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The Magazine for Nanotechnology

CURRENT TRENDS IN SPINTRONICS AND NANOSTRUCTURED MATERIALS

p. 7

HOW HIGHLY EFFICIENT PEROVSKITE SOLAR CELLS ARE VISUALIZED USING PARK AFM

p. 10

ELECTRICAL CHARACTERIZATION OF SEMICONDUCTOR DEVICE

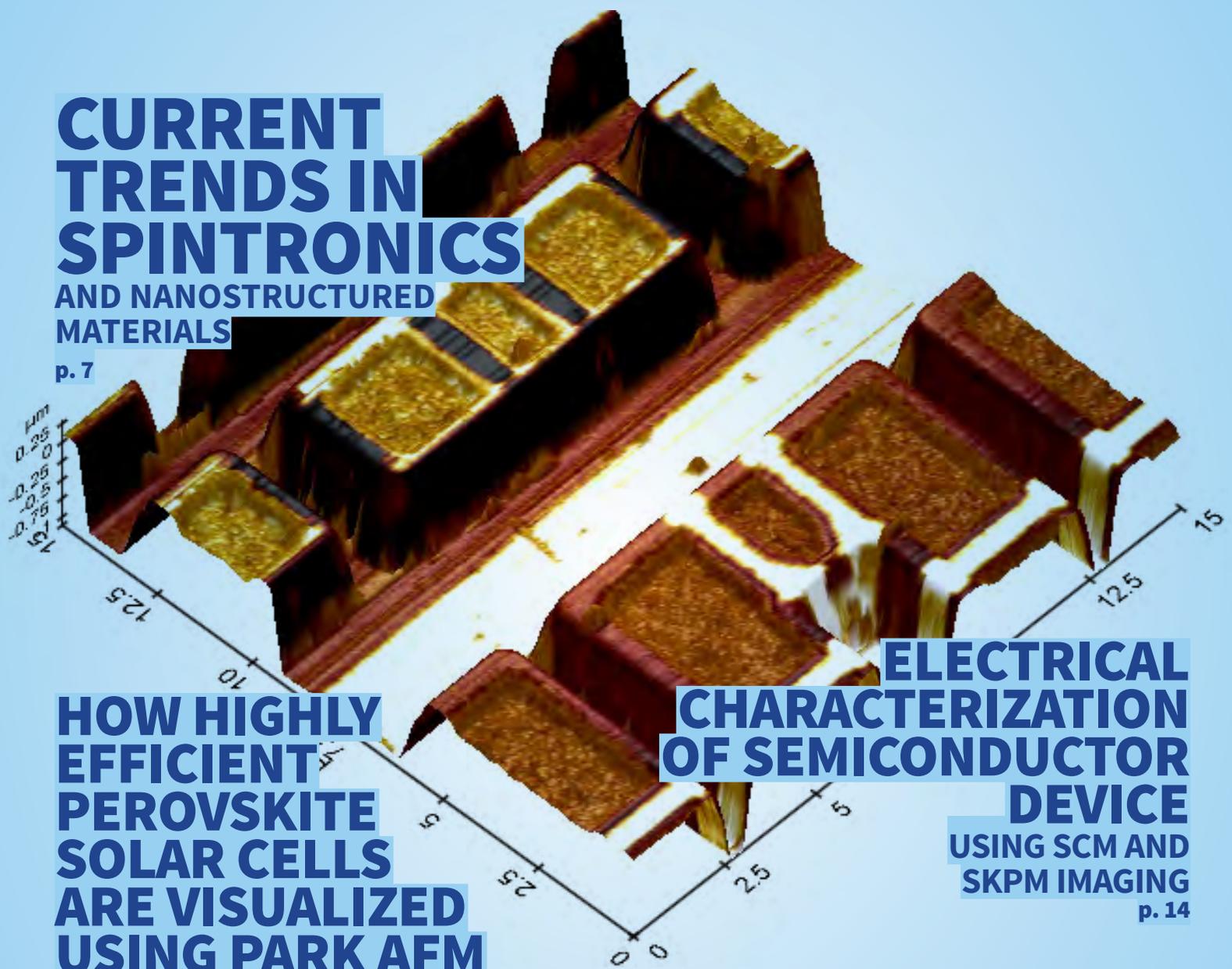
USING SCM AND SKPM IMAGING

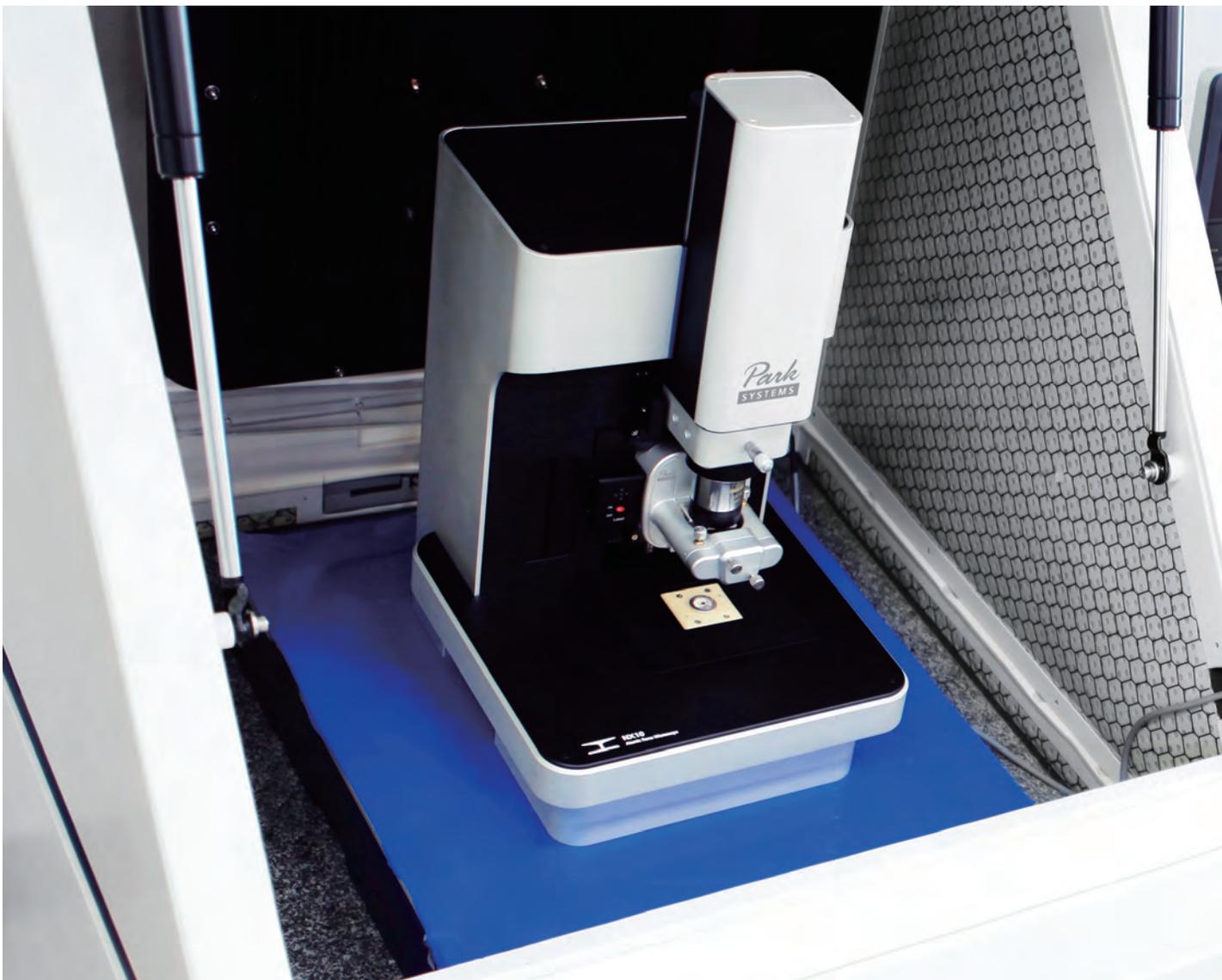
p. 14

TEXTILE NANO-CHARACTERIZATION

TOPOGRAPHY, PHASE IMAGING, AND NANOMECHANICAL PROPERTY INVESTIGATION OF POLYESTER YARN INTERACTION WITH SILICON MATRIX

p. 23





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TABLE OF CONTENTS

[Message from Editor: The Power of AFM](#) 5

[Feature Interview with Aaron Littlejohn, Dept. of Physics, Applied Physics & Astronomy Rensselaer Polytechnic Institute Nanostructure Science Lab: *Current Trends in Spintronics and NanoStructured Materials*](#) 7

[Feature Article: *How highly efficient perovskite solar cells are visualized using AFM at Department of Energy's Lawrence Berkeley National Laboratory. An Interview with Sibel Leblebic*](#) 10

[Dr. John Chen VP of Technology and Foundry Operations at NVIDIA Accelerating Yield Improvement by Finding & Eliminating Defects is the Key to Achieving “More Moore’s” and “More than Moore”](#) 13

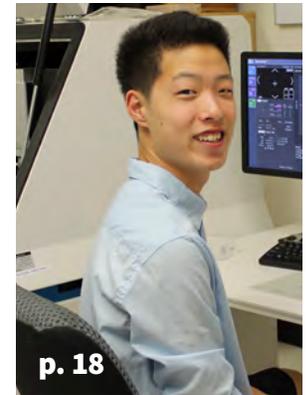
[AFM Atomic Force Microscope Technical Article by John Paul Pineda, Gerald Pascual, Byong Kim, and Keibock Lee Technical Marketing, Park Systems Inc: *Electrical Characterization of Semiconductor Device Using SCM and SKPM Imaging*](#) 14

[Feature Article from High School Intern Alvin Lee, Monta Vista High School: *Hair Damage From Sunlight Radiation Observed Using AFM*](#) 18

[Glimpses of the Nanoscale - Showcasing AFM images](#) 22

[Textile Nanocharacterization: *Topography, Phase Imaging, and Nanomechanical Property Investigation of Polyester Yarn Interaction with Silicon Matrix* by Gerald Pascual, Byong Kim, Mina Hong, John Paul Pineda, and Keibock Lee, Technical Marketing, Park Systems](#) 23

[Park Industry News: *Park Systems Receives “Frost & Sullivan 2016 Global Enabling Technology Leadership Award” and KOSDAQ “The Best Next-Generation Company Prize”*](#) 26



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Cover image:
Electrical Characterization of Semiconductor Device Using SCM

Cover photo displays 3D topography overlaid with corresponding Scanning Capacitance Microscopy (SCM) image of Static Random Access Memory device with regions of different doping levels from $2 \times 10^{16} \text{ cm}^{-3}$ to $2 \times 10^{20} \text{ cm}^{-3}$. The SCM image clearly shows various regions doped with different types of dopants and at varying concentration levels. The various steps in the color gradient are particularly helpful with observing concentration levels as several regions on the device show various shades of bright (p-type dopant presence) and dark (n-type dopant presence) color mapping. The intensity of the shading correlates to the degree with which those regions are doped with extremely bright and dark areas having the lowest and highest dopant concentration levels.

MESSAGE FROM EDITOR



Keibock Lee,
Editor-in-Chief



THE 2016 KAVLI PRIZE IN NANOSCIENCE

was awarded to Gerd Binnig, Christoph Gerber, and Calvin Quate (pictured left to right) “for the invention and realization of atomic force microscopy, a breakthrough in measurement technology and nanosculpting that continues to have a transformative impact on nanoscience and technology.”

“I AM AMAZED AT HOW TALENTED AND INNOVATIVE PEOPLE CONTINUE TO FIND NEW METHODS AND NEW APPLICATIONS. THIS TECHNOLOGY HAS GROWN MORE POWERFUL AND USEFUL THAN I IMAGINED, AND I AM PROUD AND HUMBLE TO HAVE BEEN A PART OF THIS INCREDIBLE JOURNEY.”

**DR. CALVIN QUATE,
KAVLI PRIZE LAUREATE,
STANFORD UNIVERSITY**

Dr. Sang-il Park, the founder and CEO of Park Systems (far right) was one of the researchers at Stanford University when the first AFM technology was developed by Dr. Calvin Quate, Gerd Binnig and Christoph Gerber and other researchers. Dr. Park, recognizing the potential of AFM technology for a wide range of fields was the first to commercialize AFM and since then thru innovation and collaboration with customers, Park has become a global leader in AFM manufacturing.



How AFM was born - recounted by Dr. Calvin Quate during his Kavli Prize award speech (excerpt)

The STM was a big scientific achievement. In fact, the inventors, Gerd Binnig and Heinrich Rohrer, went on to earn a Nobel Prize in physics in 1986 for their work on the STM. However, the STM had a major limitation. Since it depended on the flow of electron tunneling between a sample surface and the tunneling tip, it could only image conducting materials. Furthermore, most conducting materials, such as metals and semiconductors, are easily oxidized in ambient conditions, which required the STM to be put in a vacuum chamber.

I invited Gerd Binnig and Christoph Gerber to come to Stanford for a year. When they joined us in 1985, we wanted to see if we could create a microscope based on the principles of the STM, but that could image any surface, regardless of electrical conductivity. The solution we discovered was to add a second tip. We made a tiny flexible cantilever out of metal foil. We glued a sharp diamond tip onto the cantilever, working by hand under a microscope. We inserted this cantilever assembly between the sample surface and the

STM's tunneling tip. This allowed the device to work on all surfaces, not only metals. As the cantilever's diamond tip scanned across the surface of the sample, the inter-atomic force between the sample surface and the cantilever's tip caused the cantilever to bend. The STM tunneling tip then measured the bending motion of the metal surface of the cantilever.

In this way, the AFM was born – A for atomic resolution, F for force on the cantilever, and M for microscope. We published our 1985 work in Physical Review Letters in March 1986.

Further advancements in AFM technology, especially the non-contact mode in a vacuum, has enabled the AFM to achieve the dream of single atom resolution. As nanoscale measurements have become more and more important to scientific and technological advancement, the AFM is becoming the foundation of the nanotechnology industry, and instrumental in nanofabrication - the manufacture of tiny structures.

AFMs now use conducting cantilevers to measure electric potentials in samples, while others use current delivered from the tip to measure conductivity and transport at the

nanoscale. The technology has been an integral part of failure analysis testing for semiconductors.

With the rise of nanomaterials such as carbon nanotubes, AFM technology became the preferred tool for imaging nanostructures and other polymers. This has proven crucial to developing stronger, more effective materials to build with, create electronics, and develop exciting new technologies. AFMs are able to accurately measure interactions at an atomic level and changes in the properties of a sample after atomic rearrangement.

The AFM has many advantages over optical and electron microscopes. It provides three dimensional topographic data and can measure various physical properties with unprecedented spatial resolution. It works on almost any kind of surface, and can operate in air, in a vacuum, or in liquid. It achieves atomic resolution in a vacuum, and near-atomic resolution in air. Its only limitations are that it is still cumbersome to operate, and it is comparatively slow. In the future, I hope the AFM will become as easy to use as an optical microscope, and gain throughput as high as that of an electron microscope.



LOOKING AT HUMAN HAIR AT NANOSCALE

BY A HIGH SCHOOL RESEARCHER: HOW PARK SIMPLIFIED AFM

THIRTY YEARS AGO, Gerd Binnig, Christoph Gerber and Calvin Quate began developing a device that would enable us to see features smaller than one nanometer—less than 1/50,000 the diameter of a human hair and far smaller than any traditional microscope could manage. Now the technology of AFM thanks to Park Systems is available to everyone, witnessed by research on human hair done by our high school researcher, which shows just how user friendly AFM can be.

