conductive tip to probe local nanoscale displacements in response to electronic stimuli. These sample displacements are often very small with a low signal-to-noise ratio; therefore, a lock-in amplifier is connected to the deflection signal to selectively drive the desired frequency and by-pass unwanted signal. Since the AFM photodiode is position-sensitive, PFM can also identify the direction of electrical polarization in active piezoelectric or ferroelectric domains.

“The faculty will facilitate targeted faculty hires in this discipline in many colleges. It will be a premiere location to show our students and visitors the exciting discoveries we are making and hopefully encourage them to join our efforts.”

Professor of Materials Science and Engineering and a member of the facility’s executive committee.

“We want this facility to become an interdisciplinary research center,” said Dr. Svetlana Sukhishvili, “The facility will help users conduct research to improve multifunctional polymer-based materials that are used in many applications, including energy, health care and transportation, among others, and will benefit the entire Texas A&M research community. The project includes 29 faculty members across multiple colleges and centers, including the colleges of engineering, science, and agriculture and life sciences at Texas A&M, in addition to the Texas A&M Health Science Center, representing all entities across the university actively involved in soft materials-related research and is the only user facility in Texas specifically dedicated to the characterization of multifunctional soft materials.

Sukhishvili and the members of the executive committee are hopeful that collaborative efforts in this facility will enable new discoveries in health care, soft robotics, biomanufacturing and environmental protection by acting as a nucleus of activity for collaborative research efforts not only at Texas A&M, but ideally across the state and nation. The soft matter facility is funded through the Research Development Fund for about $1.7 million.

“AFM IS A WORKING HORSE IN OUR EXPLORATIONS OF SMART COATINGS.”

Prof. Sukhishvili is a Member of the Executive Committee of the new Soft Matter Research User Facility at the Department of Materials Science & Engineering at Texas A&M

“The faculty will facilitate targeted faculty hires in this discipline in many colleges. It will be a premiere location to show our students and visitors the exciting discoveries we are making and hopefully encourage them to join our efforts.”

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Figure 1. A schematic representation of (a-b) vertical and (c-d) lateral PFM. The AFM (a) shows vertical deflections which correspond with (b) downward or (c) lateral indentation. The piezoresponse of the dielectric was analyzed by evaluating the response to switching of the electric field. Additionally, discontinuous structures in the device were identified, which likely had a direct effect on device performance.
MULTILAYER CAPACITORS

Since piezoelectric materials have both sensing and actuating properties, they have countless applications in the electronics industry. In particular, ceramics such as barium titanate exhibit piezoelectric behavior and have proven to be robust dielectric materials in capacitors, as they can cost-effectively exhibit high intrinsic dielectric constants along with resistance to humidity and temperature. Multilayer capacitors (MLCC) are produced in large quantities, with over a trillion (10¹²) barium titanate-based MLCCs manufactured each year. They can be found in applications ranging from controlling the anti-lock brake system on a car to a heart monitor in a hospital. Society relies heavily on MLCCs to be dependable; however they can be susceptible to failure. For example, high temperatures from soldering or storage can cause thermal stress, resulting in cracking, increased current or shorts. A high-energy surge can also be catastrophic and cause high leakage currents or rupture of the device itself. However, several of the devices may not pass specification after completion through assembly line. To understand how the device fails whether during use, storage, or assembly, PFM is a powerful technique to analyze the functionality of devices, including MLCCs.

Here we report the analysis of a MLCC cross section using the vector PFM technique. Additionally, discontinuous, suggesting device failure.

Vertical PFM in VPFM, the cantilever will deflect normal to the sample surface in response to the applied bias, which indicates the presence of piezoelectric domains that point out-of-plane or normal to the sample surface (Fig. 1a-b). As a result, the EFM phase signal in the AFM will for example, appear bright for domains that point upward and dark for domains that point downward.

