

# NANOscientific

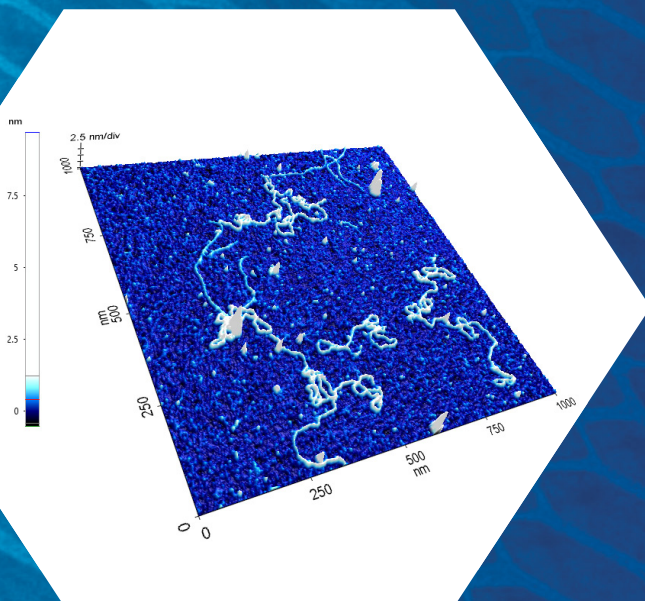
SUMMER 2017

The Magazine for Nanotechnology

## TECHNIQUES FOR STUDYING ATOMIC LEGOS AND BREAKTHROUGHS IN SUPERCONDUCTIVITY

PROF. JAMES HONE COLUMBIA UNIVERSITY

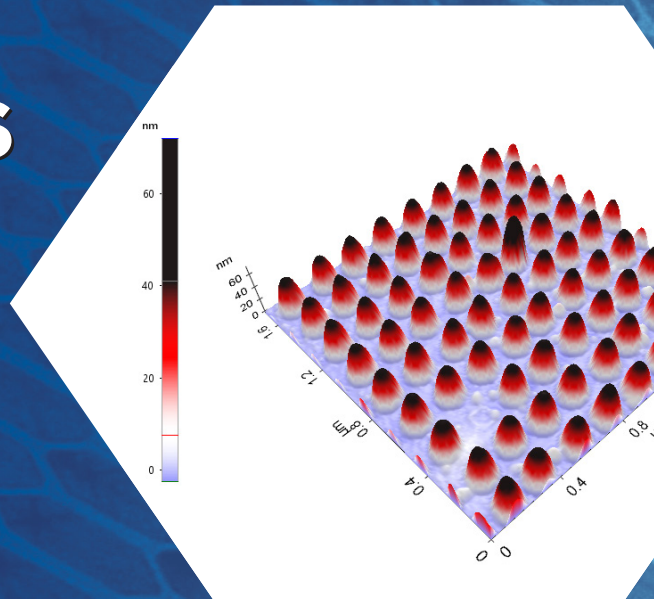
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## MEETING ENERGY NEEDS OF THE 21ST CENTURY BY IMAGING NANOPARTICLES

ELAD GROSS HEBREW UNIVERSITY OF JERUSALEM

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USING PARK ATOMIC FORCE MICROSCOPY

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## FAST IMAGING AFM

ACCELERATED METHODS TO ACQUIRE  
IN-SITU, REAL-TIME CRITICAL MATERIAL  
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The Most Accurate Atomic Force Microscope