In our effort to bring AFM to a broad market we made our AFM so user friendly that it can be operated with very little training, as verified by our article, “Hair Damage From Sunlight Radiation Observed Using AFM”, where for the first time, hair cuticles were examined under an AFM microscope to witness the effects of sunlight radiation. Park’s patented and revolutionary SMARTSCAN™ continues to bring great excitement to the field of AFM research because the accurate and reliable measurements are set up and duplicated with very little training on the part of the user, saving both time and money and letting researchers concentrate on their

data. Using a Park NX20 AFM, the hair cuticle was imaged and compared by means of comparing roughness values in Rq. The study showed that Park AFM can be used with little experience and training in large part due to the patented and revolutionary SmartScan™ point-and-click operating system.

Park AFM equipment, formerly used only by PhD and other advanced scientific researchers in their labs can now be operated by users with little or no training. “Although I had never used an Atomic Force Microscope before, I was able to utilize the Park AFM easily with little guidance, making it simple to focus more on the research,” commented Alvin Lee, the high school summer intern at Park. “With the new SmartScan operating system, the equipment is designed

to produce quality images automatically with a single click.”

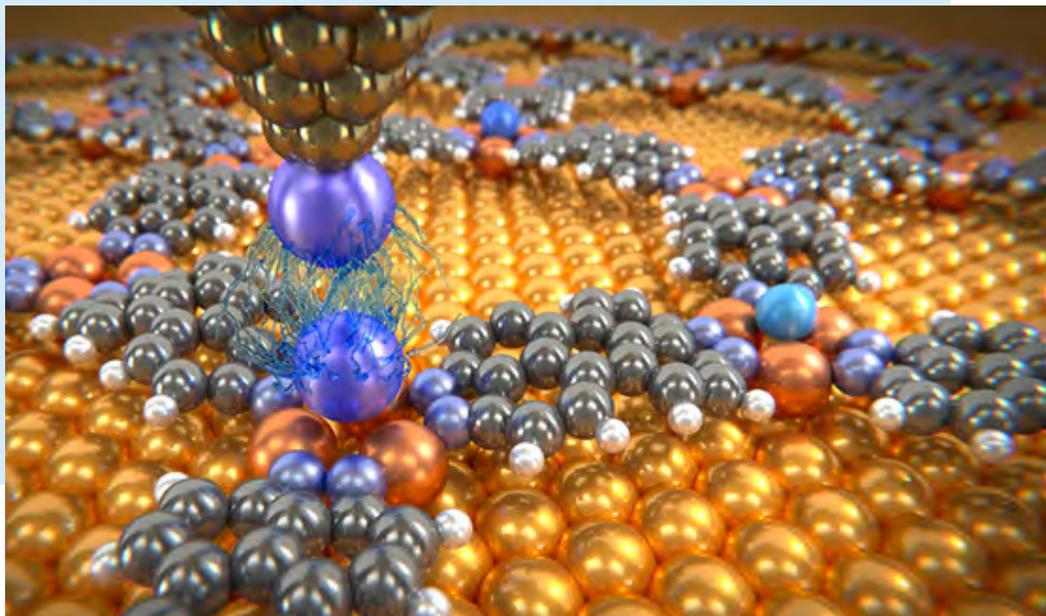
In this issue, we also have an exciting article from Lawrence Berkeley Lab, where they used the Park AFM to make a breakthrough discovery in identifying highly efficient perovskite solar cells that increase solar cell efficiency to a record 31%. We also talk to Aaron Littlejohn from Rensselaer Polytechnic Institute Nanostructure Science Lab about the latest trends in Spintronics and NanoStructured Materials, a field that continues to advance semiconductor research.

Park always looks for ways to share knowledge about NanoScience and this year, we hosted a standing room-only crowd at our Semicon West Luncheon where Dr. John Chen VP of Technology and Foundry Operations at NVIDIA presented a talk on Accelerating Yield Improvement by Finding & Eliminating Defects is the Key to Achieving “More Moore’s” and “More than Moore”, and his talk is published in this issue. We continue to host webinars on exciting topics like Electronanopatterning Strategies and using Electrochemical Scanned Probes, Stimuli-Responsive Polymers, Superhydrophobic Coatings and InSitu micropscopy using Park SICM. All of our webinars, videos and brochures can be found in the Nano Academy section of our website.

Since the invention of atomic force microscopy, advances in nanoscience are becoming more and more possible. Our shared knowledge platform is designed to help the advancement of all this new scientific research for the benefit of our society.

We are in the process of creating the Nano-Scientific website where you can view and download all of our articles and we are looking for YOUR research to highlight in our next issue. So please forward your latest NanoScientific research or story ideas.

This year, using atomic force microscopy, researchers measured van der Waals forces – very weak attractive or repulsive forces – between individual atoms for the first time. (Credit: University of Basel, Department of Physics)



CURRENT TRENDS IN SPINTRONICS AND NANOSTRUCTURED MATERIALS

AN INTERVIEW WITH AARON LITTLEJOHN, DEPT. OF PHYSICS, APPLIED PHYSICS & ASTRONOMY RENSSELAER POLYTECHNIC INSTITUTE NANOSTRUCTURE SCIENCE LAB

Can you explain briefly how you are studying new methods for synthesizing layered materials for various applications including solar cells, lithium ion batteries and integrated circuits and what benefits could result from this research?

Thin to ultrathin layered materials (and all materials in general) can be synthesized by a variety of methods from simple exfoliation – Geim and Novoselov used Scotch tape to thin down carbon to the one monolayer thickness of graphene – to more complicated techniques involving wet chemistry. When not only stoichiometry, but thickness is a crucial factor, synthesis techniques which provide direct and precise control of this parameter must be utilized. We combine well known deposition techniques (thermal deposition, sputtering, e-beam evaporation, etc.) with reactive annealing processes to form transition metal dichalcogenides (TMDCs), for example.

The materials we choose to study typically have been shown either experimentally or theoretically by density functional calculations to exhibit interesting and potentially beneficial properties. These properties may insight groundbreaking advances in nanotechnology, electronics or optoelectronics by allowing

existing devices to operate more quickly or use less energy, or by serving as the foundation for some new technology that does not yet exist. One of the great things about working in science, is that each discovery could potentially change the world in some unforeseen or unintended way. By characterizing the properties of a new material, we may provide the last puzzle piece to other scientists or engineers working on entirely unrelated projects.

What are the latest research findings regarding the growth of van der Waals heterostructures, or 2D/3D and 3D/2D material combinations?

The study of van der Waals heterostructures is an exciting and highly active area of research at the moment because of its deviations from conventional heteroepitaxy. Layered materials, which exhibit strong in-plane chemical bonds but weak physical bonds between atomic layers can in theory be exfoliated or synthesized in ultrathin form down to just one atom thick. This is highly intriguing for several reasons: 1) The properties of a film in the ultrathin form may be dramatically different than those of the bulk material. This is of fundamental interest in addition to the potential applications; 2) The next generation

Aaron Littlejohn is a fourth year Ph.D. student working under Professor Gwo-Ching Wang in the Nanostructure Science Laboratory of Rensselaer Polytechnic Institute's Department of Physics, Applied Physics and Astronomy. He is a researcher in Rensselaer's Center for Materials, Devices and Integrated Systems (cMDIS), a multidisciplinary collaboration between scientists and engineers from a variety of fields founded with the ultimate goal of fostering a safe, secure, and sustainable world by making significant contributions



to science and technology. His work is supported through Focus Center – New York (FC-NY) as part of a partnership between the state government, leading research institutes, and the U.S. microelectronics, optoelectronics, bioelectronics and telecommunications industries. FC-NY is a founding member of the Interconnect Focus Center (IFC) – a cooperation between universities and the semiconductor research industry – and one of four primary sites along with Massachusetts Institute of Technology, Stanford University and Georgia Institute of Technology.

Aaron's past research has involved studying the effects of deposition parameters on the electronic and optoelectronic properties of semiconductor alloy films. By slightly altering the growth environment many materials properties can be tuned to serve the needs of a specific application. He also researches the growth of van der Waals heterostructures, or 2D/3D and 3D/2D material combinations. This involves the growth of layered materials on bulk, non-layered substrates and traditional materials on layered substrates, and deviates from conventional heteroepitaxial film growth due to fundamental differences in the bonding mechanisms at the interface. He is currently working on developing new methods for synthesizing layered materials for various applications including solar cells, lithium ion batteries and integrated circuits. One exciting project involves studying the magnetic properties of vanadium disulfide (VS₂), a material which shows promise for implementation in next-generation spintronic systems.

of electronic devices may be made using ultrathin films, allowing them to be much smaller. This could be beneficial in increasing speed and efficiency while simultaneously using less material to do so. If material costs of production can be reduced, devices will cost less. Devices have already been engineered which use monolayer graphene and molybdenum disulfide (MoS₂), to name a couple.

In addition, by utilizing layered materials as the substrates for growth of other materials, one can take advantage of film atoms nucleating on a 2D surface which is free of dangling bonds (unpaired valence electrons). The bonding at the 2D/3D interface is thus much weaker than in traditional epitaxial growth. This allows for the growth of strain-free films without the requirement of lattice matching, a restriction which dramatically limits the choice of substrates in the growth of conventional heterostructures. Without strain at the interface, films grown by van der Waals epitaxy (vdWE) should be high quality with few defects, making them prime candidates for device implementation.

In addition to the study of a variety of 2D/3D and 3D/2D material combinations, recent applications of vdWE involve the growth of nonplanar nanostructures (e.g. nanowire arrays) for use in semiconductor devices. Future research will continue to demonstrate the viability of vdWE as a method for epitaxial film growth and potentially delve into the growth of ultrathin films on ultrathin substrates, taking the trend of scaling down to the next level.

What is methylammonium lead tribromide (MAPbBr₃) and how is it being used to create a dramatic reduction in the radiation doses used during medical examination of the human body? Are there any other potential uses for (MAPbBr₃) in other applications?

Methylammonium lead tribromide (MAPbBr₃) falls under the class of materials known as organoleadtrihalide perovskites (OTPs) which have received much attention lately for use in photovoltaics and radiation detectors. MAPbBr₃ specifically has recently been used to fabricate an X-ray detector which is capable of high sensitivity sensing of very low doses of X-rays [Wei et. al, Nature Photonics 10, 333-340, 2016]. This material attenuates X-ray photons much more efficiently than do materials that are currently used in X-ray detectors. As a result, less of this material is needed to capture X-rays than is needed for others. In



“THE PARK XE-7 ATOMIC FORCE MICROSCOPE IS ABSOLUTELY CRITICAL TO MY RESEARCH. IT ALLOWS ME TO GENERATE 3D RECONSTRUCTIONS OF SAMPLE SURFACES TO VIEW THEM WITH HIGHER MAGNIFICATION THAN IS POSSIBLE BY OTHER MICROSCOPY TECHNIQUES. I USE THESE SURFACE SCANS TO EXTRACT QUANTITIES FOR HEIGHT-HEIGHT CORRELATION ANALYSIS FROM WHICH I CAN DETERMINE ROUGHNESS PARAMETERS. I AM CONSISTENTLY ABLE TO ACHIEVE SUB-NANOMETER RESOLUTION WHICH ENSURES THE ACCURACY OF MY DATA. I USE THE LARGE RANGE SCAN MODE TO COLLECT SURVEY SCANS MEASURING AS MUCH AS HUNDREDS OF SQUARE MICRONS IN AREA AND OBTAIN FINER IMAGES FOR QUANTITATIVE PURPOSES IN SMALL SCAN MODE. THE PRECISION OF THE PARK AFM’S NON-CONTACT MODE REPEATEDLY GIVES SCANS OF THE HIGHEST QUALITY. “

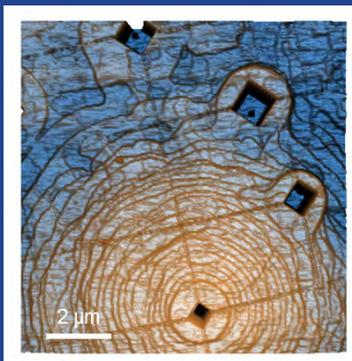
AARON LITTLEJOHN, DEPT. OF PHYSICS, APPLIED PHYSICS & ASTRONOMY RENSSELAER POLYTECHNIC INSTITUTE NANOSTRUCTURE SCIENCE LAB

addition, the product of charge carrier mobility (μ) and charge carrier lifetime (τ) (or $\mu\tau$ product), which serves as a benchmark for charge collection efficiency is a record high for MAPbBr₃, explaining the new detector’s high efficiency and sensitivity. This detector could reduce the intensity of radiation necessary to evaluate the human body during medical examination. The exciting optoelectronic properties of MAPbBr₃ may also allow it to be used in high efficiency hybrid perovskite solar cells and environmental gas detectors [Fang et al. Science Advances 2016;2:e1600534].

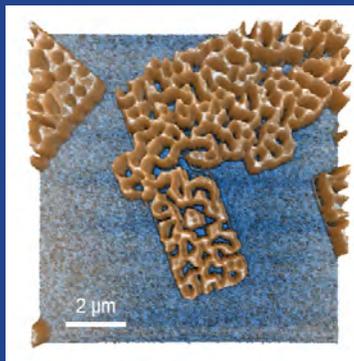
How do you use Park Atomic Force Microscope in your research and which

features to you find the most useful?

The Park XE-7 atomic force microscope is absolutely critical to my research. It allows me to generate 3D reconstructions of sample surfaces to view them with higher magnification than is possible by other microscopy techniques. I use these surface scans to extract quantities for height-height correlation analysis from which I can determine roughness parameters. I am consistently able to achieve sub-nanometer resolution which ensures the accuracy of my data. I use the large range scan mode to collect survey scans measuring as much as hundreds of square microns in area and obtain finer images for



MAPbBr₃ film – 10 × 10 μm scan
Contours reveal atomic layer step edges of just a couple angstroms in height. Regular features (square trenches and straight lines) formed due to strain relief within the film. Square trenches approx. 8 – 10 nm deep. 'Grid lines' approx. 1 – 2 nm deep.



MAPbBr₃ film – 10 × 10 μm scan
AFM allows us to explore the effects of deposition conditions on growth mechanisms in ultrathin films. This is a region of the same film which we believe was exposed to a slightly different environment (either through exposure to contaminant atoms, or different temperature or pressure). The lighter areas (film) are about 20 nm taller than the blue areas.



“RELATIVE TO TODAY’S ELECTRONICS, SPINTRONIC CIRCUITS HAVE THE POTENTIAL TO EXHIBIT INCREASED DATA PROCESSING SPEEDS AND DECREASED POWER CONSUMPTION, MAKING FOR A REVOLUTION IN COMPUTING ABILITY. THIS HAS SHAPED THE RESEARCH EFFORTS OF MANY INDUSTRY AND ACADEMIC GROUPS TOWARD FINDING MATERIALS TO MAKE NEXT-GENERATION SPINTRONIC DEVICES A REALITY. ULTRATHIN TO MONOLAYER VS₂ SHOWS PROMISE FOR USE IN SPINTRONIC DEVICES BECAUSE OF ITS UNIQUE FERROMAGNETIC PROPERTIES WHICH CAN BE DIRECTLY MANIPULATED BY APPLYING ISOTROPIC STRAIN.”

quantitative purposes in small scan mode. The precision of the Park AFM’s non-contact mode repeatedly gives scans of the highest quality.

Are the nanoscale images created by AFM essential to your current research and why?