Lateral PFM in LPFM, to detect the piezoelectric domains that are pointed in-plane, the sample would exhibit a displacement in the lateral surface. This lateral movement would result in a torsional displacement of the cantilever and would be detected by the position sensitive photodiode as a lateral deflection indicating a polarization direction parallel to the sample surface (Fig. 1c-d).

Vector PFM in piezoelectric samples with arbitrary crystallographic orientations, the application of a tip bias will result in both in-plane and out-of-plane displacements. By simultaneously collecting both VPFM and LPFM signals, vector PFM can be performed to determine the final direction of the polarization vector in nanometer-sized grains. 

EXPERIMENTAL

A cross section of a MLCC was analyzed using a Park N5000 AFM and the LPFM signal was acquired using a scan rate of 0.2 Hz. A conductive NANOSENSORM PointProbe® Plus-Electrostatic Force Microscopy (PPPF-EFM) cantilever (nominal spring constant k = 2.8 N/m and resonant frequency F = 25 kHz) coated with PtIr5 on both the front and back-sides and normal to the surface 25 nm was used. The AFM tip was biased with 2V AC with no additional external bias applied to the sample surface effects as friction.

RESULTS AND DISCUSSION

AMLC is typically monotonic with an alternating dielectric (ceramic) and metal (electrode) layers that extend to corresponding connecting terminals at either end of the device (Fig. 2a). In this work, we characterize the cross section of a MLCC (cross section direction (Fig. 4a) and the relationship between strain and electric field (Fig. S5). The theoretical curves demonstrating the relationship between strain and electric field (Fig. 5d) and the relationship between polarization and electric field (Fig. S5a) are shown for reference. Since the amplitude directly measures the displacement of the sample, the strain response as a function of sample bias can be measured in the dielectric of the MLCC (Fig. 5b, asterisks). Fig. 5c shows a characteristic “butterfly” shape that is similar to the ideal strain versus bias curve. The coercive voltage, which is a measure of ability to withstand an external electric field without depolarizing, is 0.7 V (See figure caption for details). The theoretical hysteresis loop of the phase (or polarization) response of a ferroelectric material is shown in Fig. 5d. The true response of the MLCC dielectric with a sweeping electric field is shown in Fig. 5f demonstrates and sharp transition at the bias voltage of ~0.7 V. The offset of this value between sweeping from a negative to a positive voltage and back demonstrates retention performance of the material. Repeated polarization reversal could also provide information about ferroelectric fatigue, demonstrates and sharp transition at the bias voltage of ~0.7 V. The offset of this value between sweeping from a negative to a positive voltage and back demonstrates retention performance of the material. Repeated polarization reversal could also provide information about ferroelectric fatigue.
CONCLUSIONS

Here we demonstrated the use of LPFM to characterize the cross section of a MLCC. This technique enables nanoscale characterization of piezoelectric domains within the dielectric of the capacitor. The electrode was distinguished from the dielectric and discontinuities in the device were identified as regions that would be expected to compromise device performance. Both the response in strain (amplitude) and polarization (phase) as a function of applied bias were explored to evaluate material characteristics including hysteresis and coercive voltage. Overall, the ability to characterize the piezoresponse of materials at the nanoscale and quantify the polarization vector of a material with applied electric field enables researchers to perform local electric measurements and establish structure-property relationships for multiple applications.

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Figure 5. PFM switching spectroscopy on the dielectric material of a MLCC. (a) The theoretical strain-bias response from a ferroelectric material. 1-5 depicts the behavior with increasing bias and 6-10 shows the response to reducing bias. Points 3 and 8 on this curve provide the coercive voltage for the material. (b) The PFM amplitude image and the (c) corresponding “butterfly” shape of the amplitude as the electric field was swept from -9 to +9 V (red) and back (blue). The red asterisk denotes the region at which the spectroscopy was performed. (d) The theoretical polarization-bias response from a ferroelectric material. 1-5 depicts the behavior with increasing bias and 6-10 shows the response to reducing bias. The distance between points 3 and 8 reflects retention performance of the material. (e) PFM phase image and (f) the corresponding phase behavior as the electric field was swept from -9 V (red) and back (blue). The red asterisk denotes the location at which the spectroscopy was performed.