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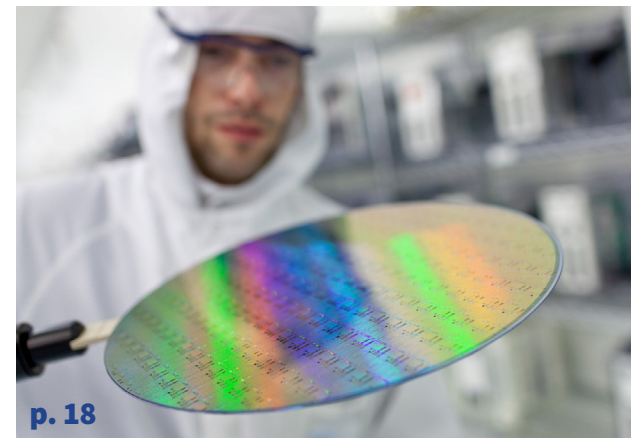
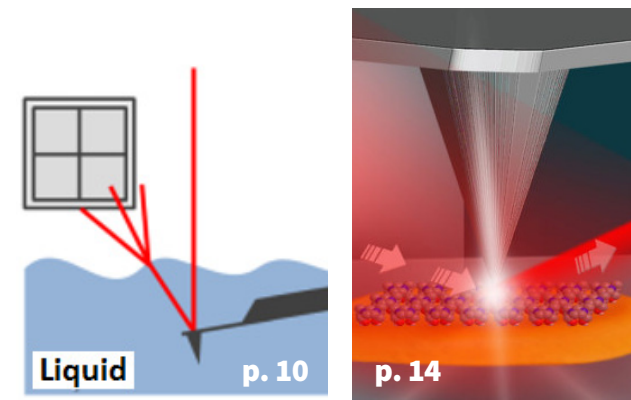
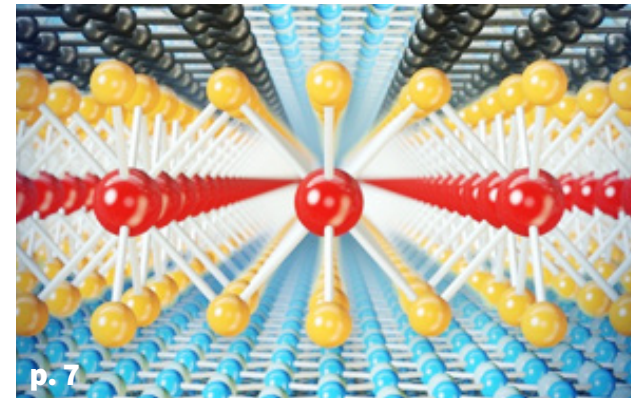


*Park*  
SYSTEMS

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## NANOscientific

The Magazine for Nanotechnology

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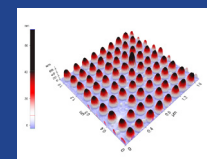
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## Park SYSTEMS

NANOscientific is published quarterly to showcase advancements in the field of Nanoscience and technology across a wide range of multidisciplinary areas of research.

The publication is offered free to anyone who works in the field of Nanotechnology, Nanoscience, Microscopy and other related fields of study and manufacturing.

For inquiries about submitting story ideas, please contact Deborah West, Content Editor at [debbie@nano-scientific.org](mailto:debbie@nano-scientific.org) for inquiries about Advertising in NANOscientific, please contact Gerald Pascal at [gerald@nano-scientific.org](mailto:gerald@nano-scientific.org)



Cover image: Figure 1 caption: DNA – Non-Contact Atomic Force Microscope topography image of plasmids acquired in liquid. The sample exhibits thinner segments with a linear structure and thicker segments exhibiting a supercoiled structure. Scan size: 1  $\mu\text{m}$  x 1  $\mu\text{m}$ .

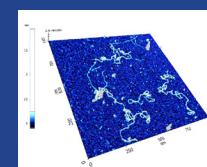


Figure 2 caption: Array – Atomic Force Microscope topography image of a magnetically patterned array. Note two features of interest on the sample which may be possible defects: 1) a missing switch in the image foreground and 2) a taller than normal switch in the image background.