The nanoscale images collected using our Park AFM is essential to our current research. They provide a way to examine the morphology of the films grown in our lab which we use to calibrate deposition parameters. Surface roughness is one crucial parameter to measure when characterizing a film. It has major implications to the quality of interfaces at heterojunctions, which plays a large role in device efficiency, for example in solar cells. We can also deduce growth mechanisms, or how the film nucleates and evolves, from a film’s morphology as revealed by AFM. The analysis software also makes it easy to create publication quality figures.

Can you tell us generally how spintronics is changing data storage and the semiconductor industry?

The theoretical foundation of spin-dependent electron transport has only been around for the past couple of decades. Spintronics, short for spintransport electronics, will use electric and magnetic fields to manipulate the spin angular momentum of electrons in addition to their charge as a second degree of freedom. Spin adds to the information transmitted in a circuit and can be maintained even without applying power. Thus relative to today’s electronics, spintronic circuits have the potential to

exhibit increased data processing speeds and decreased power consumption, making for a revolution in computing ability. This has shaped the research efforts of many industry and academic groups toward finding materials to make next-generation spintronic devices a reality.

What is vanadium disulfide (VS₂) and how might the study of the magnetic properties be used in next-generation spintronics systems?

Vanadium disulfide (VS₂) is a layered transition metal dichalcogenide (TMDC) comprised of layers of V atoms sandwiched between layers of S atoms. In the past, it has been studied as a potential anode for lithium ion batteries, supercapacitors, and moisture sensors. Because it exhibits a layered structure, it should be possible to exfoliate or to grow VS₂ in monolayer form.

One major area of research in spintronics is that of finding materials which will be suitable for implementation in spintronic systems. Ultrathin to monolayer VS₂ shows promise for use in spintronic devices because of its unique ferromagnetic properties which can be directly manipulated by applying isotropic strain. By increasing strain across the VS₂ monolayer from -5% to +5%, the magnetic moments of the atoms within the material increase monotonically [Y. Ma et al. ACS Nano 6, 2, 1695-1701, 2012]. This unique property could be utilized in many devices including spintronics as a mechanical switch for control of spin-polarized electron transport. However, these properties have only been predicted by density

functional calculations – I hope to synthesize and characterize this material’s electronic and magnetic properties to study the calculations’ validity. I’m also working on synthesizing different oxides of vanadium (VO₂, V₂O₅), which show promise due to the tunability of their ferromagnetic properties.

What advances in spintronics do you see in the future that might have a great impact on society?

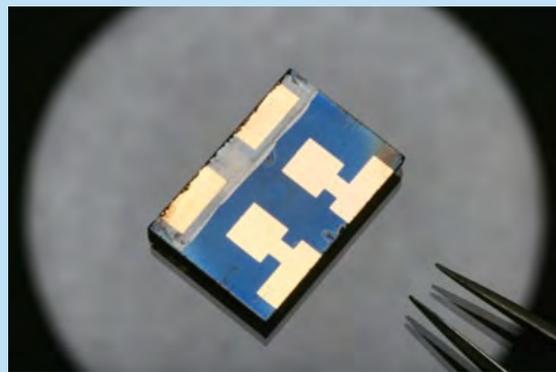
Because I’m working on the ground floor of materials synthesis and characterization and not the engineering of said materials into devices, this one is admittedly a bit tough for me to answer. . .but I can say that the impact of spintronics will be great. Faster, more powerful devices will come in smaller packages at lower cost to manufacturers. This will mean an enhancement in quality and accessibility of our current technology as well as the invention of new technologies which may solve problems currently facing the world and improve our standard of living. It’s impossible to say just how much spintronics will change the world, but the future is certainly exciting.

FEATURE INTERVIEW

AN INTERVIEW WITH SIBEL LEBLEBICI AT THE DOE-FUNDED MOLECULAR FOUNDRY AT LAWRENCE BERKELEY LAB ABOUT

HOW HIGHLY EFFICIENT PEROVSKITE SOLAR CELLS ARE VISUALIZED USING PARK AFM

SIBEL LEBLEBICI, PART OF A TEAM OF SCIENTISTS FROM THE DEPARTMENT OF ENERGY'S LAWRENCE BERKELEY NATIONAL LABORATORY (BERKELEY LAB) REPORTED NEWS IN JULY 2016 ABOUT A RECENT DISCOVERY THAT COULD DRAMATICALLY BOOST THE EFFICIENCY OF PEROVSKITE SOLAR CELLS HIDDEN IN THE NANOSCALE PEAKS AND VALLEYS OF THE CRYSTALLINE MATERIAL, RESULTING IN EFFICIENCIES IN SOLAR POWER AT THE HIGHEST PERCENTAGE EVER RECORDED.



Perovskite solar cell prototype, These solar cells convert photons to electricity more rapidly than any other material to date, which has gone from just 3 percent in 2009, now predicted to reach up to 31 percent based on new information on how to maximize them.

Solar cells made from compounds that have the crystal structure of the mineral perovskite are inexpensive and easy to fabricate, like organic solar cells. These solar cells convert photons to electricity more rapidly than any other material to date, which has gone from just 3 percent in 2009, now predicted to reach up to 31 percent based on new information on how to maximize them. More research is needed, but the promising studies reveal a significant possibility for increasing efficiency of perovskite solar cells to up to 31 percent.

This research was performed using a state-of-the-art atomic force microscopy technique, developed in collaboration with Park Systems, which utilizes a conductive tip to scan the material's surface. The method also eliminates friction between the tip and the sample. This is important because the material is so rough and soft that friction can damage the tip and sample, and cause artifacts in the photocurrent.

Why did you decide to investigate perovskites?

I chose to investigate hybrid halide perovskites for solar cells because I was extremely excited by the rapid increase in efficiency from

approximately 3% in 2009 to over 22% today. These high efficiencies are remarkable for a solution processable, polycrystalline solar cell material. When I became interested in perovskites a few years ago, the majority of the research focused on methods to improve solar cell efficiency by optimizing the film growth conditions and the energy level alignments for the other layers in the solar cell. There had also been some research on understanding the macroscale electronic properties of the material. I, however, wanted to learn more about the perovskite material on the nanoscale to understand what might be limiting its efficiency.

What did you discover about the wide variety of performance of perovskites and how were you able to produce the results?

We discovered that there is significant performance heterogeneity within individual perovskite grains in a polycrystalline perovskite film and that this heterogeneity is facet dependent. This facet dependence is due to some facets having a significantly higher density of surface trap states than others. We used conductive atomic force microscopy to locally measure the photocurrent and open

circuit voltage, two critical parameters for solar cell efficiency. To achieve reproducible results on the very rough perovskite surface, we employed the PinPoint scan mode, in which the conductive AFM tip approaches the surface, reaches a specific force between the tip and the sample, records the current, and retracts the tip at each pixel. Using PinPoint mode allowed us to eliminate all artifacts in the current measurement that result from a rough surface.

How might your discovery lead to better solar cells?

We discovered that the perovskite solar cell performance is highly dependent on the facet exposed. As a result, if the polycrystalline films can be grown such that only the high performing facets interface with the electrodes, the overall solar cell efficiency can be greatly improved.

Can you explain how you worked with Park Systems (Atomic Force Microscope manufacturer) to create the right tool for your research?

We worked together with Park Systems to improve the PinPoint mode capabilities such that it was optimized for conductive AFM. We visited the Park Systems office in Santa Clara



Sibel Leblebici recently graduated with her Ph.D. in materials science and engineering at University of California, Berkeley. Her research focused on fabricating and characterizing emerging solar cell materials including organic semiconductors and hybrid halide perovskites. She is now a postdoctoral research at the Dept of Energy funded Molecular Foundry at Lawrence Berkeley National Lab.

“WE USED CONDUCTIVE ATOMIC FORCE MICROSCOPY TO LOCALLY MEASURE THE PHOTOCURRENT AND OPEN CIRCUIT VOLTAGE, TWO CRITICAL PARAMETERS FOR SOLAR CELL EFFICIENCY. TO ACHIEVE REPRODUCIBLE RESULTS ON THE VERY ROUGH PEROVSKITE SURFACE, WE EMPLOYED THE PARK SYSTEMS PINPOINT™ SCAN MODE, IN WHICH THE CONDUCTIVE AFM TIP APPROACHES THE SURFACE, REACHES A SPECIFIC FORCE BETWEEN THE TIP AND THE SAMPLE, RECORDS THE CURRENT, AND RETRACTS THE TIP AT EACH PIXEL.”



Sibel Leblebici is part of the Weber-Bargioni group, a highly interdisciplinary and collaborative team at the Molecular Foundry, focused on exploring fundamental optoelectronic nano material properties to ultimately provide a set of rules that enable the systematic development of next generation light harvesting materials. Pictured above, Sibel Leblebici with Dr. Weber-Bargioni with the Park Atomic Force Microscope used in the research on perovskites for solar cells.



The Molecular Foundry is a Department of Energy-funded nanoscience research facility that provides users from around the world with access to cutting-edge expertise and instrumentation in a collaborative, multidisciplinary environment



Alexander Weber-Bargioni, lead Scientist, opto electronics research group at the Molecular Foundry of the Lawrence Berkeley National Laboratory.

“Our goal is to image, understand and eventually control opto electronic process in novel light harvesting materials and architectures. Currently we are focused on characterizing opto electronic properties in inorganic semiconducting and metal oxide nanowire systems, inorganic nano crystal assemblies and small molecule organic semiconductors. “

“We are blessed to be part of the Molecular Foundry – one of the most interdisciplinary and collaborative nano research centers. Six complementary facilities integrate the expertise of physicists, chemists, biologists and engineers, covering in house state of the art synthesis and fabrication of nanoscale materials, their characterization and the modeling to develop a complete understanding of physical and chemical properties of nano building blocks.”

“We don’t use FREE solar power today, which is an unfortunate detriment to humanity and our planet. I would gladly spend 5X more on my energy each month to leave a safe planet for my children to inhabit. Humanity makes iPhones that fit in our back pocket while containing more computing power than the guidance computers for the Apollo 11 mission. We really should be able to develop materials that cost effectively converts the free sun energy into electrical and chemical energy.”

FEATURE INTERVIEW

to explain what we wanted to measure and to better understand the capabilities of the system and engineers from Park Systems often visited our lab to suggest improvements. Additionally, we suggested improvements to the software to have greater control over the PinPoint mode settings.

What was the technique that was developed and how was it used? Was this new AFM tool essential to your findings?

Without the PinPoint scan mode and the improvements that were made specifically for measuring current, the experiments would have been much more challenging and the current images would have many artifacts. Using this new technique allowed for much more reproducible current measurements.

What is the estimated increased efficiency in the higher performing facets you found in perovskite and why are these facets better at light harvesting?

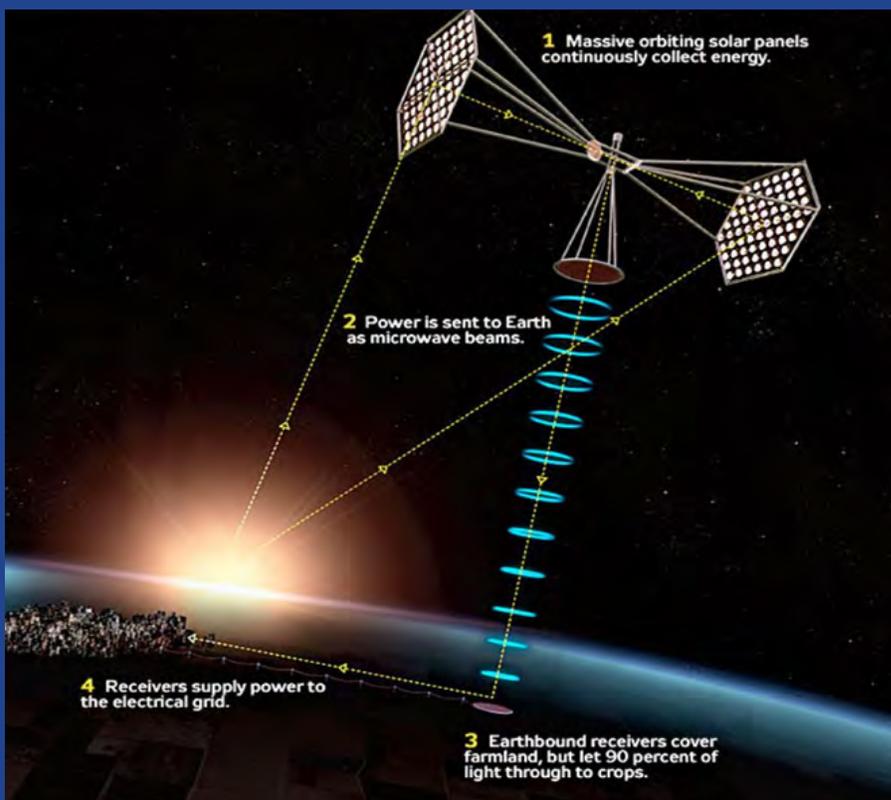
We observed up to an order of magnitude difference in photocurrent and differences of open circuit voltage up to 0.6 V between facets of the same grain. These results suggest that the efficiency of perovskite solar cells could approach the theoretical efficiency by growing the film such that only the high performing facets interface with the electrodes. These high performing facets have a very low surface defect density; thus, they have a greater short circuit current and open circuit voltage.

How is the information at Molecular Foundry shared word-wide with other researchers?

The Molecular Foundry is a user facility, meaning that anyone in the world can submit a proposal to use cutting-edge expertise and instrumentation for their research. We also have research groups within the Molecular Foundry that regularly publish research articles.

What other research projects are you working on or plan to work on in the future?

We are currently working to determine the crystallographic orientation of the high and low performing facets in hybrid halide perovskites.



Basic concept of solar energy collected and delivered via satellite and microwave technology. (Illustration: Courtesy Artemis Innovations)



SOLAR POWER FROM SPACE

A SOLAR POWER SATELLITE IN SPACE WOULD PROVIDE CONTINUOUS POWER TO PEOPLE ON EARTH BECAUSE IT WOULD HAVE ENDLESS ACCESS TO SUNLIGHT—WITHOUT INTERFERENCES FROM CLOUDS, THE ATMOSPHERE, OR NIGHT—AND WOULD NOT NEED STORAGE. STUDIES BY A TEAM AT THE NAVAL RESEARCH LAB CONCLUDED WHAT IT IS DEFINITELY TECHNICALLY FEASIBLE, BUT THE QUESTION AS ALWAYS REMAINS THE ECONOMICS. SPACE SOLAR IS CLEAN AND CONSTANT, ALLOWING IT TO SERVE AS A BASELOAD SOURCE. “THERE’S NO OTHER ENERGY SOURCE KNOWN THAT COULD SWITCH FROM ONE PART OF THE PLANET TO ANOTHER PART SO QUICKLY,” SAID SPACE ENTREPRENEUR AND FORMER NASA EXECUTIVE JOHN C. MANKINS, WHOSE SOLAR POWER SATELLITE DESIGN, SPS-ALPHA, APPEARS IN HIS 2014 BOOK, THE CASE FOR SPACE SOLAR POWER.

ACCELERATING YIELD IMPROVEMENT BY FINDING AND ELIMINATING DEFECTS IS THE KEY TO ACHIEVING “MORE MOORE’S” AND “MORE THAN MOORE”



*Dr. John Y. Chen,
VP of Technology and Foundry
Operations at NVIDIA*

Dr. John Y. Chen, VP of Technology and Foundry Operations at NVIDIA, presented an informative talk about opportunities and challenges for new applications in the semiconductor industry, titled **Growing the Semiconductor Industry as Moore's Law Slows Down** to a standing room only audience at the Park Systems AFM Semicon West luncheon on July 12, 2016. Dr. Chen discussed how improving yield to levels beyond even today's demanding standards would be the most profound way to grow the industry and advance its technology. His talk explained why nanoscale metrology with modern tools, such as Park AFM, is critical to discover, learn, and eliminate the defects that prevent semiconductor products from becoming viable.