DEVELOPING ENERGY FROM NATURAL WOOD WITHOUT TOXIC OR CORROSIVE CHEMICALS

AN INTERVIEW WITH JIAHUA JACK ZHU
ASSISTANT PROFESSOR CHEMICAL & BIOMOLECULAR ENGINEERING

Please explain briefly how carbon is manufactured from natural wood and why it is a “greener” method?

The unique structured carbon is a replica of cellulose framework in the natural wood. Wood is like a building structure with steel frame (cellulose and hemicellulose) and concrete (lignin). By carbonizing the framework with a heat treatment process, the cellulose framework can be fixed as carbon framework. Then, lignin is selectively removed to left porous framework structure behind. Different from conventional physical and chemical activation processes, where corrosive chemicals or high-temperature process up to 800 oC are required to generate micropores, our method does not involve any chemicals. All that is needed is nitrogen, air, and a carefully programmed heating profile. By the combination of these three elements, the porous structure of carbon can be easily tuned without involving chemicals.

How does this process activate without corrosive chemicals?

The use of corrosive chemicals in traditional processes is to etch out micropores and enlarge surface area. It is very challenging to generate inter-connected pore channels. We developed a mild oxidation process that interconnected pore channels can be created by hot air. The principle is similar but the process is more economic and environmentally friendly.
Can this process be used on other biomass resources? This is a general method can be used for a great variety of biomass resources. So far, we have demonstrated the effectiveness of this method in processing softwood, hardwood, cotton and bamboo into mesoporous carbon and more testing is on the way.

How long will it be before this process is used in manufacturing? We published our first research article in 2015 and immediately attracted industrial interests. A patent was filed (US 20160272502 A1) afterwards. Right now, we are working on the process optimization for better quality control of the porous carbon products. I would expect 2-3 years before it goes to manufacturing.

How are magnetic nano composites used to remove metal from polluted water? The greatest advantage of magnetic nanocomposites is its high adsorption efficiency and easy recycling. A well-known fact is that the smaller adsorbent usually comes with better adsorption capacity, while the separation and recycling of smaller adsorbent becomes more challenging. The magnetic nanoparticles embedded in bulk absorbent can attract toxic heavy metal ions or reduce them to less toxic forms. Meanwhile, magnetic nanoparticles enable fast separation by external magnetic field.

What are self organized Nano Crystals and why are they called the thermal highway? Filler technology is widely adopted in the industry to produce thermally conductive materials. To reach satisfactory thermal conductivity, fillers need to be interconnected to cut off the thermal barrier between the particles. It can be done with extremely large fraction of fillers in matrix, however, the processability, materials cost and mechanical property of the composites will be sacrificed. Self-organized nanocrystals are thermal fillers that can be grown in polymer. The unique feature of this technology is to create interconnected crystal structure without interfaces. The phonon can be efficiently transported along the crystal structure without scattering. Therefore, such composite has great advantage in thermal conduction as compared to conventional composites.

How does Atomic Force Microscope help you image your research? Since composites hold a promising future in the thermal management area and the interface is the major thermal barrier in composites, the focus of our research is to understand the thermal transport behavior across different filler/matrix interfaces. By using Scanning Thermal Microscopy technique on Atomic force microscope platform, we can clearly identify the cylindrical structure by Park’s new 3D AFM is having a huge impact on the performance of vertical devices such as FinFET, TFET, STT-MRAM and others.
REAL LIMITS TO NANO ELECTRONICS: INTERCONNECTS AND CONTACTS

Modern electronics has advanced at a tremendous pace over the course of the last half century primarily due to enhanced performance of MOS transistors due to dimensional scaling. Silicon bulk CMOS dominated the microelectronics industry in the past. However, future Si technology is reaching practical and fundamental limits. To go beyond these limits FinFETs have been introduced and novel device structures like surround gate FETs, TunnellFETs, etc. and potentially higher performance material like Ge, III-Vs, carbon nanotubes and 2D materials are being aggressively studied.