Keibock Lee,  
Editor-in-Chief

## MESSAGE FROM EDITOR

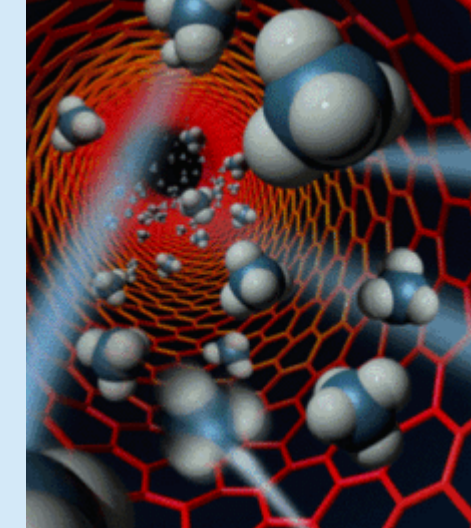
In today's global society, Nanotechnology is playing a crucial role in scientific advances on a massive world-wide scale. Scientific collaboration is at a record high with shared knowledge and scientific "think tanks" imaging, creating and transforming at record speed. The internet connects thoughts, ideas and inspiration globally. Nanoscience with all its potential for an amazing future is unfolding on this planet, shaping a scientific revolution crucial for emerging technologies that will change our world.



In this issue of Nano-Scientific, we have a special report on the work of Dr. James Hone, Professor of Mechanical Engineering at Columbia University where his team is developing novel breakthroughs in superconductivity using stacks of 2D materials. The interdisciplinary research team includes universities and research institutes in Japan, Korea, and Europe.

We also feature an article on DNA, one of the most significant discoveries of the last century, and explain how DNA molecules are imaged in liquid using Atomic Force Microscopy (AFM) tools from Park Systems. This article was written in collaboration with Park Systems and Jason Kahn the Department of Chemistry & Biochemistry, University of Maryland.

We have a Feature article about imec, the



world-leading R&D and innovation hub in nanoelectronics and digital technologies bringing together brilliant minds from all over the world for a sustainable future. This article includes an interview with imec Senior Researcher Dr. Tae-Gon Kim in Belgium about how in-line inspection using Park 3D AFM is providing solutions for semiconductor manufacturers.

This issue also includes a feature interview with Dr. Elad Gross, Senior Lecturer, Institute of Chemistry at the Hebrew University of Jerusalem and the work they are doing with



Lawrence Berkeley National Laboratory to uncover the full potential of heterogeneous catalysis to meet the energy-needs of the 21st century society.

We also report on Fast Imaging AFM, accelerated methods to acquire in-situ, real-time critical material surface topography and property change information at the nanoscale.

The Park AFM Scholarship awardees are featured in this issue, highlighting some of the research being done by the extraordinary young scientists across the globe committed to work together, using nanoscience to build a better world for all of us.

We hope you enjoy this issue and please send us your story ideas and share your nanotechnology news with us!

## A LIST OF GLOBAL NANOTECH ORGANIZATIONS

**The International Association of Nanotechnology** is a non-profit organization with the goals of fostering scientific research and business development in the area of Nanoscience and Nanotechnology for benefits of society. The Association fosters friendship, equality and cooperation amongst its members around the world. The Association conducts workforce training programs to equip a new generation of scientists, engineers and technicians working in nanotech and cleantech industries. Website: [www.nanotechnologyworld.org](http://www.nanotechnologyworld.org)

**The Nanotechnology World Association** provides its members with access to a vast network of more than 70,000 individuals and organizations who are leading the research, development, manufacturing and commercialization of nanotechnology worldwide. The association was created to help accelerate the integration of nanotechnologies in various industries -- such as medical, energy, electronics, transportation and materials -- by providing information, resources and tools, by connecting researchers and organizations, and by fostering knowledge sharing and cooperation. Promoting collaboration and networking is a part of the process towards sustainable success. Opening to new interdisciplinary approaches represents a new challenge for material scientists. This is particularly important in fields such as Nanoengineering, Nanoelectronics and Nanomedicine. Website: [www.ianano.org](http://www.ianano.org)

**The Nanotechnology Industries Association (NIA)** is the leading voice of the nanotechnology industries. On behalf of membership across Europe and around the world, we support the development of nanotech innovations that improve the lives of consumers, preserve our environment and advance our world.