In addition to Dr. Chen's talk, Dr. Sang-il Park, CEO and Founder of Park Systems, outlined Park's history of over 2 decades of continuous growth which led to an IPO last December on KOSDAQ, where they recently received the "Best Next Generation Company Award".

NVIDIA has been releasing products with more than double the number of transistors every 1-2 years, exceeding the cadence of Moore's Law. Its latest GP100 consisting of 15 billion transistors, 50 billion contacts, and 50 billion minimum size vias for the 12 metal layers. This 600 mm² die, integrated with HBM (High Bandwidth Memory) stacks on a silicon interposer performs at TFLOPS (Tera Floating Operations per Second) levels enabling many graphics and computing applications. Even though Moore's Law is slowing down, there are still opportunities for "More Moore's" and there will be "More than Moore". Semiconductor products such as GPUs, which utilize massive parallel processors, can always use more transistors for higher performance.

Today, Deep Learning, the modern artificial intelligence, is taking off because companies such as NVIDIA can provide powerful super computing engines fueled by "Big Data" in the cloud. NVIDIA's GPUs are the workhorse brains of the Deep Learning machines which invite the creation of many new applications.

While the prices of a single chip from \$351 (1971) to \$393 (2015) remained much the same, the features on the chip have greatly shrunk, with much more advanced capabilities at reduced costs. The minimum feature size of chips from 1971 to 2015 went from 10 μm to 16/14 nm, the transistors per chip increased from few thousands to several billions, and remarkably, the prices of 1,000 transistors went from \$150 down to \$0.03. However, going forward with further scaling, the economical benefit can hardly justify the increase cost of wafer manufacturing unless we can improve yield faster and better than ever. Hence, improving yield

is the most profound way that we have to extend Moore's Law. Whether to achieve a state of "More Moore's" by reaching beyond the 7nm semiconductor fabrication process or "More than Moore" by advancing 3D integration techniques, we can no longer afford product waste due to yield loss. Dice with billions of transistors, GPU/DRAM modules with 3D integration—the yield for these needs to be perfect. The goal must be perfection from 100 billion vias to 10,000 u-bumps with absolutely no defects, even latent ones. Dr. Chen explained that achieving great yield with defectivity at < 1 DPPB (Defective Parts per Billion) level demands essentially perfection in manufacturing. He preached the industry must work together to take the detection and the elimination of defects to the next level.

The key to doing so, Dr. Chen argues, is to adopt nanoscale metrology in earnest, metrology with the ability to image at sub-nanometer resolution, in three-dimensions, and perform at the DPPB level. He encouraged instrumentation vendors such as Park Systems, whose AFM system product line is being continuously refined to meet such stringent requirements.

Dr. Sang-il Park followed Dr. Chen to elaborate on the background of Park Systems and gave the luncheon attendees a walkthrough on the company's AFM technology and its future plans. "Since Park Systems developed the first commercial AFM in 1989, we have experienced 25 years of continuous growth and product innovation, the longest history of AFM business in the industry," commented Dr. Park, Park Systems Founder and CEO. "We have more than 1,000 Park AFM systems in use in over 30 countries around the world and our future plans include continued expansion into the global AFM market."

Over the years, the ability of Park Systems to consistently outpace the competition with critical technology innovations for semiconductor manufacturing's cutting-edge wafer production has made them a world-leader in Atomic Force Microscopes and nanometrology. At the AFM luncheon, Dr. Sang-il Park highlighted the fast-paced leadership role Park Systems has in nanoscale innovations to improve accuracy and reliability as well as to reduce cost for semiconductor manufacturers. He also spoke on the company's role as a leading innovator in emerging nanoscale microscopy and metrology technology. Park recently introduced Park NX20 300 mm, the only AFM on the market capable of scanning the entire sample area of 300mm wafers using a 300mm vacuum chuck while keeping the system noise level below 0.5 angstrom (\AA) RMS. This achievement will greatly improve production yields and is an example of the company's efforts to help the semiconductor industry find continued growth and success in the wake of a slowing Moore's Law.

ELECTRICAL CHARACTERIZATION OF SEMICONDUCTOR DEVICE USING SCM AND SKPM IMAGING

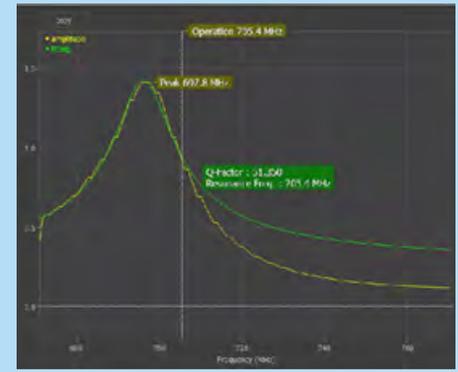


Figure 1.
Resonance RF curve displaying the SCM detector signal (V) versus frequency (MHz). The optimal frequency to oscillate the resonator to achieve the highest detection sensitivity in SCM imaging is 705.4 MHz.

By John Paul Pineda, Gerald Pascual, Byong Kim, and Keibock Lee
Technical Marketing, Park Systems Inc., Santa Clara, CA, USA

INTRODUCTION

Semiconductor devices are the foundation of modern electronics due to their importance in the function of electrical circuitry with components such as transistors, diodes, and integrated circuits. These devices have become ubiquitous in a wide range of applications. The most common of which are the design and manufacture of 1) common analog appliances such as radios and 2) digital circuits for use in computer hardware. [1] Key electrical parameters such as dopant concentration level, carrier type, and defect densities are fundamental factors that influence the performance of semiconductor devices. Thus, a technique that can measure these characteristics and investigate samples with nanoscale features must be utilized in evaluating device reliability. There are several methods for the characterization of semiconductors. Examples include Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Secondary Ion Mass Spectroscopy (SIMS), Electron Beam Induced Current (EBIC) and one-dimensional Capacitance Voltage (C-V), among others [2]. However, some of these methods are destructive, others have laborious sample preparation requirement, and others still do not effectively determine two-dimensional quantities of sub-device scale. The need for next-generation characterization tools was driven by these shortcomings as well as the realization that ever-smaller device geometry and high reliability requirements were beginning to trend within the industry. To satisfy this new degree of difficulty in the metrology of semiconductor device

processes, various types of scanning probe microscopy (SPM) have been deployed to meet the challenge. Scanning Capacitance Microscopy (SCM) and Scanning Kelvin Probe Microscopy (SKPM) combined with Atomic Force Microscopy (AFM) are the most powerful methods for characterization of semiconductor devices because of their non-destructive scanning ability, accuracy in measurements of samples with nanoscale features, and the lack of any sample preparation. In addition, the integration of these methods with AFM enables it to acquire both topography and electrical property data simultaneously without changing the sample or tip. To this end, SCM and SKPM were used to investigate an SRAM device and the data shows that these techniques are effective means for the electrical properties characterization of semiconductor devices.

EXPERIMENTAL

An SRAM sample [3] was investigated using a Park NX20 AFM system [4]. The electrical properties of the sample were characterized under ambient air conditions using two different techniques: SCM and SKPM. A cantilever with a metal-coated tip was utilized in both techniques. In SCM [5], the sample topography is collected using contact mode AFM simultaneously with capacitance imaging all in a single scan. The electrical properties of the sample are measured from the variation in radio frequency (RF) amplitude signal due to changes in capacitance between the tip and the sample. The hardware configuration of this mode consists of several modules including the cavity resonator, frame module, SCM probehead, SCM sample holder,

and an SCM cantilever chip with a connected probe wire. The SCM probehead was connected to an RF sensor, comprised of the cavity resonator and frame module, to detect the change in capacitance between the probe tip and the sample during scanning. The frame module generates and amplifies the driving signal which oscillates the resonator during SCM measurements. The cavity resonator transforms the capacitance change between the probe tip and the sample into an RF signal. The resonant frequency of the resonator is proportional to $1/\sqrt{LC}$, where L is inductance of the resonator, and C is the capacitance. For this study, a resonance RF curve with a peak of 697.8 MHz was used (see Figure 1). This curve is steepest at an operating resonant frequency of 705.4 MHz. This is the point on the curve where changes in amplitude due to frequency shifts, induced by tip-sample capacitance changes, would be most easily observed.

The output signal from the resonator is monitored and coupled with a lock-in technique to acquire the final capacitance map reported here. In this experiment, a lock-in amplifier, embedded internally in the NX electronics, with an AC voltage frequency of 17 kHz was selected after optimizing the scan parameters for acquiring topography data. Parameters for SCM imaging were also optimized by closely monitoring the SCM signal. The AC bias amplitude selected was 1 V, while for AC bias, the phase chosen was a 0° reference phase. A second order filter with a 1 ms time constant was also selected for monitoring the output signals. A sensitivity value of 1 V was set to remove unwanted noise in the signals.

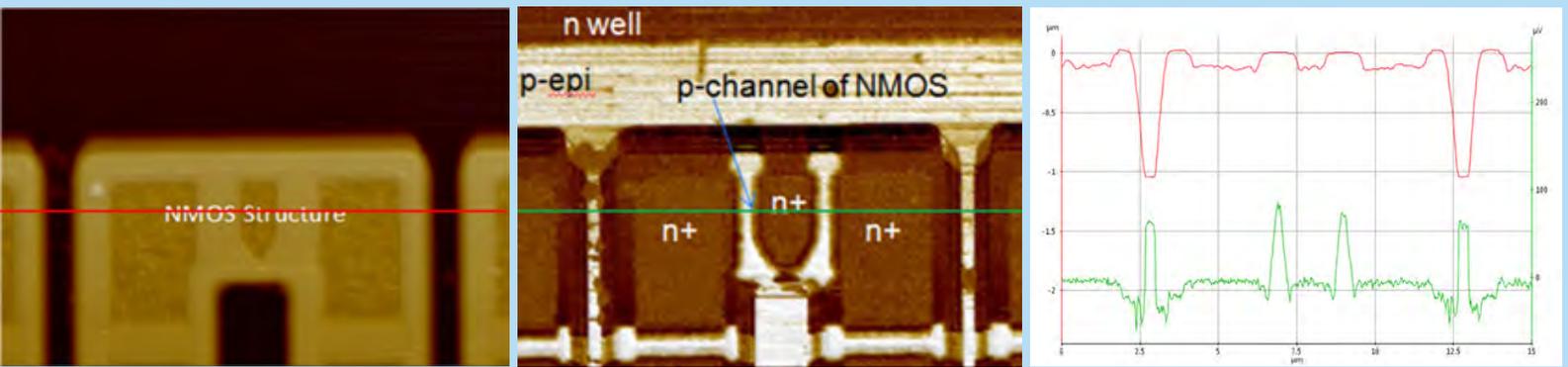


Figure 2. Topography (top-left) and SCM (top-right) data acquired from the sample device. Topography line profile (red line, y-axis on left) and SCM line profile (green, y-axis on right): Doping level: p-epi ($2 \times 10^{16} \text{ cm}^{-3}$), n well ($2 \times 10^{17} \text{ cm}^{-3}$), p channel ($1 \times 10^{17} \text{ cm}^{-3}$), n+ contacts ($2 \times 10^{20} \text{ cm}^{-3}$).

In SKPM mode, there are two interaction forces between the AC biased tip and the sample: the electrostatic force and Van der Waals force. The Van der Waals force is harnessed to generate the sample's surface topography while the electrostatic force between the tip and sample generates data for the sample's electrical properties. The obtained cantilever deflection signal contains both sets of information; therefore, a method that can completely separate these signals is the key to successful imaging. There have been methods introduced to accomplish this, one of which is two-pass scanning. However, this method is two times slower than typical AFM imaging as two separate scans need to be conducted. In the Park NX20, lock-in amplifiers embedded in its electronics are used to separate signals. This allows for the acquisition of both topography and EFM data in a single-pass scan. Two amplifiers are used by the system, named lock-in 1 and lock-in 2. Lock-in 1 obtains the topography information by analyzing the tip motion caused by the Van der Waals interaction, while lock-in 2 obtains electrical property information by analyzing the frequency of the applied AC voltage signal to the tip which, in turn, generates an electrostatic force interaction with the sample. The frequency of the applied AC voltage signal is chosen to be smaller ($\sim 17 \text{ kHz}$) than the cantilever oscillation frequency ($70\text{--}330 \text{ kHz}$), enough so that the two signals do not interfere each other [6]. In this experiment, lock-in 2 with an AC voltage frequency of 17 kHz was selected after optimizing scan parameters for topography data acquisition. Furthermore, a separate DC bias was applied to the cantilever and controlled for to create a feedback loop that would zero out

the electrical oscillation between the tip and the sample caused by the application of an AC bias to the cantilever. The value of this offsetting DC bias that zeroes out the AC bias-induced electrical oscillation is considered to be a measure of surface potential [6, 7].

RESULTS & DISCUSSIONS

The region of interest in this investigation is in the sample's NMOS region. The acquired images from each technique were analyzed using XEI software developed by Park Systems which mapped the acquired signals to a color table. The topography data acquired in both techniques clearly show the NMOS region but no significant information related to the type and level of dopant concentration. This is in contrast with the electrical property data acquired in each mode showing not only the NMOS structure, but also the type and level of doping concentrations across the sample. Figure 2, shown below, is the topography and SCM measurement of the SRAM sample with regions of different doping levels from $2 \times 10^{16} \text{ cm}^{-3}$ to $2 \times 10^{20} \text{ cm}^{-3}$. The SCM image clearly shows various regions doped with different types of dopants and at varying concentration levels. The various steps in the color gradient are particularly helpful with observing concentration levels as several regions on the device show various shades of bright (p dopant presence) and dark (n dopant presence) color mapping. The intensity of the shading correlates to the degree with which those regions are doped with extremely bright and dark areas having the lowest and highest dopant concentration levels. For example, the device's p-channel with a doping level

of $1 \times 10^{17} \text{ cm}^{-3}$ is clearly visible in the SCM image. The narrow regions of the p-channel approximately 100 nm wide show the separation of regions with alternating dopant types in the configuration of a typical NPN transistor. Furthermore, the resolution of SCM is high enough to show multiple darker spots present in what device fabricators intended to be a continuously solid bright line of positively doped material on the left side of the device. Characterizing electrical properties with this level of detail can be key in understanding the functionality of a semiconductor device.

As with the SCM and topography images, the corresponding line profiles generated after scanning can also yield significant insight into the design of the device. Here we first consider the line profile for the topography data indicated in red on Figure 2. When compared to the SCM image with the device features labeled, one can see that each NPN transistor device is separated from the next by boundaries nearly $1 \mu\text{m}$ in depth with high levels of positive dopant concentration. The edges of each boundary are bordered by a slightly raised area of about $0.1 \mu\text{m}$. If one were to overlay this topography data onto the SCM image, the edges of each boundary can be seen to have lower dopant concentration (darker color) than the n+ contacts making up the NPN transistors. This is further supported by the line profile of the SCM data (green line on figure 2) where the regions immediately before and after the boundaries (which again, feature large decreases in positive dopant concentration) are measured at slightly more negative μV levels

ATOMIC FORCE MICROSCOPE (AFM) TECHNICAL ARTICLE

(-30 μV) than the rest of the negatively doped portions (-10 μV) of the device shown in the SCM image. An additional observation can be made in the central portion of the area of interest where p-channels of an elevated height around 0.1 μm can be detected perpendicular to the p-epi region of the device. These central p-channels have a relatively lower concentration of positive dopant (approaching 80-90 μV) where they are seen bright along the both green line in the SCM image as well as in the image as a whole.