As device scaling continues, parasitic source resistance largely dominated by contact resistance is beginning to limit the device performance. Historically the method to reduce p, by increasing doping density thereby thinning the barrier, thus allowing more tunneling current. This method works well for n-Si, p-Si, many III-Vs and 2D materials largely dominated by the inability to dope them heavily. There are many other alternatives to reduce contact resistance such as, metastable doping, Fermi level de-pinning and band engineered heterostructures.

While novel structures and materials have enhanced the transistor performance, the opposite is true for the interconnects that link these transistors. Looking into the future the relentless scaling paradigm is threatened by the limits of copper/low-k interconnects, including excessive power dissipation, insufficient communication bandwidth, and signal latency for both off-chip and on-chip applications. Many of these obstacles stem from the physical limitations of copper/low-k electrical wires, namely the increase in copper resistance as wire dimensions and grain size become comparable to the bulk mean free path of electrons in copper and the dielectric capacitance.

Thus, it is imperative to examine alternate interconnect schemes: carbon nanotubes (CNT), graphene, optical interconnects, three-dimensional (3-D) integration and heterogeneous integration of advanced technologies on the silicon platform.

What are 3D self-assembled devices? Do you know where research on these type of devices is being performed?

3D self-assembled devices are created by influencing molecules to bind one another in large numbers to create 3D structures. This occurs all the time in biological processes and now we are applying research in related fields such as supramolecular chemistry to predict how and eventually get elements to self-assemble into structures useful for specific applications such as transistors on a semiconductor chip.

Self-assembly is one of the holy grails of nanotechnology and is subsequently a very hot topic for research and industrial labs around the world to explore.

How will advances in 3D integration affect our technology? Can you give some examples of future products or technologies that do not exist now but may once we achieve heterogeneous 3D integration on silicon platforms?

3D integrated circuits are strongly considered to be answer to ever-growing market demands for continuously miniaturize and improve our electronics. As we select and optimize from the currently fragmented playing field of myriad 3D integration techniques, we should see next-generation tech products with greater storage capacity, lower power requirements, more efficient thermal designs, higher brightness and more vivid displays and lighting, and faster networking throughout than ever before. For example, currently, smart watches are generally positioned in the market as fitness trackers or complementary devices to more powerful mobile devices like smart phones due to the watches’ technical limitations. With advanced 3D integration capabilities, we may finally be able to un-tether next-generation smart watches and have them available as more stand-alone products than they currently are.

How is Park AFM (or just AFM in general) used in the emerging and future technologies for 3D heterogeneous integration? Does AFM play an important role in helping advance these technologies more quickly and why?

To perform the direct surface bonding of one wafer to another (as in 3D crystallization techniques), each of the wafers need to have exceptionally smooth surfaces with roughness values below 1 nm. Atomic force microscopy plays a significant role in obtaining surface roughness measurements of silicon wafers to be used in KSu fabrication.
Question: Park Systems is the leading AFM technology for both academic and industrial users. What were the key factors for Park’s enormous success in advancing AFM technology for both types of users who often have very different requirements?

Answer: During the last 20 years, we have intensively developed the core technology of AFM, such as flat scan systems, True Non-Contact mode, and advanced software. We also had a master plan to cover a full line of products from basic research AFM to the high-end fully automated in-line AFM. Since all of our products were designed with the same concept, they share a common platform. For example, we develop and manufacture the flexure scanners that we use in both research AFM and industrial AFM. This enables our development and production teams to be cross-sectional and address both academic and industrial demands. This operation model allowed us to share the developments for each type of users with the other user classes. Hence, you can see a development on the academic models that was initially developed for an industrial user and vice versa.