NIA and our Members are committed to the safe, sustainable and beneficial use of nanotechnology and nanomaterials across all industries. We believe in fostering a better understanding of nanotechnology's important role in society and building a positive global environment for nanotech innovation. [www.nanotechia.org](http://www.nanotechia.org)



## FEATURE INTERVIEW

# TECHNIQUES FOR STUDYING ATOMIC LEGOS AND BREAKTHROUGH IN SUPERCONDUCTIVITY

## JAMES HONE

WANG FON-JEN PROFESSOR OF MECHANICAL ENGINEERING  
COLUMBIA UNIVERSITY

Dr. James Hone's group is an interdisciplinary research group focused on novel materials synthesis and device nano-fabrication. We use carbon nanotubes, graphene, self-assembled nanostructures, and textured substrates to study both fundamental properties and explore new applications in nano-electro-mechanical systems, biomechanical systems, nanoscale and molecular electronics and opto-electronics. The research has diverse applications in radio-frequency signal processing electronics, optical signal processing, energy generation, biological and molecular sensors, and immunology. The group is highly collaborative and works with research groups in Physics, Chemistry, Material Science, Electrical Engineering, and Mechanical Engineering.

Over the past 10 years, these Columbia researchers have performed pioneering work in the first such 2D material, graphene—an atomically thin form of carbon. In the past five years, the field has expanded to include a large number of other materials—insulators, semiconductors, metals, and exotic materials such as superconductors.

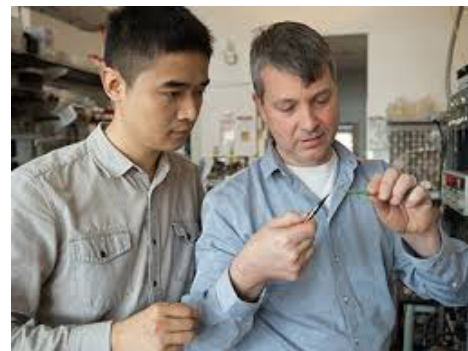
In 2013, Dr. Hone's group of researchers demonstrated for the first time that it was possible to electrically contact an atomically thin two-dimensional (2D) material only along its one-dimensional (1D) edge,

rather than contacting it from the top, which has been the conventional approach. Known as Atomic Building Blocks, techniques for stacking these Atomic Legos and improving the quality is part of the Hone Group's mission. "We work with many materials and techniques for handling and assembling 2D materials," states Hone. They look at graphene 2D material for example separated into single atomic layers and develop techniques to restack them into heterostructures. Research on graphene and other 2D atomic crystals is intense and likely to remain one of the hottest topics in condensed matter physics and materials science for many years.

The goal is to study single materials in the cleanest possible way for example encapsulated graphene in a cleanest possible environment. Then they can start combining them to get new functionality. Once the new functionality is achieved, they can create interfacial superconductivity by putting two layers together.

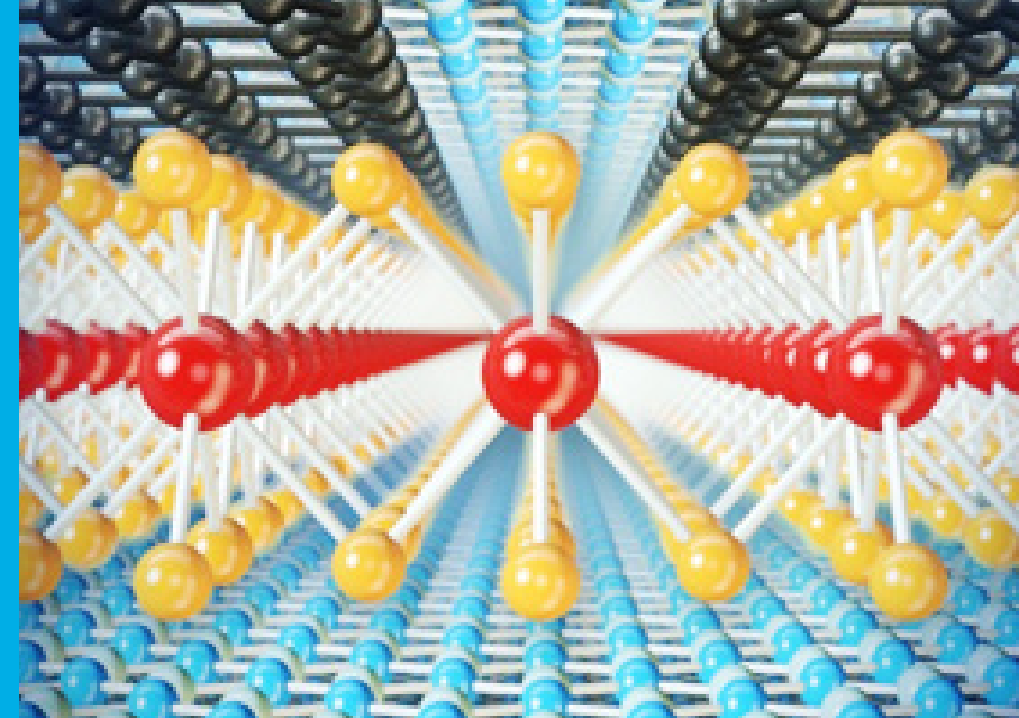
Dr. Hone's group has developed novel breakthroughs in superconductivity using stacks of 2D materials. One area is modulating light for optoelectric devices. The future area of study includes examining how much of what we do now with electrical signals can be done with light.