While SCM provides excellent topography and electrical property data with high spatial resolution, it is a technique that is enabled through the purchase of additional hardware from many microscopy vendors. Furthermore SCM must be performed using contact mode AFM which results in the consumption of probes at an accelerated pace compared to non-contact mode AFM. In situations limited by hardware availability or fiscal considerations, electrical characterization of semiconductor devices can still be performed at a diminished, yet viable manner using SKPM, a surface potential measurement technique. Although not as effectively detailed, SKPM can provide researchers with images and data comparable to that acquired with SCM. Both techniques can characterize the structure of the device and reveal the dopant concentrations in various regions across the area of interest. Areas with negative dopant concentrations are again presented as darker areas and those with positive dopant concentrations are shown to be brighter. The key differences between the two techniques are that of lateral resolution and dopant detection sensitivity. Comparing the SCM image with the SKPM image, one can see the p-channels of the device are much wider when observed in SKPM. One possible explanation of this difference is that SKPM is designed to see potential over an entire sample surface whereas SCM makes direct contact with the sample surface to detect capacitance responses. An alternative explanation would be that SKPM can be influenced by charges in the ambient air around the tip as well as in moisture that has adhered to the sample, both of which are possible sources of alterations in charge distribution. SCM on the other hand has the tip engage the surface of the tip directly, penetrating through any possible moisture layer, creating a point of direct contact which is less likely to be affected by parasitic charges from the scanning environment.

SUMMARY

The topography and electrical properties of an SRAM sample have been characterized using SCM and SKPM with a Park NX20 AFM system. The data collected in this investigation reveals that both techniques can provide qualitative and quantitative information for electrical characterization of semiconductor devices. The results demonstrate that SCM provides greater lateral resolution and higher contrast mapping of electrical properties, including dopant type and level of concentration, when compared to SKPM. However, SKPM remains an effective source of data that can be used to reach similar conclusions about a sample under investigation as SCM. Overall, the techniques described in this study will successfully provide researchers and device engineers with key electrical parameters information to better evaluate the device reliability and monitor semiconductor device processes at nanoscale level.

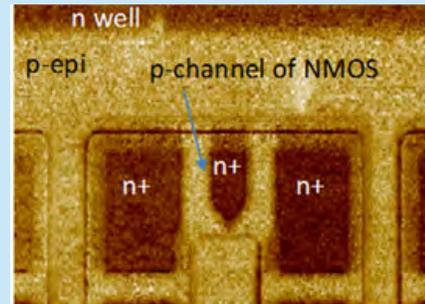
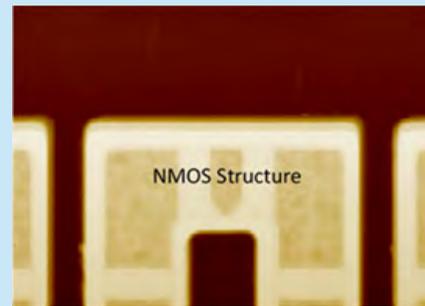


Figure 3. Topography (left) and SKPM (right) data acquired in SKPM mode. p-epi ($2 \times 10^{16} \text{ cm}^{-3}$), n well ($2 \times 10^{17} \text{ cm}^{-3}$), p-channel ($1 \times 10^{17} \text{ cm}^{-3}$), n+ contacts ($2 \times 10^{20} \text{ cm}^{-3}$).

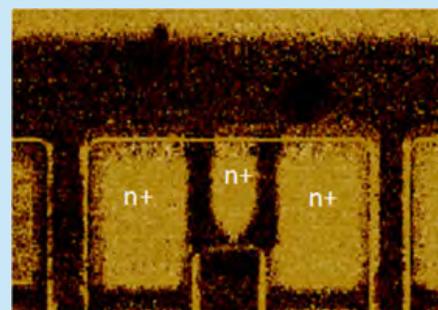
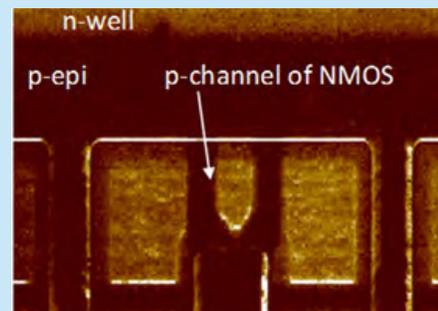
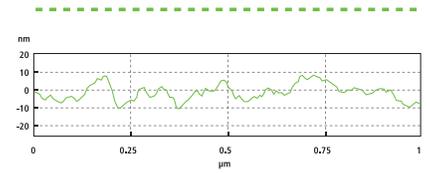
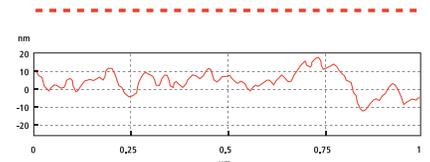
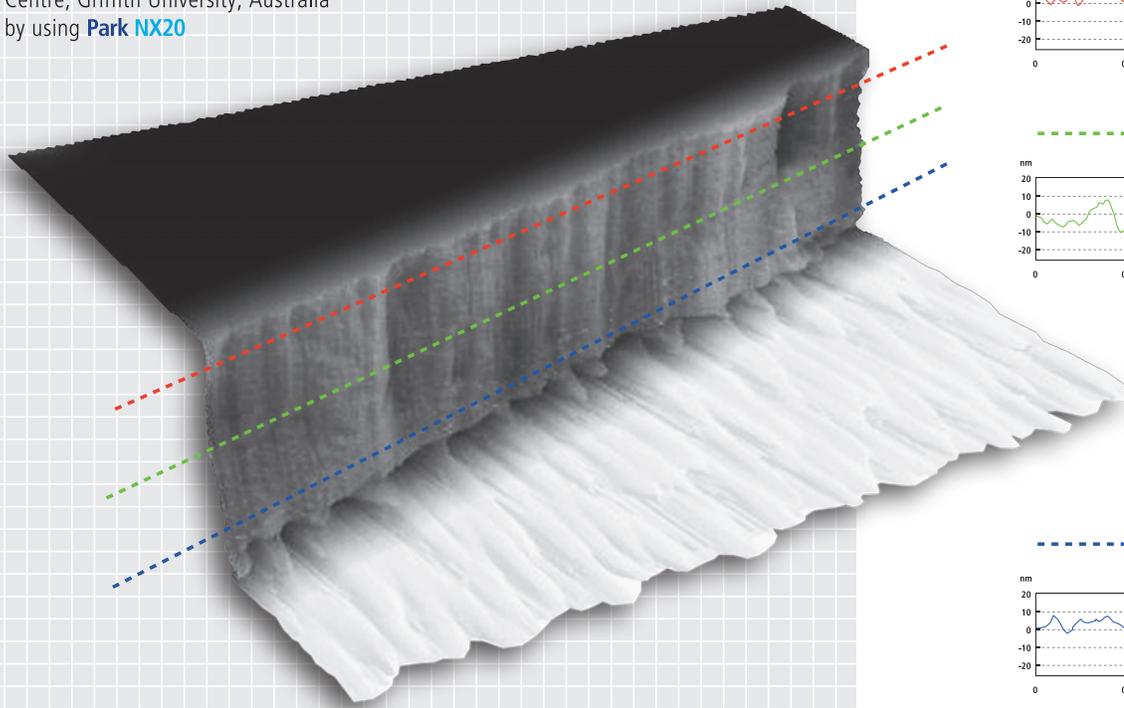
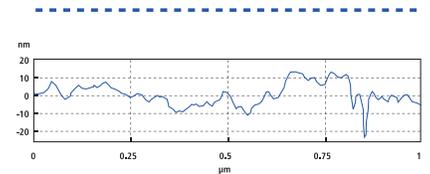


Figure 4. EFM amplitude (top-left), EFM phase (top-right), p-epi ($2 \times 10^{16} \text{ cm}^{-3}$), n well ($2 \times 10^{17} \text{ cm}^{-3}$), p-channel ($1 \times 10^{17} \text{ cm}^{-3}$), n+ contacts ($2 \times 10^{20} \text{ cm}^{-3}$).

Etched Sidewall of a Silicon Carbide (SiC) film
The Queensland Micro and Nanotechnology
Centre, Griffith University, Australia
by using **Park NX20**



LINE PROFILES OF SIDEWALL



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Park
SYSTEMS

HAIR DAMAGE FROM SUNLIGHT RADIATION

CHARACTERIZED USING ATOMIC FORCE MICROSCOPY

By Alvin J. Lee, Monta Vista High School, Cupertino, California

INTRODUCTION

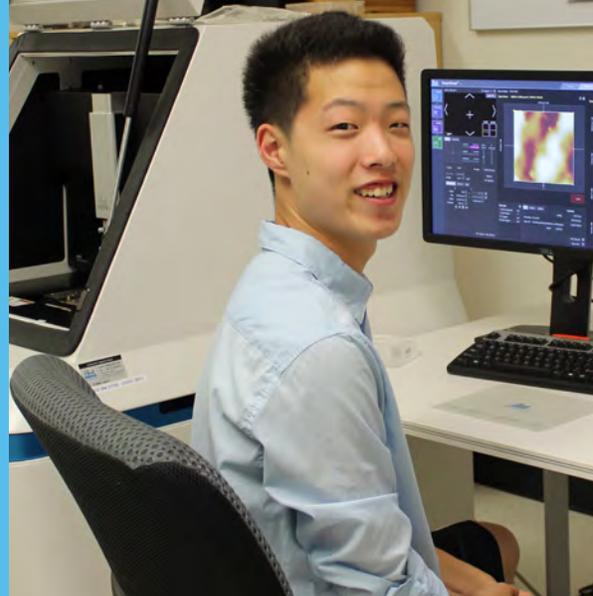
This study's objective is to observe the effects of long term exposure of sunlight on hair fiber. Many fear the damaging effects of prolonged sunlight exposure on skin, yet few stop to think about the implications of such damage on hair. Black hair is known to be more resistant to sunlight damage than blonde hair[1] due to black hair containing the photo-unstable pigment pheomelanin in levels only a fifth of what is found in blonde hair[2]. This key difference suggests that solutions to mitigate the damage of prolonged sunlight exposure on hair will need to vary based on the target population's hair color. Using a Park NX20 AFM system, the damage done by sunlight radiation on black and blonde hair will be compared by means of comparing surface roughness values (given as Root Mean Square roughness, Rq) before and after receiving a standardized dose of extended sunlight exposure.

Because blonde hair contains five times more pheomelanin (a substance known for its low photostability) than black hair, it is expected that more free radicals will be produced in blonde hair. These free radicals are chemically reactive particles created in the presence of radiation. They effectively destroy the hair cuticle by reacting with nearby stable molecules and eroding the hair's natural structure[3]. Hair after prolonged exposure to sunlight is deduced to be rougher due to what is expected to be an uneven formation of craters and pockets caused by free radical wear. Consequently, changes from this radiation should also be visible in the cross section as we expect that the cuticle layers will thin out.

EXPERIMENTAL

Using the Park NX20 AFM, nanoscale images are able to be produced by tracing the topography of the scan site with a probe. The AFM probe will move up and down based on the topography of the sample and using the captured probe movement data, the Z position at each point on the sample can be translated into an image. The AFM fits the scope of the research for several important reasons. The most commonly used instrument for looking at hair on a very small scale is the scanning electron microscope (SEM). The problem with use of an SEM instrument is its requirement for a conductive surface. This sample preparation is not only tedious and expensive, but it also alters the sample and its composition. Furthermore, the AFM is also able to track many of the sample's mechanical properties such as roughness—one of the key values that will be used in the analysis.

Virgin straight hair from the author and virgin straight blonde hair from a true blonde 24-year old female volunteer are imaged under the Park NX20 to examine their natural topographies. The samples are mounted onto a piece of carbon tape to aid both the accurate landing of a noncontact (such as PPP_NCHR or OMCL-AC160TS) AFM probe tip onto the surface of the hair as well as the increase the stability of the sample as it is scanned. To maintain a consistent tip-sample landing location, landmark features of the area near the hair are observed and selected, and the areas of carbon tape near the hair were pushed in using tweezers under the optical microscope for positional accuracy. All operations of the Park NX20 were facilitated with Park SmartScan, the



Park High School Summer Researcher, Alvin Lee in front of the AFM he used in his research on Hair Damage from Sunlight Radiation. Lee, who had never used an Atomic Force Microscope before was able to operate Park NX 20 easily, creating the first AFM images of human hair.

tool's operation software. Non-Contact mode was used throughout all AFM operations.

To apply a treatment of prolonged sunlight exposure, hair samples, hereafter referred to by their color ("black" and "blonde") are left out in direct sunlight in a petri dish for a total of 42 hours, ranging from 6 to 8 hours of exposure at a time. During the hours of exposure, the UV index is recorded to quantitatively assess the intensity of the sunlight. Over the course of 42 hours, the average UV index was 7.6. For a cross-sectioning of the hairs to analyze possible internal damage, black and blonde samples from before and after the sunlight exposure treatment were cross-sectioned. The hairs were laid out, lined up laterally, and embedded in an epoxy to be cured. The cured epoxy is then turned upright so that the tips of the hair pointed upwards (see Figures 4a-d). The top of this setup was then ground down and polished with silica film, lowering the roughness to 0.04 μm .

RESULTS AND DISCUSSIONS

Surface Roughness. To compare roughness in Rq, the 5 μm by 5 μm images were flattened once in the X direction and once in the Y direction based on which linear regression fit the peaks the best. This is to avoid any macroscopic contour. These 5 μm by 5 μm regions are found by taking a larger image to get a complete view of the entire hair sample and then choosing a region in which there are no visible steps or scales in the hair (see Figure 1a). For sunlight-treated hair, three 5 μm by 5 μm regions were taken. The bottom 5 μm by 2 μm area of the selected region is then used to find the roughness (see Figure 1a for

WITH PARK SMARTSCAN OPERATING SYSTEM ATOMIC FORCE MICROSCOPE IS AVAILABLE TO A BROADER USER-MARKET
Smartscan, designed to produce quality images autonomously with a single click is standard on all Park AFM products making it possible for all users to harness the power of AFM

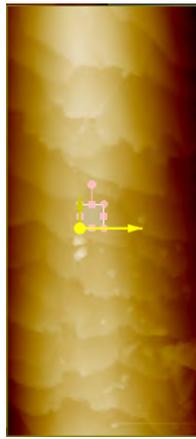


Figure 1a - Example of selecting a $5\mu\text{m} \times 5\mu\text{m}$ region on a sample hair for surface roughness analysis. Above is a $40 \times 100 \mu\text{m}$ AFM topography image of black hair after 42 hours of sunlight radiation. A small region on the surface, free of any step or scale edges, has been chosen for roughness measurement.

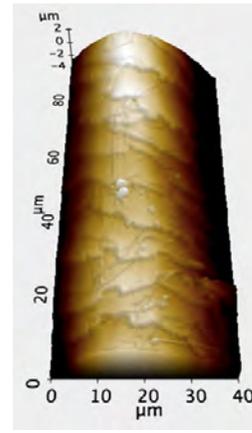


Figure 1b - 3D AFM topography image of the sample in Figure 1a. Because the AFM is able to process Z-directional data by moving the tip up and down, as well as track from side-to-side, such images as above can be produced. 3D renderings of the AFM topography were invaluable in confirming the selected site for roughness measurement was free of step and scale edges.

example). After 42 hours of sunlight radiation, surface roughness decreased in both black and blonde. The images shown are the non-flattened scans displayed with a high-contrast color filter.