Question: How challenging is it to perform such measurements?

Answer: How challenging is it to perform such measurements?

Question: Park Systems has high frequency cantilevers that can scan much faster. Combined with bi-lateral scan and optimized navigation, our in-line AFM can meet the throughput requirement. Since AFM can measure actual dimensions directly, AFM has been a solution for local height variations of SADP Fin and semiconductor surfaces. Recently, SKPM has also been used to study the electrical properties of organic materials, devices, and biological materials. To eliminate any confusion, let us look into SKPM’s synonyms for this technique:

- SKPM: Scanning Kelvin Probe Microscopy, or SKPM, was introduced as a tool to measure the local contact potential difference between a conducting atomic force microscopy (AFM) tip and the sample, thereby mapping the work function or surface potential of the sample with high spatial resolution. Since its first introduction by Nonnenmacher [1], SKPM has been used extensively as a unique method to characterize the nanoscale electrical properties of metal or semiconductor surfaces and semiconductor devices. Recently, SKPM has also been used to study the electrical properties of organic materials, devices, and biological materials. To eliminate any confusion, let us look into SKPM’s synonyms for this technique:

- SKPM: Scanning Kelvin Probe Microscopy
- KPFM: Kelvin Probe Force Microscopy
- SSPM: Scanning Surface Potential Microscopy
- SKFM: Scanning Kelvin Force Microscopy
- SP-AFM: Surface Potential Atomic Force Microscope

**INTRODUCTION**

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**MICROSCOPY**

SKPM will be used in this document, as it is the most widely used descriptor for this technique.

The term ‘Kelvin force’ refers to similarities between this microscopic technique and the macroscopic technique, which is the Kelvin probe method. However, the methodology is somewhat different, but the measurement is equivalent for both techniques. For clarity, this note will refer only to the microscopic technique, SKPM.

**FUNDAMENTALS OF SKPM**

The SKPM measures Contact Potential Difference (CPD) between a conducting AFM tip and a sample. The CPD (VCPD) between the tip and sample is defined as:

\[
\Delta V = -e \ln \left( \frac{V_{sample}}{V_{tip}} \right)
\]

Where \( V_{sample} \) is the potential of the sample surface and \( V_{tip} \) is the potential of the tip. The CPD is defined as the difference in potential between the tip and sample.

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**HOW TO OBTAIN SAMPLE POTENTIAL DATA FOR SKPM MEASUREMENT**

**USING SKPM WITH PARK AFM TO ANALYZE ELECTRICAL PROPERTIES OF METAL, SEMICONDUCTOR DEVICE SURFACES, ORGANIC AND BIOLOGICAL MATERIALS**

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\Delta V = -e \ln \left( \frac{V_{sample}}{V_{tip}} \right)
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Where \( V_{sample} \) is the potential of the sample surface and \( V_{tip} \) is the potential of the tip. The CPD is defined as the difference in potential between the tip and sample.

**HOW TO OBTAIN SAMPLE POTENTIAL DATA FOR SKPM MEASUREMENT**

**USING SKPM WITH PARK AFM TO ANALYZE ELECTRICAL PROPERTIES OF METAL, SEMICONDUCTOR DEVICE SURFACES, ORGANIC AND BIOLOGICAL MATERIALS**

**INTRODUCTION**

Scanning Kelvin probe microscopy, or SKPM, was introduced as a tool to measure the local contact potential difference between a conducting atomic force microscopy (AFM) tip and the sample, thereby mapping the work function or surface potential of the sample with high spatial resolution. Since its first introduction by Nonnenmacher [1], SKPM has been used extensively as a unique method to characterize the nanoscale electrical properties of metal or semiconductor surfaces and semiconductor devices. Recently, SKPM has also been used to study the electrical properties of organic materials, devices, and biological materials. To eliminate any confusion, let us look into SKPM’s synonyms for this technique:

- SKPM: Scanning Kelvin Probe Microscopy
- KPFM: Kelvin Probe Force Microscopy
- SSPM: Scanning Surface Potential Microscopy
- SKFM: Scanning Kelvin Force Microscopy
- SP-AFM: Surface Potential Atomic Force Microscope

**MICROSCOPY**

SKPM will be used in this document, as it is the most widely used descriptor for this technique.