**"OUR RESEARCH FOCUSES ON BASIC UNDERSTANDING OF HOW TO ASSEMBLE THESE NANO BUILDING BLOCKS INTO MATERIALS AND STRUCTURES, AND WHAT PROPERTIES EMERGE WHEN WE DO SO. THIS UNDERSTANDING WILL ULTIMATELY LEAD TO CONCEPTUALLY IMPORTANT AND USEFUL NEW ELECTRONIC/ MAGNETIC DEVICES, OPTOELECTRONIC SYSTEMS, AND THERMOELECTRIC MATERIALS"** JAMES HONE, PAS3 DIRECTOR



**THE HONE GROUP AT COLUMBIA UNIVERSITY DEVELOPS TECHNIQUES TO "STACK" DIVERSE MATERIALS INTO LAYERED STRUCTURES THAT ARE ATOMICALLY PERFECT—DOING ON A DESKTOP WHAT COULD ONLY PREVIOUSLY BE DONE USING COMPLEX HIGH-VACUUM APPARATUS.**

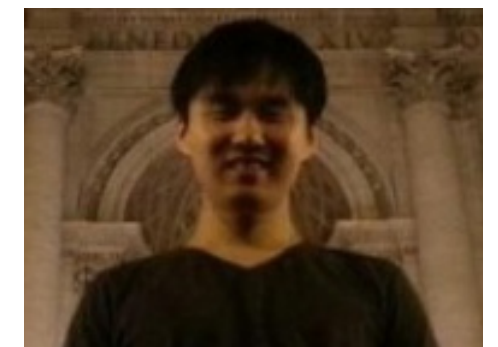
*Molybdenum disulfide encapsulated between layers of boron nitride*



**THE SHARED MATERIALS CHARACTERIZATION LABORATORY PROVIDES MICROSCOPY, SPECTROSCOPY AND DIFFRACTION INSTRUMENTS FOR MATERIALS RESEARCHERS IN CHEMISTRY, PHYSICS, AND ENGINEERING. THE FACILITY SPECIALIZES IN THE CHARACTERIZATION OF SURFACES, FILMS, MAGNETIC MATERIALS, LAYERED MATERIALS AND OTHER NANOSTRUCTURES, AND CRYSTALLINE/ POLYCRYSTALLINE MATERIALS.**

**The MRSEC provides crucial research funding to develop fundamental understanding of these new materials and realize their potential for application. It focuses on new materials with novel properties useful for magnetic memory; power conversion; and phase transitions that may be induced by optical, mechanical, thermal, and other stimuli and brings together researchers from a diverse background from many institutions.**

In 2015, Columbia University was awarded a \$15 million six-year grant from the National Science Foundation for a new Materials Research Science and Engineering Center (MRSEC) the Center for Precision Assembly of Superstratic and Superatomic Solids (PAS3) under the direction of Professor James Hone. Research partners include Brookhaven National Laboratory, IBM, and DuPont, as well as universities and research institutes in Japan, Korea, and Europe. The center comprises two interdisciplinary research groups (IRGs) focused on building higher-dimensional materials from lower-dimensional structures with unprecedented levels of control. Each IRG is targeted at demonstrating the promise of creating novel materials with new functionality and exceptional properties.