All topography images in Figure 2 have peaks colored orange due to the high-contrast filter. Seen above, the Rq values actually decreased after sunlight exposure occurred, contrary to our hypothesis. The difference of the average untreated hair Rq and the average treated Rq is 0.5 nm. The image of the untreated sample (Figure 2a) contains visible bumps that were most likely eroded from due to free radical damage and flattened out until it became similar to the surfaces in Figures 2b-d. ΔRq of the untreated blonde hair and of treated blonde hair is, on average, 2.1 nm. This is nearly four times that of black hair. Based solely on the visual qualities of the topography seen in each image of Figure 3, it is evident that the roughness would indeed decrease. While blonde hair does have many bumps on the surface after the 42 hours of irradiation, the pre-treatment sample (Figure 3a) had large peaks and craters on the surface which are not observed in its post-treatment counterparts.

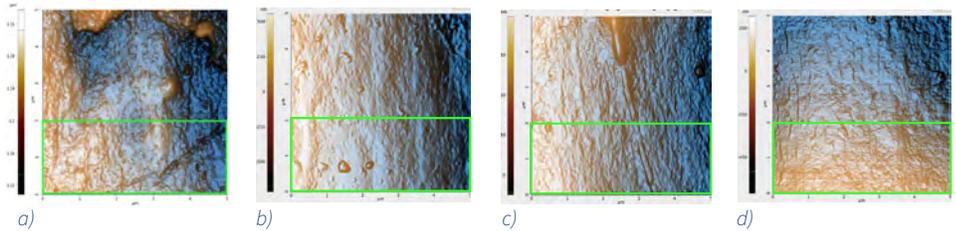


Figure 2 - AFM topography images of the black hair samples: (a) before any prolonged exposure to sunlight; Rq: 4.5 nm, (b) first sample after 42 hours of sunlight, Rq: 4.1 nm, (c) second sample after 42 hours of sunlight, Rq: 4.9 nm, and (d) third sample after 42 hours of sunlight, Rq: 3.1 nm. The green box in Figure 2a is the region in which the Rq value was calculated for that sample; regions of identical dimensions and position were used to calculate the Rq values of the other samples.

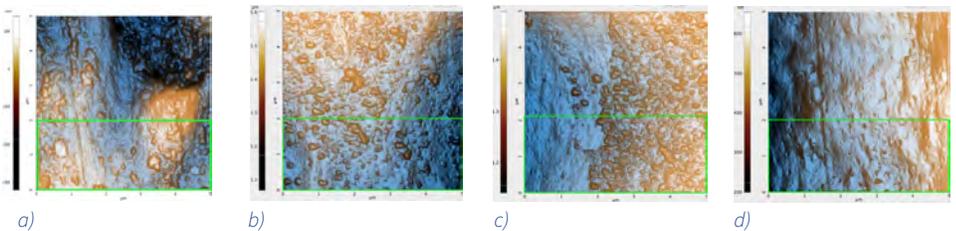


Figure 3 - AFM topography images of the blonde hair samples: (a) before any prolonged exposure to sunlight, Rq: 8.2 nm, (b) first sample after 42 hours of sunlight, Rq: 7.4 nm, (c) second sample after 42 hours of sunlight, Rq: 7.0 nm, and (d) third sample after 42 hours of sunlight, Rq: 4.0 nm. The green box in Figure 3a is the region in which the Rq value was calculated for that sample; regions of identical dimensions and position were used to calculate the Rq values of the other samples.

Cross Sections: By examining the width of the cuticle, approximately how much of the hair was worn out after prolonged exposure to sunlight can be determined. Theoretically, because of the radiation-induced free radicals that form on the outermost layer of our hair, we should see our samples' cuticles decrease in size. Both topography and amplitude data were recorded when scanning the cross section with AFM. Amplitude imaging was used primarily to help produce high-contrast visuals to make the cuticle region more visible and identifiable for analysis.

According to our cross section results, it appears that both black and blonde cuticles

got smaller after the sunlight treatment: black decreased by $0.1\mu\text{m}$ while blonde decreased by $1.5\mu\text{m}$. Knowing that blonde is less photostable than black hair and will therefore be more susceptible to UV damage, this data supports the idea that increased pheomelanin levels in blonde hair correlate to increased photodamage as compared to their black counterparts.

CONCLUSION

It appears that because both types of hair had their surface roughness values lowered as a function of time when exposed to sunlight, prolonged exposure to sunlight affects both dark and fair-colored hair. The

form of this damage did not come in the hypothesized form of increased roughness however, as both the surface roughness and cross section analysis suggest that sunlight radiation actually smoothens the surface of hair decreasing the width of the cuticle in a more uniform manner than expected. It also appears that the ΔRq for blonde hair is greater than that of black hair despite being exposed for the same amount and intensity of sunlight. This suggests black hair degrades at a slower rate than its blonde counterpart does, an effect that is supported by previous research into pheomelanin levels in these types of hair. Because the cuticle is the layer meant to receive the most wear, it would make sense

that structurally, the hair will become weaker as the cuticle thins out. Studies show that tensile strength (breaking resistance when pulled) decreases after hair has been exposed to certain UV treatments [4]. Other mechanical properties that would most likely change due to prolonged exposure to sunlight are ductility and friction. Frictional force would likely decrease due to the roughness of hair decreasing after exposure. It would seem that hardness would not change much due to the cuticle still remaining after exposure, but it may change due to the chemically reactive species (free radicals) making new byproducts that are different in hardness than the proteins that make up hair.

Based on the findings, hair damage is not much of a high priority concern for black haired individuals, as the cuticle only decreased in width by $0.1\mu\text{m}$, a distance almost negligible as it is less than 4% of the original width. Blonde hair, on the other hand, may need much more tending to when under a large amount of exposure. Over the course of 42 hours, the blonde hair cuticle decreased in width by more than half its original width ($1.5\mu\text{m}$ down from $2.6\mu\text{m}$), meaning in less than 84 hours, there would be no cuticle left to protect the surface of the hair. To confirm, the initial pre-treatment cuticle thickness of our samples are in line with reports using much larger sample sizes [5]. The relationship of pheomelanin to this phenomenon, if any, could not be clearly identified within the scope of this study. With pheomelanin known to be housed in the hair shaft's cortex rather than in the exterior cuticle, any free radical-caused damage would have to travel outward through the hair shaft to affect the cuticle layer. The effect of this speculated free radical travel was not visually detected in either the topography or amplitude images of the post-treatment sample cross sections. It is quite possible that the high levels of cuticle degradation in blonde hair may be due to another unidentified structural factor. Keeping only the observable cuticle damage in mind, the mechanical properties of blonde hair will have been negatively affected. For example, sun-damaged blonde hair may have also affected the keratin bundles in the shaft's cortex, making the hair much easier to snap due to a decrease in tensile strength. There are, however, products on the market such as keratin-based shampoos that have been promoted as being essential in repairing damage from sunlight. The veracity such claims by existing products were beyond the scope of this study, but may be a fruitful topic to explore in the future.

Finally, a more controlled environment may be better for testing property differences in hair. For example, had a UV lamp been used rather than sunlight so that one could quantitatively assess how much radiation went into the hair, it would be easier to show the changes in hair as a function of time exposed to "x" amount of radiation. Because the UV index was specifically designed for skin damage when it was first created, it may not have been the best form of measurement to assess UV exposure, but other tools for recording sunlight intensity were not available at the time of experimentation. Such considerations would be well placed in follow-up studies on this very topic.

ACKNOWLEDGMENTS

Thank you to Park Systems, Dr. Byong Kim, Dr. Mina Hong, and Gerald Pascual for making this research possible.

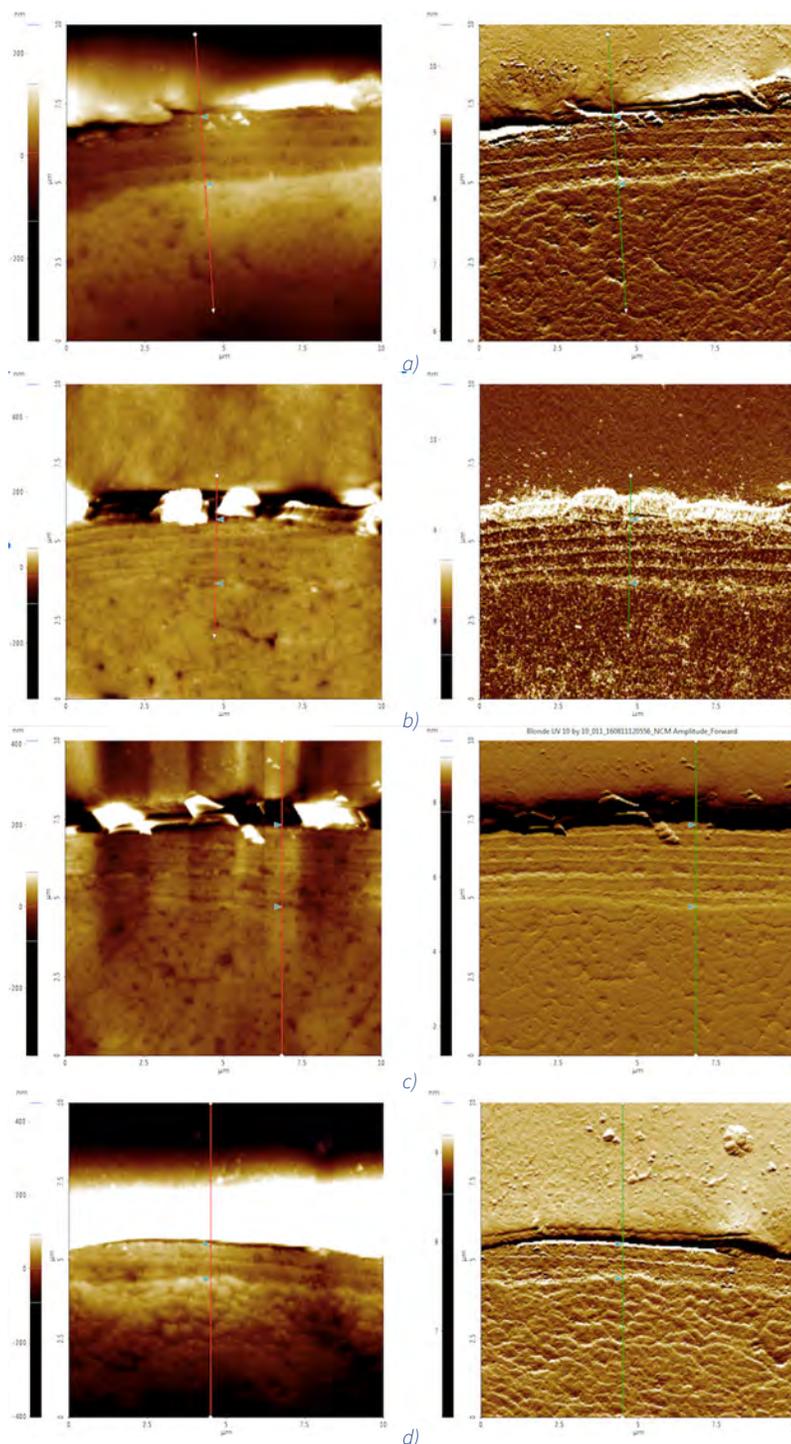


Figure 4 – AFM topography (left) and amplitude (right) images of cross sectioned hair samples: (a) black before 42 hours of sunlight exposure, cuticle width = $2.1\mu\text{m}$, (b) black after 42 hours of sunlight exposure, cuticle width = $2.0\mu\text{m}$, (c) blonde before 42 hours of sunlight exposure, cuticle width = $2.6\mu\text{m}$, and (d) blonde after 42 hours of sunlight exposure, cuticle width = $1.1\mu\text{m}$. The cuticle width is estimated by measuring the distance between the two triangular bluish color markers shown in the each amplitude images.

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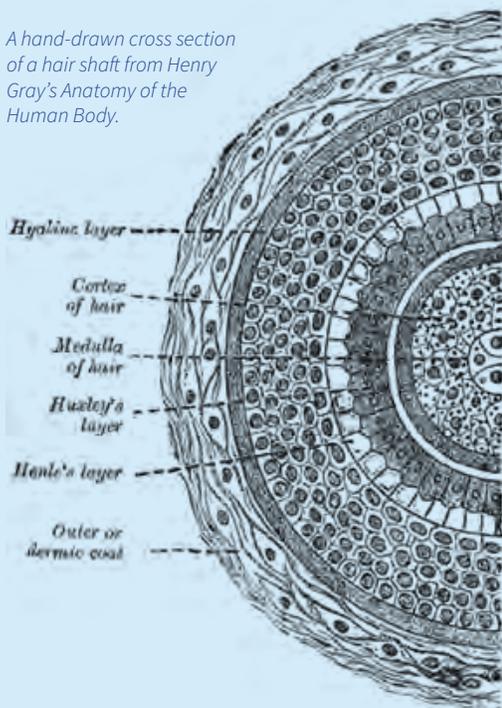
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APPENDIX A THE STRUCTURE OF THE HUMAN HAIR SHAFT

A hand-drawn cross section of a hair shaft from Henry Gray's Anatomy of the Human Body. Each strand of hair on your body is composed of layers of structures that each have specific functions. The outermost layer, the cuticle, is the main focus of this study. It is formed from sheets of thin dead cells that overlap one another, producing scale-like structures that can be seen with a microscope. The cuticle provides protection for the interior of the hair's shaft and is even coated with lipids to help repel water. Contrary to popular belief, the cuticle does not give hair its color; in fact, it is transparent and exposes the next structural layer underneath.

The color of one's hair is actually determined in this next layer, the cortex. This region is the most highly organized area of the hair shaft containing both the keratin bundles which give hair its mechanical strength as well as melanin pigments. The number, distribution, and types of melanin in the cortex are what determine the hair's color. Pheomelanin, the substance hypothesized to be the critical factor causing the difference in damage levels in this study's samples, along with eumelanin are the two types of melanin found in hair.

A hand-drawn cross section of a hair shaft from Henry Gray's Anatomy of the Human Body.



The innermost structure in the hair shaft is the medulla. This region is perhaps the least understood of the entire shaft, but can be likened to the pith of a fruit skin or bone marrow. Mitochondrial DNA can be extracted in large amounts from the medulla—a process useful for some types of forensic investigation.

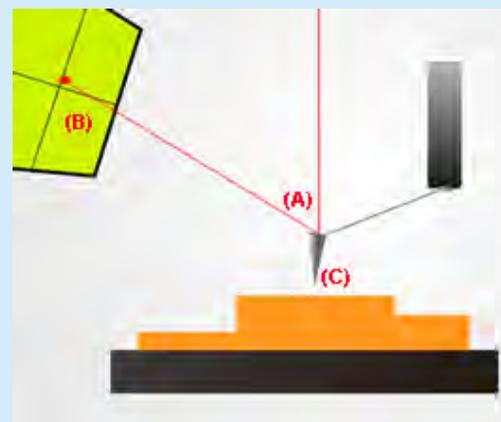
APPENDIX B BASIC MODES OF OPERATION IN AFM

Atomic force microscopes (AFM) are tools that can gather topographical information by running a mechanical probe across the surface of a target sample. This mechanical probe consists of a spring-like cantilever which has a sharp tip fixed to its free end. AFM systems can be run in one of several basic modes of operation:

(1) Contact mode. In this mode, the probe is brought into physical contact with the sample and is gently traced across laterally line-by-line until the entire sample has been scanned. Over the course of the scan, the probe's vertical motion as it rises with peaks, sinks into dips, and twists around features on the sample's surface are recorded. The lateral and vertical movements of probe are combined by software to produce an image of nanoscale features at the scan site.