The term ‘Kelvin force’ refers to similarities between this microscopic technique and the macroscopic technique, which is the Kelvin probe method. However, the methodology is somewhat different, but the measurement is equivalent for both techniques. For clarity, this note will refer only to the microscopic technique, SKPM.

**FUNDAMENTALS OF SKPM**

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Equations 2.8 and 2.9 are correct. They are mutually inverted. This proves that the inverted signs, positive and negative expressions in adjacent points along the y-axis. It can be seen in the metal regions in the potential difference (Vext) image Figure 2. SKPM schematic used by Park Systems. Figure 3 (e) is the line profile data of the mean values for 16

\( \omega \) is electrical force

\( \omega \) is used for capacitance

\( \omega \) (Equation 2.6) is used to measure frequency

\( \omega \) a static deflection of the AFM tip. \( F \) with \( FDC \) (Equation 2.5) results in

This equation can be divided into three components (due to the electrical force) will be superimposed to the mechanical oscillation of the AFM tip. A lock-in amplifier is employed to measure the VCPD, to extract the \( F \). The output signal of the lock-in amplifier is directly proportional to the difference between VCPD and VExt. The VCPD value can be measured by applying \( VCPD \) to the AFM tip, such that the output signal of the lock-in amplifier is nullified and \( F \) reaches zero: Subsequently, the value of \( VCPD \) is obtained for each point on the sample surface and maps the work function or surface potential of the whole sample surface area. The contact potential difference, VCPD, is thus given for the two cases as:

\[
V_{\text{CPD}} = \begin{cases} 
V_{\text{Ext}} & \text{for} \ V_{\text{ext}} < 0 \\
-V_{\text{Ext}} & \text{for} \ V_{\text{ext}} > 0
\end{cases}
\]

where Equations 2.8 and 2.9 are for the cases of voltage application on the sample and the tip, respectively. After the nullifying procedure, i.e., when VCPD, we obtain VExt=±VCPD, where “+” and “−” refer to external bias applied to the sample and the tip, respectively.

**SKPM MODE FOR PARK SYSTEMS**

There are various methods of measuring the SKPM mode in AFM. Among them, Park Systems uses two frequencies as shown in Figure 2. Two implemented lock-in amplifiers in the controller are used for each frequency modulation. One frequency is used to oscillate the cantilever and obtain a surface image using frequency, which is the term for oscillating the cantilever using piezo-electric material. The other frequency directly signals the cantilever at 17.3 kHz which is the frequency generally used for SKPM.

The topography signal and potential signal are acquired from each frequency simultaneously and two images are created without affecting each other. This allows the user to obtain a surface image and a potential image using a single scan. The topography signal is obtained by keeping the distance constant between the tip and sample. The other potential image is obtained by applying a default external voltage and potential measurement voltage on the cantilever as described in Figure 2.

**NHCAu cantilever of NANOSSENSORS** was used for this work. The cantilever has a metallic layer coated on both sides of the cantilever and has a typical tip radius of curvature smaller than 50nm. The resonant frequency and force constant is 330 kHz and 42Nm−1, respectively. The offset between the tip and the surface sample may occur as shown in Figure 3(a). The cause for this offset is the electrical factors that occurs from the VAC amplitude. Therefore, to know the offset in the SKPM measurement, it is necessary to measure the HOPG or calibration sample, which have work function values that are known in advance. One point is to be noted—the difference between the Au and Al area must be constant according to the applied direction because there is an absolute difference in work function.