Sunwoo Lee, Ph.D. Student in the Hone Group at Columbia University is currently conducting a force-displacement test on the fabricated graphene accelerometers using an atomic force microscope (AFM) to validate their theoretical sensitivity.

# IMAGING OF PLASMIDS IN LIQUID USING TRUE NON-CONTACT MODE ATOMIC FORCE MICROSCOPY

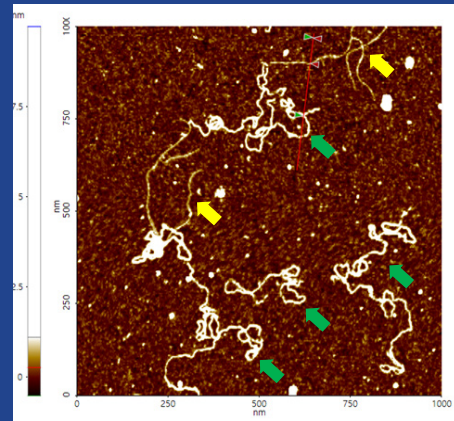


Figure 2. Non-contact mode AFM topography image of plasmids. The segments indicated with yellow arrows have linear structures whereas those with green arrows exhibit supercoiled structures. Scan size: 1 x 1  $\mu\text{m}$ .

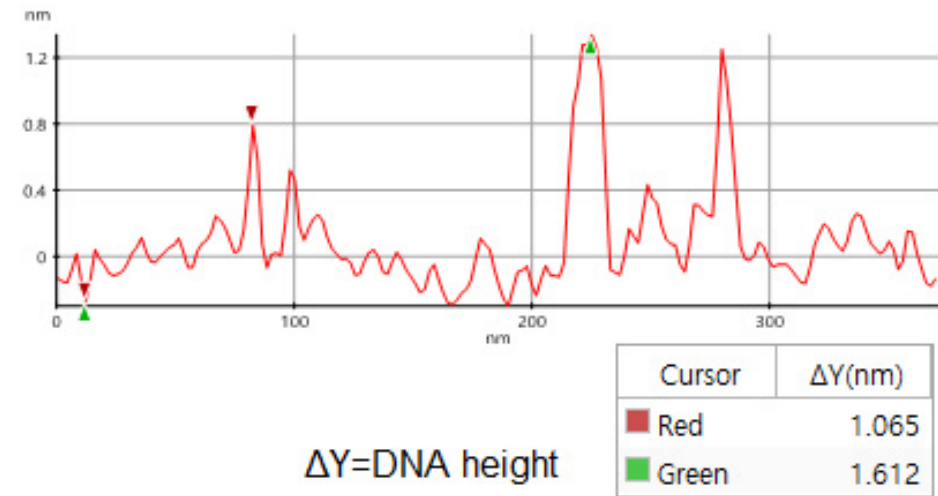


Figure 3. Line profile of non-contact mode AFM topography data taken from the sample shown in Figure 2. Red marker: smaller, linear DNA structure, green marker: larger, supercoiled DNA structure)

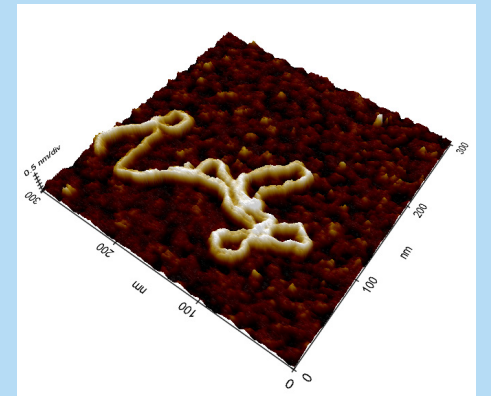


Figure caption: 3D view of high-resolution in-liquid AFM image of the individual supercoiled Plasmid DNA immobilized on Mica substrate acquired in the buffer solution.