(2) Non-contact mode. This was the mode used in this hair study. As its name suggests, the probe's cantilever is made to oscillate at a designated reference amplitude just above the sample surface. The probe tip avoids making direct physical contact with the sample as it scans. As the probe tip approaches the sample surface, the amplitude of the cantilever's oscillation decreases. The system's built-in feedback loop detects this change and lifts the probe such that the cantilever oscillation returns to its original reference amplitude. These precise vertical movements are then combined with other recorded lateral movements to render an image of the scanned site.

ATOMIC FORCE MICROSCOPE (AFM) TECHNICAL ARTICLE



A basic setup for non-contact mode atomic force microscopy. Note the deflected red laser beam (A) shining back up into a sensor (B). This allows the system to keep track of changes in the probe's (C) position. This vertical information along with recorded lateral movements are then combined by software to render an image of the sample's topography.

Along with non-contact mode, another AFM term used in this study was amplitude imaging. An AFM amplitude image is a 2D display of the error signal obtained when using non-contact mode. It shows the degree at which the cantilever's oscillation amplitude deviated from its original reference amplitude at single points in time. This is helpful in visualizing certain topographical features with dramatic slope changes such as steep drops and sharp climbs (places where amplitude can change dramatically in a short amount of time), but is less useful in imaging existing features with more modest, steady changes in slope like shallow depressions (places where amplitude changes take place over large amounts of time).

(3) Tapping mode, is also used by some AFM users. Usually considered an alternative to non-contact mode, this technique also oscillates the cantilever above a sample surface but at much higher amplitudes. While capable of producing usable images, the tip deterioration rate is accelerated—a problem to keep in mind as blunted tips produce lower resolution images and need to be replaced for optimal results.

SHOWCASING AFM IMAGES

GLIMPSES OF THE NANOSCALE

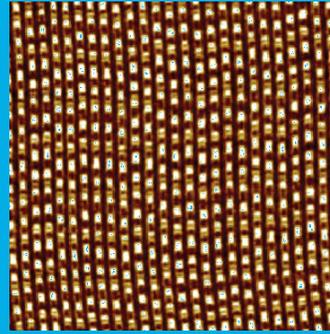
WITH DIMENSIONS SMALLER THAN THE WAVELENGTHS OF VISIBLE LIGHT, NANOSCALE STRUCTURES ARE VIRTUALLY INVISIBLE—EXCEPT WHEN OBSERVED WITH THE POWER OF ATOMIC FORCE MICROSCOPY (AFM)

By Park Systems Staff

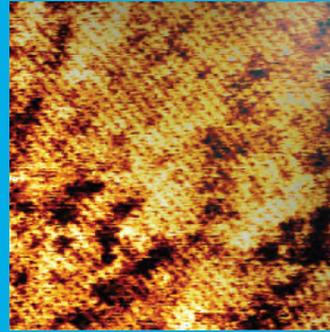
In our ongoing quest to understand and master the world around us, researchers from various fields have turned to looking at the world at nanometer resolution. Investigations at these dimensions can no longer be performed with diffraction-limited tools such as optical microscopes. As a result, AFM and other Scanning Probe Microscopy techniques have become very attractive options to determine not just the topology of a sample, but also to explore various other properties as well.

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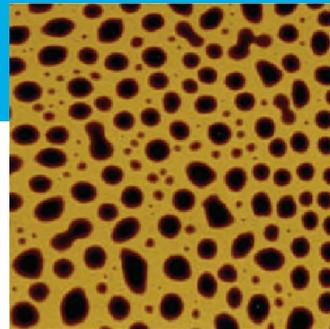
SPM techniques such as Scanning Ion Conductance Microscopy and AFM have been used to investigate biological matter such as cellular membranes under in-situ, liquid conditions. AFM has also been instrumental in novel polymer development such as the furthering of our knowledge of lubrication performance in tribology studies as well as the fabrication of higher-performance photoresist materials for wafer production. Inroads have also been made in the hard disk drive sector with the myriad nanoscale magnetic traits of a disk slider now openly characterized and ready for optimization by design engineers. Similar stories can be found again in the semiconductor industry—Scanning Capacitance Microscopy (SCM) is an extremely effective technique to verify the dopant concentration in new device designs. Dopant concentration is a materials property that cannot be gauged using topography data and can only be measured using SCM.



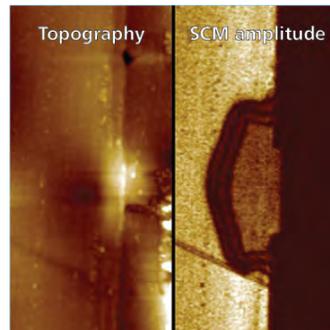
High-resolution magnetic property image of hard disk drive media
Scan area: $5 \times 5 \mu\text{m}$
Probe: PPP-MFMR
Technique: Magnetic Force Microscopy (MFM)



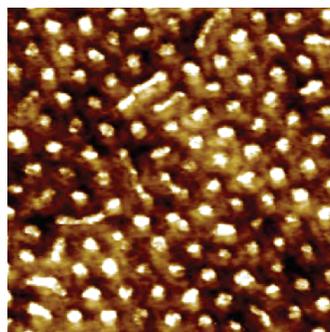
Topography of a bacterial S-layer membrane in liquid
Scan area: $500 \times 500 \text{ nm}$
Technique: Atomic Force Microscopy (AFM)



Phase image of lubricant on a plastic board
Scan area: $30 \times 30 \mu\text{m}$
Technique: Atomic Force Microscopy (AFM)



Topography of a FET structure vs. its dopant structure; the brightness in the SCM amplitude image correlates to dopant concentration in this part of the device
Technique: Scanning Capacitance Microscopy (SCM)



Topography of a block copolymer
Scan area: $0.5 \times 0.5 \mu\text{m}$
Probe: SSS-NCHR
Technique: Atomic Force Microscopy (AFM)

TEXTILE NANO-CHARACTERIZATION: TOPOGRAPHY, PHASE IMAGING, AND NANOMECHANICAL-PROPERTY INVESTIGATION OF POLYESTER YARN INTERACTION WITH SILICON MATRIX

By Gerald Pascual, Byong Kim, Mina Hong, John Paul Pineda, and Keibock Lee
Technical Marketing, Park Systems Inc., Santa Clara, CA, USA

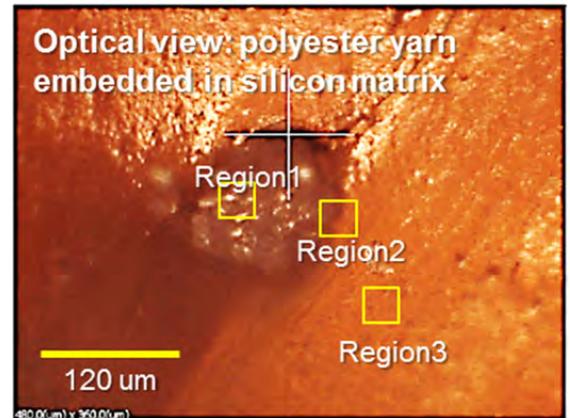


Figure 1: Cross-section of Silicon gel matrix with embedded strand of polyester yarn. Three regions were selected for further investigation: Region 1 on the yarn itself, Region 2 at the interfacial region between the yarn and matrix, and Region 3 on the matrix surrounding the yarn.

ABSTRACT

Textiles research and development is rapidly turning to nanomaterials to create new fabric blends that have increased performance for traits such as damage resistance, breathability, and even self-cleaning. To better inform materials design strategies, it is necessary to have a tool and techniques capable of measuring not only nanoscale topographies of material components, but their nanomechanical properties as well. Atomic Force Microscopy (AFM) is a solution well-suited to explore and characterize these traits. To this end, a silicon gel matrix and polyester yarn sample was prepared for examination with a commercial AFM system, the Park NX10 from Park Systems. Non-contact mode AFM from Park Systems was used to perform topography and phase imaging. Force-distance spectroscopy plus force-volume mapping was used for nanomechanical property characterization. The acquired data reveals that the hardness of the yarn is about 100 times greater than the matrix it is embedded in with forces being measured in nanonewton resolution and distances in micrometers. This investigation of the textiles sample is reflective of AFM's effectiveness in allowing textiles researchers to explore the root, nanoscale causes of desirable macroscopic traits in novel fabric blends and further improve upon them.

INTRODUCTION

Nanotechnology plays an exciting role in the creation of new fabrics for novel purposes. To better integrate nanomaterials into existing

fabrics, it is very important to understand the characteristics of these new additions. For example, knowing that particular nanofibers have a high threshold for tearing would suggest that weaving them into a fabric already known to be tear-resistant would further improve that trait without adding much in weight. This is a nanoscale analogue to what has been done to create tough, but light fiber reinforced plastics such as fiberglass. Atomic Force Microscopy (AFM) can be used to study the interactions between the fibers and the fabric matrix they will be inserted into to make new textiles. Analysis of the topography of these next-generation textiles as well as their various nanomechanical properties is well within AFM's capabilities and can provide knowledge helpful in adjusting textile composition to maximize performance gain and minimize production cost.

EXPERIMENTAL

A silicon matrix sample embedded with strands of polyester yarn was provided by Prof. Yan Vivian Li at Colorado State University [1]. The AFM experiment was conducted using that sample under ambient air conditions using a commercial AFM system, the Park NX10 from Park Systems [2], in non-contact mode for topography imaging and phase imaging. Both sets of data are acquired at the same time; topography imaging is useful for observing three-dimensional features on the surface of the sample while phase imaging yields data that can be correlated to the sample's elasticity. The nanomechanical properties of the sample were

characterized using force-distance spectroscopy and force-volume mapping. These results were then correlated to data acquired by phase imaging. In force-distance spectroscopy, force-distance (f-d) curves are used to measure the force that an AFM probe applies vertically to a single point on a sample surface. F-d curves are plots of the cantilever's deflection, as measured by a position-sensitive photodetector, versus the extension of a piezoelectric scanner [3]. Force-volume mapping builds on force-distance spectroscopy in that it takes an array of single measurement points and turns the collected f-d curves across the sample surface into a 2D characterization map of hardness [3]. Hardness is defined as the slope of the f-d curve given in the units of N/m. Lastly, phase imaging is an AFM technique that makes use of the shift in a cantilever's oscillation as its tip moves across different features on the sample. The difference in the input signal for a tip's oscillation versus the ensuing oscillation's output signal is referred to as a shift in phase [4]. This particular signal can be correlated to several material properties such as elasticity.

RESULTS & DISCUSSIONS

A cross-section view of the silicon matrix (pink in color) is shown in Figure 1, which was taken with an optical microscopy built in to the Park NX10 AFM system, with a strand of polyester yarn (dark circular feature in center, 100-150 μm in diameter). Three regions were selected for investigation. Region 1 is on the exposed strand of yarn. Region 2 is in the interface between the yarn

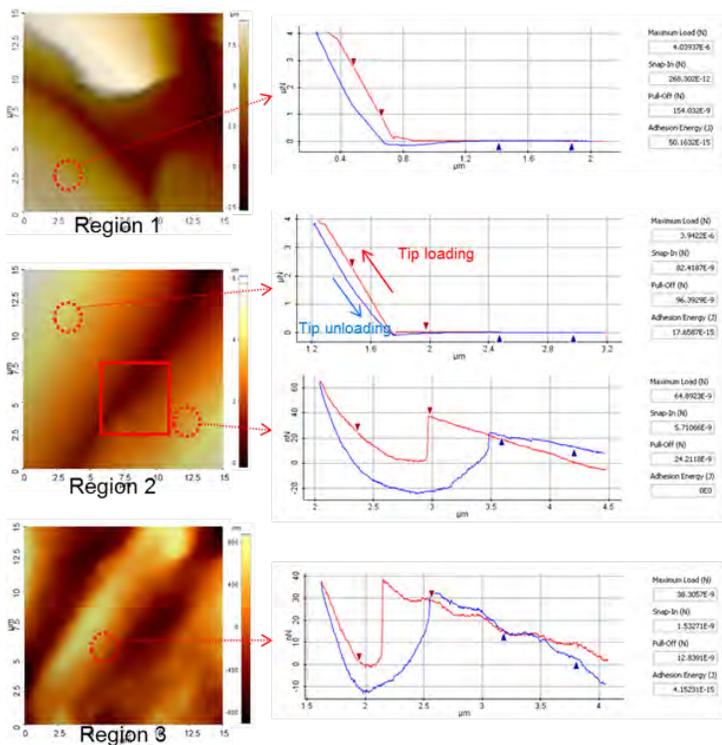


Figure 2. Non-contact AFM topography images with corresponding f-d curves of selected sites inside each of the three regions pictured in Figure 1.

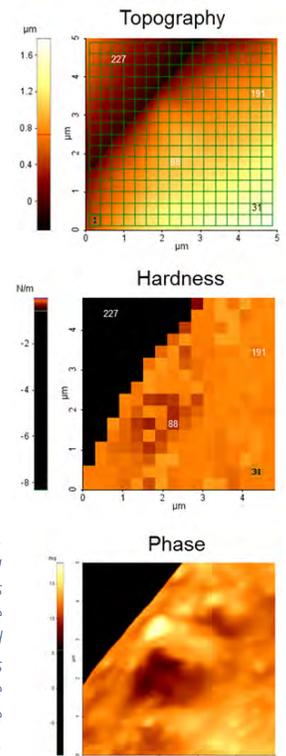


Figure 3. Force-volume mapping array of the sample's interfacial region and the hardness map generated from it. The hardness data correlates to the phase interfacial region.

and the silicon matrix surrounding it. Region 3 is in an area composed entirely of the matrix.

Figure 2 contains non-contact AFM topography images taken from each of the regions selected in Figure 1 as well as f-d curves that were measured at selected sites within each of those regions. The shape of the f-d curves correlate to a measure of the physical interaction between the tip and sample; in this case, the tip-sample distance vs. the force load on the tip cantilever. The slope of an f-d curve is steeper when the tip presses on a harder sample.

In Region 1, a location on the sample's yarn, a site in its lower-left corner was selected for force-distance spectroscopy. The f-d curve at this site reveals the force applied by the tip to the sample increases by about $4 \mu\text{N}$ over a distance of about $0.5 \mu\text{m}$ as the tip pushes down onto the surface. In Region 2, a pair of sites was measured at the sample's interfacial region: the first site is in the left half of the image (the area composed of yarn) and a second site in the right half of the image (an area composed of the matrix). The f-d curve for the yarn side of the region again exhibits a force of about $4 \mu\text{N}$ being applied by the tip to the sample over a distance of about $0.5 \mu\text{m}$. The f-d curve at the site corresponding to the matrix portion of the region yields different data. An initial increase of just under 40 nN in the force applied by the tip to the sample occurs over a distance of approximately $1.25 \mu\text{m}$. At which point a steep drop-off of about 40 nN in the tip-applied force is observed. It is possible that the

drop-off in applied force is due to the tip being pulled onto the surface of the silicon matrix. A second increase in tip-applied force immediately afterward as the probe continues pressing onto the matrix. This second increase in force is measured to be about 50 nN over a distance of $0.5 \mu\text{m}$. In Region 3, a location on the sample's silicon matrix, we observe a two-stage force load increase similar to the one exhibited by the matrix half of the previously investigated interfacial region. The initial tip-applied force builds up to about 40 nN over a distance of $1.5 \mu\text{m}$ just before sharply decreasing to about 0 nN . Again, this sudden decrease in applied force is speculated to be the moment in which the tip has snapped onto the surface of the silicon matrix. Shortly thereafter, a second increase in tip-applied force is again observed as the probe continues to be pushed down onto the matrix resulting in a load increase of about 40 nN over a distance of about $0.5 \mu\text{m}$. In this investigation, f-d curves taken from a region within a strand of yarn show a force load on the tip's cantilever that is approximately 100 times greater than the load shown on an f-d curve from the silicon matrix at the similar tip-sample distances.