**ANALYSIS & REPEATABILITY ABOUT SKPM**

The purpose of SKPM is to obtain the work function for the measuring specimen, not the VCPD between the tip and the sample. Therefore, accurate analysis is essential. To obtain the exact work function of the sample, it is necessary to measure several cantilevers and average them to obtain more accurate work function values of the samples. Calibration of the system must be done by using a sample that has a work function that is already known, for instance HOPG. First, a precise tip’s work function must be measured using the same tip with a given work function. This process is done to eliminate the electrical offset that could possibly happen during SKPM measurement. Secondly, after measuring the SKPM of the sample, the work function of the tip is obtained by using Equation 2.10. Finally, repeating this process several times and averaging the results will produce more accurate results.

The sample consisted of three different materials: Au, Si, and Al. For the calibration surface sample, Au was selected to become the base material. The work functions of Si and Al were determined as explained above. The theoretical work function values and the experimentally determined work function values of Si and Al are shown in Table 1.

**REFERENCE**

PARK SYSTEMS OFFERS ATOMIC FORCE MICROSCOPE (AFM) SCHOLARSHIP

Park Systems Park AFM Scholarship Award is eligible to postdoctoral students or researchers working in nanotechnology research using Park AFM. Park Systems will locate a Park AFM for those who do not have access to that system but wish to use it for their scholarship application. As progress for nanotechnology research and development advances at an unprecedented rate, universities worldwide offer degree programs in nanotechnology. Park Systems, world-leading manufacturer of Atomic Force Microscopes is offering two monetary scholarships to promote the education of future scientists and engineers in a number of nanoscale research areas that require advanced nano microscopy for analysis and to promote shared research findings and methodologies amongst researchers.

For more information on the Park AFM Scholarship, go to http://www.parkafm.com/index.php/media/programs/park-afm-scholarship

RECENT PARK AFM SCHOLARSHIP AWARD WINNERS

JAMEY GIGLIOTTI

PHD CANDIDATE GEORGIA INSTITUTE OF TECHNOLOGY, SCHOOL OF MATERIALS SCIENCE AND ENGINEERING

Jamey Gigiotti is a PhD candidate at the Georgia Institute of Technology in the School of Materials Science and Engineering. He began his tenure at Georgia Tech studying the growth of nanoscale piezoelectric films before joining the Epitaxial Graphene Lab where he continues to work in close collaboration with researchers at Georgia Tech Lorraine in Metz, France. Before coming to Georgia Tech, Jamey completed his undergraduate education at Penn State University, a few hours from his hometown of Harrisburg, Pa. While at Penn State, he worked on piezoelectric micromachined ultrasonic transducers for medical imaging and sonotweezing applications. This research took him to Germany to work with ultrasound simulation experts. While the focus of his research has changed several times, a unifying theme has been the deposition and characterization of nanoscale material systems. He plans to continue along this theme, but with a new focus after graduation.

1. Summarize the research you are doing and explain briefly how it will impact society. Why is your research important?

Nanoelectronics are a pervasive technology, but have not undergone a transformative technology change in decades. Epitaxial graphene, a single layer of carbon atoms arranged in a honeycomb lattice on a single crystal SiC substrate, is a promising technology platform to disrupt the silicon industry due to its unique electronic transport and robust thermal and chemical stability. However, despite billions of dollars in research, graphene technology has not trickled into industrial products, largely due to the need to reliably integrate graphene with dielectric and semiconducting materials to make useful and reliable electronics. Boron nitride, a dielectric isomorph to graphene, is an ideal candidate material which has been shown to preserve the electronic transport of graphene. Yet, to date, a scalable method to produce graphene-BN heterostructures has not been found. My research focuses on developing novel deposition techniques to enable epitaxial growth of boron nitride layers directly on pristine graphene surfaces, a necessary step to achieving industrially relevant graphene-based nanoelectronics.