**JOHN PAUL PINEDA, GERALD PASCUAL, CATHY LEE, BYONG KIM, AND KEIBOCK LEE**  
**PARK SYSTEMS INC., SANTA CLARA, CA, USA**

**JASON KAHN**  
**THE DEPARTMENT OF CHEMISTRY & BIOCHEMISTRY,**  
**UNIVERSITY OF MARYLAND, COLLEGE PARK, MD, USA**

## INTRODUCTION

Deoxyribonucleic acid (DNA) is one of the most significant discoveries of the last century, primarily because it revolutionized many fields including genetics, molecular biology, medical research, agriculture, forensic science, and many others. DNA is a biological molecule that acts as the storage of genetic information essential for bodily function, growth, and the reproduction of all living organisms. This information is located in the nucleus of every cell in an organism and can be transmitted not only from one cell to another but also

from parents to offspring. Hence, knowledge on the structure of DNA is very valuable in understanding how the transfer of genetic information occurs.

Several techniques have been used in studying the structure of DNA, one of which is electron microscopy (EM). However, several issues on sample preservation have arisen with this technique due to its tedious sample preparation requirements. As a result, the atomic force microscope (AFM) was developed to overcome such shortcomings.

**AFM's capability to image samples under in-liquid conditions preserves the condition of biological samples and provides higher quality high-resolution images [1].**

The True Non-Contact imaging mode of AFM tools from Park Systems allows for the measurement of sample topography without requiring a tip-sample interaction that can lead to sample damage and tip degradation. Thus, this mode is preferred when measuring samples that are sensitive to surface deformation rather than conventional contact and tapping modes. The advanced Z scanner design of a Park AFM is the key feature in achieving the non-contact imaging performance that defines Park's True Non-Contact mode: the highest imaging resolution and most accurate topographical data whilst maintaining tip sharpness and minimizing the chance of damaging the sample surface [2].

## EXPERIMENTAL

A 45 ng sample of plasmids (a type of small DNA molecule) was dispersed on a mica substrate and imaged with a Park NX10 AFM under in-liquid conditions using True Non-Contact mode. A cantilever with a relatively low nominal resonance frequency of 110 kHz and a nominal spring constant of 0.09 N/m was utilized in this experiment.

The non-contact AFM image is acquired by measuring the changes in the vibrational amplitude of the cantilever induced by the attractive van der Waals force as the cantilever is mechanically oscillated near its resonant frequency during the scan. The measured changes are compensated for by the AFM's feedback loop which maintains the cantilever's constant amplitude and distance. Non-contact mode measures the topography of the sample surface by using this feedback mechanism to control the Z scanner movement [2]. In liquid conditions, the cantilever's resonant frequency decreases by 1/3 of its original value in air due to dampening effects; a similar decrease is observed in its amplitude as well. In addition, multiple peaks can also be observed on a frequency sweep in liquid—fewer fluctuations are observed in air. Thus, the appropriate resonant frequency was chosen before the measurement procedure by selecting the highest peak near the region around 1/3 of the original resonant frequency and the amplitude was set to around 0.9 nm. The feedback system of the AFM uses a superluminescent diode (SLD) beam reflected from the cantilever. However, due to the instability of the liquid surface during measurement, the reflected SLD beam will scatter. For this reason, a liquid probehead with shielded glass was designed by Park Systems to avoid this problem. This type of probehead was utilized in the experiment.

## RESULTS & DISCUSSION

A high resolution image of the plasmid sample was successfully acquired and processed using XEI software developed by Park Systems. The sample was expected to consist of uniform linear plasmids. However, the topography data in Figure 2 revealed that the 1 by 1  $\mu\text{m}$  scanned region contains a mixture of linear and supercoiled plasmid DNA strands. The strands with larger structures are marked with green arrows and exhibit a supercoiled appearance with diameters of approximately 19 nm while the strands with smaller, linear structures are marked with yellow arrows and appeared to be in a relaxed state with diameters of approximately 8 nm. It was also observed that the supercoiled DNA strands appeared to be longer when compared to the linear strands.