In order to increase the scope of the study from single points to whole sections of the sample's surface, force-volume mapping was applied as depicted in Figure 3. This technique allows us to create a 2D map of the sample's nanomechanical properties—in this case, hardness. To begin, a small area within Region 2 as shown in Figures 1 and 2 was selected for hardness mapping

as the interfacial region of the sample would allow us to investigate both the yarn and matrix simultaneously. This location is indicated by the inset red square in the non-contact AFM topography image for Region 2 shown in Figure 2. The location for force-volume mapping measures $5 \times 5 \mu\text{m}$ and is depicted in all of the images making up Figure 3. The next step was to overlay a 16×16 grid over the location to be mapped thus creating an array of 256 total sites. F-d curves were measured at the midpoint of each of these sites and the collected data were translated into a 256 pixel hardness map where each pixel represents the sample hardness detected at each site's midpoint. Note the sharp difference in the colors of the hardness map's pixels which closely follows the interfacial region's border between the yarn and the matrix as shown in Figure 3's topography image. A similar phenomenon is observed with the phase image as well—the upper-left portion of the phase image corresponds to the yarn and has a profoundly different phase signal than the remaining portion of the image depicting the matrix. This indicates that the phase shifts in the cantilever's oscillation are markedly different when the probe moves across the yarn versus across the matrix—suggesting a difference in material elasticity.

The final leg of the investigation repeated the f-d curve comparison that was depicted in Figure 2; however, this time the sites selected for analysis were exclusive to the interfacial region of the sample focused on in Figure 3. A total of 4 sites were selected from the array of 256

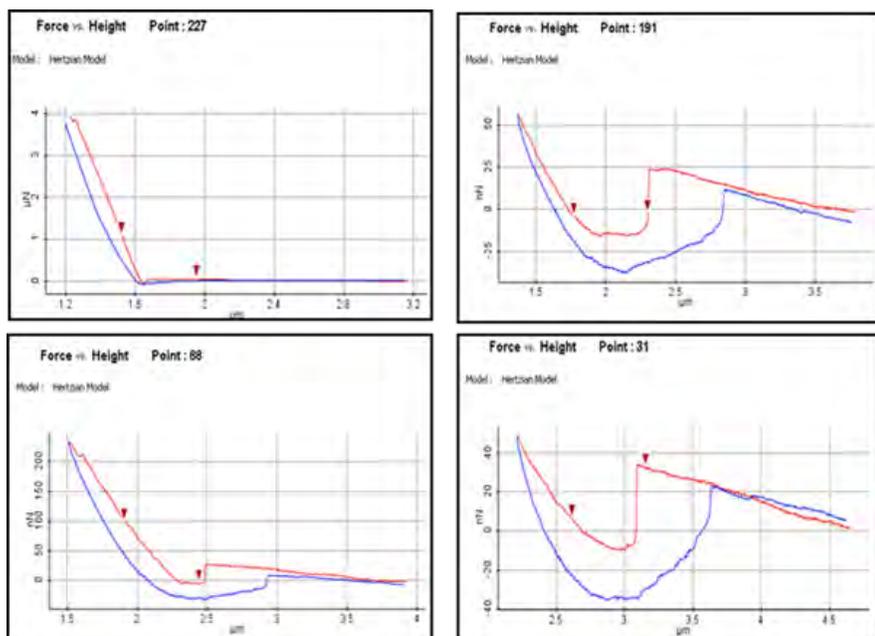


Figure 4.
F-d curves of sites 31, 88, 191, and 227 from the sample's interfacial region hardness map as shown on Figure 3.



The AFM experiment was conducted using using a commercial AFM system, the Park NX10 from Park Systems

superimposed on the topography image in Figure 3 and are referred to hereafter as sites 31, 88, 191, and 227. The first three sites are located within the matrix area of the interfacial zone whereas site 227 is located on the yarn. The f-d curves for each of the four sites are shown in Figure 4. The f-d curve of site 227 is shaped as expected given our experience in measuring Region 1 (yarn) on Figure 2. Again, a force load of about 4 μN over a distance of about 0.5 μm is observed. Sites 31, 88, and 191 also have f-d curves that were also anticipated by having previously measured Region 3 (matrix) on Figure 2. These three sites all have the same force load increase marked by a steep drop-off in tip-applied force as the probe snaps onto the matrix. The ensuing second force load increase is again measured to be around 40 nN over a distance of 0.5 μm . This 2D mapping data is consistent with the single-point force-distance spectroscopy data acquired earlier in the investigation.

Given that the polyester yarn has been observed here to be around 100 times harder than the gel matrix it is embedded in, it is reasonable to propose that the gel's original resistance to certain types of damage may have been positively augmented by the embedded yarn. The nanomechanical property focused on during this investigation was hardness. The hardness of a material is generally correlated to its elasticity, plasticity, and/or its resistance to fracture. In fiber reinforced plastics, embedded fibers allow a novel composite to remain in one piece despite having a large, shattering force applied to it. The composite also has

potentially significant weight savings when compared to the material it is replacing. When applied to textiles and apparel, nanofibers such as the yarn investigated here can be embedded in more than just gel matrices and have been woven into existing fabrics such as cotton to confer traits such as increased aerosol filtration [5] or the ability to self-clean [6]. A nanofiber specifically selected for its hardness may conceivably increase a composite fabric's damage resistance leading to immediate uses in ballistics as well as other applications that require clothing with heightened durability with acceptable or even negligible increases in weight.

SUMMARY

The topography image, phase image, and a nanomechanical property map (based on f-d curve data) of a silicon gel matrix and polyester yarn sample were all created using the Park NX10 from Park Systems, a commercially available AFM system [2]. The data collected in this study suggests the yarn is approximately 100 times harder than the matrix it is embedded in. All data acquisition was performed with forces being measured on the order of nanonewtons across distances as small as micrometers. Performing such high-precision measurements demonstrates AFM's ability to characterize key material properties at nanoscale. This is especially important to understand the interactions of components in novel composites such as next-generation textiles which are now being designed with

nanomaterials in mind. Investigations such as the one conducted here can help researchers comprehend the source of macroscopic effects blending materials may manifest starting at the smallest of scales. This knowledge can in turn inform subsequent strategies to design future iterations with increasing performance and decreasing cost.

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PARK SYSTEMS, WORLD LEADING MANUFACTURER OF ATOMIC FORCE MICROSCOPES RECEIVES KOSDAQ'S THE BEST NEXT-GENERATION COMPANY PRIZE

Park Systems was awarded **The Best-Next Generation Company Prize** from the Korea Exchange trading board KOSDAQ during the 8th KOSDAQ Awards Ceremony held June 23, 2016. Park Systems was selected from more than a thousand publicly traded corporations in KOSDAQ by the committee comprised of MOTIE (Ministry of Trade, Industry and Energy), the Financial Supervisory Board, Korea Exchange Board, and the related academia members to receive the award.

Park Systems was selected for their long history as a leader in atomic force microscopy, providing continuous nanoscale advances in both technology and products and for their solid business performance. In December of 2015, Park Systems became a publicly traded company on KOSDAQ and received "AA" from two separate rating agencies on advanced technologies, becoming the first company listed on the KOSDAQ in 2015 through the special technologies IPO program.

"Park Systems is honored to be selected from such a long list of excellent companies to receive this prestigious award as The Best Next-Generation Company by KOSDAQ," stated Dr. Sang-il Park, CEO and founder of Park Systems who worked as an integral part of the group at Stanford University that first developed AFM technology and created the first commercial AFM in 1988. "Our mission is to pursue continuous innovations for AFM technology as we take the next quantum leap forward in scientific discovery."

In 2015 Park Systems formed a partnership with IMEC, a worldwide nano-electronics research center, for the next generation of nano-instrumentation for inline atomic force microscopy technology development. This partnership extends to a wide-ranging customer base from the global semiconductor consortium members and major institutions in the future.

About Park Systems

Park Systems was founded in 1997 and holds 32 patents related to AFM technology, including True Non-Contact Mode™ using decoupled XY and Z scanners, PTR measurements of HDD application, NX-Bio technology using Scanning ion conductance microscopy (SICM) on live cell, 3D AFM, and fully automated AFM operation software (SmartScan™). Park Systems's major customers include thousands of prestigious universities and international research agencies world-wide.



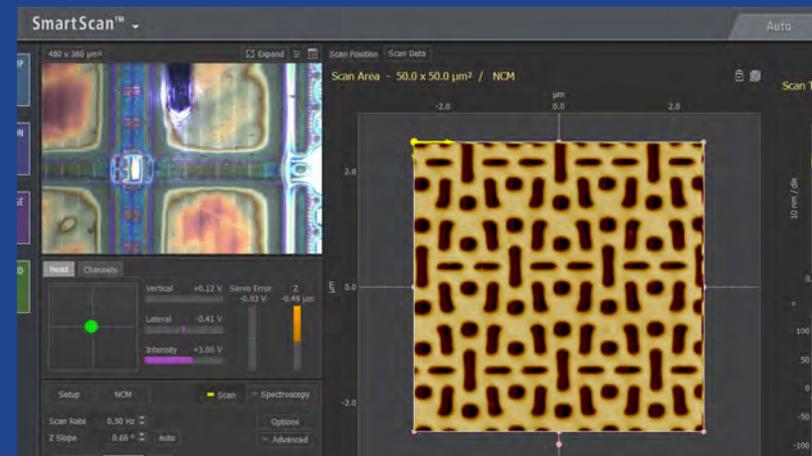
"PARK SYSTEMS IS HONORED TO BE SELECTED FROM SUCH A LONG LIST OF EXCELLENT COMPANIES TO RECEIVE THIS PRESTIGIOUS AWARD AS THE BEST NEXT-GENERATION COMPANY BY KOSDAQ"

"OUR MISSION IS TO PURSUE CONTINUOUS INNOVATIONS FOR AFM TECHNOLOGY AS WE TAKE THE NEXT QUANTUM LEAP FORWARD IN SCIENTIFIC DISCOVERY!"

**DR. SANG-IL PARK, CEO AND
FOUNDER OF PARK SYSTEMS**



Dr. Sang-il Park (right), CEO and Founder Park Systems accepts The Best Next-Generation Prize at the 8th KOSDAQ Awards Ceremony held June 23, 2016



PARK SYSTEMS, WORLD LEADING MANUFACTURER OF ATOMIC FORCE MICROSCOPES RECEIVES FROST & SULLIVAN 2016 GLOBAL ENABLING TECHNOLOGY LEADERSHIP AWARD

Based on thorough recent analysis of the global atomic force microscopy (AFM) market, Frost & Sullivan awarded Park Systems with the **2016 Global Enabling Technology Leadership Award**, for their ability to create AFM equipment and services that offer unparalleled measurement accuracy, repeatability, and reliability, exceeding any of the competitive products on the market. Frost & Sullivan Best Practices awards recognize companies in a variety of regional and global markets for demonstrating outstanding achievement and superior performance in areas such as leadership, technological innovation, customer service, and strategic product development. Industry analysts compare market participants and measure performance through in-depth interviews, analysis, and extensive secondary research to identify best practices in the industry. Park's Atomic Force Microscopes demonstrate cutting edge technology where unmatched performance resulted in customized product lines used at leading manufacturers world-wide. "One of the key differentiators for Park Systems is their proprietary SmartScan™ technology that allows any researcher to make high resolution accurate images without AFM expertise, and often five times faster for a comparable quality image than done by an expert," said Frost & Sullivan Industry Analyst Mariano Kimbara. "The company's diverse and powerful range of AFM products highlights their zeal to innovate and embrace the challenge to continuously extend the capabilities of nanoscale microscopy in a landscape that is rapidly evolving."

Since 1997, Park Systems has added significant innovations to their original AFM design to revolutionize imaging methodologies and enhance the user experience, resulting in their unbridled success. Park Systems holds 32 patents related to AFM technology, including

True Non-Contact Mode™ using decoupled XY and Z scanners, PTR measurements of HDD application, NX-Bio technology using Scanning ion conductance microscopy (SICM) on live cell, 3D AFM for sidewall measurements critical for latest semiconductor advancements, and automation AFM operation software (SmartScan™). SmartScan fully automatizes AFM imaging making it very easy for anyone to take an image of a sample at nanoscale resolution and clarity comparable to one taken by an expert. Park Systems has a full range of AFM systems that provide solutions for researchers and industry engineers across a wide spectrum of disciplines including chemistry, materials, physics, life sciences, semiconductor and data storage. Used by thousands of the most distinguished academic and research institutions worldwide, Park is recognized as an innovate partner in nanoscale technologies.

Park Systems's founder, Dr. Sang-il Park, was part of the original Stanford University-based team that developed the AFM technology in the early 1980s. By founding Park Systems, Dr. Park commercialized the first AFM, providing researchers and engineers with access to the exciting new technology. The company continues to develop and launch innovative AFM and AFM-related services to meet consistently new customer needs worldwide.

Park Systems has a distinguished customer service record, exemplified by the customer base of the world's leading institutions including thousands of prestigious universities and international research agencies world-wide. The company recently launched the market's first ever 9-to-9 Live Support, connecting customers with experts that can diagnose and fix problems from 9:00 a.m. to 9:00 p.m. Pacific time. Additionally, the

Park SmartScan™ for Atomic Force Microscope is standard on all Park AFM products. This revolutionary operating software auto performs all the necessary operations for imaging and intelligently decides on the optimum image quality and scan speed, all autonomously, saving the user time and money.



"PARK ATOMIC FORCE MICROSCOPES TRADEMARK DESIGN PRODUCES HIGH DATA ACCURACY AT NANOSCALE RESOLUTION, USING PATENTED TECHNOLOGY THAT DELIVERS ACCURATE AND COST-EFFECTIVE RESULTS," STATES KEIBOCK LEE, PRESIDENT PARK SYSTEMS. "WE ARE HONORED TO RECEIVE THE FROST & SULLIVAN AWARD FOR OUR ADVANCED TECHNOLOGICAL INNOVATIONS IN ATOMIC FORCE MICROSCOPY."

company provides a user forum, where customers can ask questions, respond to each other, and receive guidance from Park Systems' technicians and an interactive Nano Academy with a shared knowledge platform that includes webinars, a news magazine and user demonstration videos.

Each year, Frost & Sullivan presents this award to the company that has demonstrated uniqueness in developing and leveraging new technologies, which significantly impacts both the functionality and the customer value of new products and applications. The award lauds the high R&D spend towards innovation, its relevance to the industry, and the positive impact on brand perception.

Park SmartScan™

Bringing the power and versatility
of Atomic Force Microscopy to everyone

Park
SYSTEMS

www.parkAFM.com

Park SmartScan™ is a revolutionary operating software for Park AFMs that lets even inexperienced, untrained users produce high quality nanoscale imaging through **three simple clicks** of a mouse in auto mode, which rivals that made by experts using conventional techniques. SmartScan manual mode also provides all of the functions and tools necessary for more seasoned users to feel at home. This combination of extreme versatility, ease-of-use, and quality makes Park atomic force microscopes the most powerful and yet the easiest to use AFMs.



Park AFM Series
Enabling Nanoscale Advances



Park NX10



Park NX20



Park NX-Hivac



Park NX-Bio

For more information, email us at inquiry@parkafm.com or visit us at www.parkafm.com