2. What is the most useful part of using Park AFM for your research? Please explain what features are most useful and why?

Graphene and BN are both 2D materials and demand a unique suite of characterization tools including scanning probe, electron microscopy, diffraction, and optical techniques. Scanning probe techniques are highly sensitive to surface topography and chemical states. As such, the Park AFM provides us with a direct probe of the individual atomic layers and can aid in differentiating SiC buffer layer, graphene, and boron nitride regions, which is extremely difficult with scanning electron microscopy. For this, lateral force microscopy (LFM) is vitally important and can identify nanoscale regions of graphene and boron nitride from the SiC substrate. Graphene and boron nitride, both sp2 bonded materials, interact only very weakly with the tip compared to SiC, which provides a stark imaging contrast. This information aids in our understanding of how our systems nucleate and grow, which guides the development of our custom deposition tools and processing conditions to achieve higher quality materials with more control over their morphology.

“PARK AFM PROVIDES US WITH A HIGH-RESOLUTION AND RELIABLE MEASUREMENT OF THE THICKNESS AT THE SUB-NANOMETER LEVEL.”

XIN YIN

PHD CANDIDATE IN THE DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING AT UNIVERSITY OF WISCONSIN-MADISON

Xin Yin is currently a PhD candidate in the department of Materials Science and Engineering at University of Wisconsin-Madison. He joined Professor Xudong Wang’s group in 2012. His research interests include study of growth kinetics in various 2D nanomaterials, and synthesis and electronic properties of nanomaterials.

1. Summarize the research you are doing and explain briefly how it will impact society. Why is your research important?

Two-dimensional (2D) nanomaterials, with just one or a few atomic layers, exhibit physical properties dissimilar to those of their bulk counterparts. However, the current 2D materials have been largely limited to naturally layered materials like graphene and transition metal dichalcogenides.

My research mainly focuses on the growth of atomically thin layered materials. With a unique growth method, single-crystalline nanomaterial-thick ZnO nanosheets with the size up to 200 micrometers are realized at water-air interface with surfactant monolayer as a template. More importantly, the thickness of ZnO nanosheets could be tuned from one unit cell to four unit cells.

This is the first time to realize the growth of atomically thin layered materials. On one hand, it provides an opportunity to study the electronic, photonic, and mechanical properties emerging from the ultrathin feature of ZnO. For example, our investigation has found that the work function shows monotonic increase with nanosheet thickness. This thickness-dependent work function provides a good flexibility in designing heterojunctions.

2. What is the most useful part of using Park AFM for your research? Please explain what features are most useful and why?

Since we focus on the synthesis of 2D nanomaterials, the characterization on the thickness of the nanosheets is important. Park AFM provides us with a high-resolution and reliable measurement of the thickness at the sub-nanometer level. Moreover, to locate at one specific position for the nanosheets and electrical properties, as well as the mapping measurement, such as the surface potential mapping and current mapping, provides a chance to recognize and investigate the property difference from point to point.
The most versatile atomic force microscope for analytical and electrochemistry

Park NX12
The most versatile atomic force microscope for analytical and electrochemistry

Built on proven Park AFM performance
Equipped with inverted optical microscope

Proven Performance
The Park NX12 is based on the Park NX10, one of the most trusted and widely used AFMs for research. Users can rest assured that they are taking measurements with a cutting-edge tool.

Built for Versatility
Multi-user labs need a versatile microscope to meet a wide range of needs. The Park NX12 was built from the ground up to be a flexible modular platform to allow shared facilities to invest in a single AFM to

Competitive Pricing
Early career researchers need to do great work with cost-effective tools. Despite its outstanding pedigree, the Park NX12 is priced affordably—ideal for those on a constrained budget.

To learn more about Park NX12 please call: +1-408-986-1110 or email: inquiry@parkAFM.com

www.parkAFM.com