**The line profile that generated by XEI in Figure 3 provides the height information of DNA strands. The data shows the supercoiled strands have a height of 1.6 nm whereas the height of the linear strand was only 1 nm.**

In its normal environment (cell nucleus), the DNA twist itself to overcome distortion as it is subjected to torsional stress during replication. A similar phenomenon could have happened in the linear DNA during this experiment. It is speculated that the linear DNA became supercoiled due to the torsional stress during sample preparation. The end of the plasmid possibly stuck to the substrate more tightly than expected, and as the DNA is subjected to torsional tension, the DNA twists around and bunches up on itself resulting to a larger and supercoiled structure with increasing orders of twisting [3].

## SUMMARY

The structure of a plasmid sample was successfully characterized by the Park NX10 AFM using True Non-Contact mode imaging under in-liquid conditions. The topography data revealed that the sample is characterized by the presence of DNA with two different superstructures: a linear configuration and a supercoiled one speculated to be due to torsional tension during sample preparation. One linear plasmid was revealed to have a width of 8 nm and a height of 1 nm. By comparison, one of the supercoiled plasmids had a width of 19 nm and a height of 1.6 nm. Overall, the technique described in this study will successfully provide researchers nanoscale information that is significant in monitoring the effects of structural damage and conformational change in DNA.

## REFERENCES

- [1] A. Baro, et al., Atomic Force Microscopy in Liquid: Biological Applications. Wiley-VCH, Pages 233-237.
- [2] J. Pineda, et al., True Non-Contact Imaging of Various Samples, Retrieved January 12, 2017, from <http://www.parkafm.com/index.php/medias/nano-academy/articles>
- [3] H. Hansma, et al., Reproducible Imaging and Dissection of Plasmid DNA Under Liquid with the Atomic Force Microscope, AAAS, Pages 1180-1181.

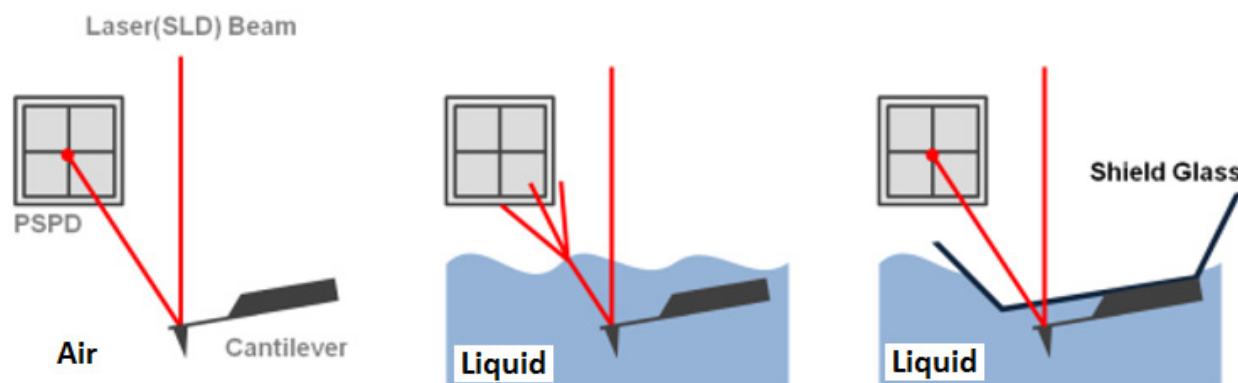


Figure 1. The feedback mechanism of True Non-Contact mode AFM in air and in liquid scan conditions. The shielded probehead in the rightmost image demonstrates its utility in maintaining optimal SLD beam and PSPD performance.

# IMEC REPORTS IN-LINE 3D AFM BY PARK SYSTEMS PROVIDES SOLUTIONS FOR SEMICONDUCTOR MANUFACTURERS

AN INTERVIEW WITH TAE-GON KIM, SENIOR  
RESEARCHER AT IN-LINE INSPECTION AND  
METROLOGY GROUP IMEC BELGIUM

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**IMEC IS THE WORLD-LEADING R&D AND INNOVATION HUB IN NANOELECTRONICS AND DIGITAL TECHNOLOGIES BRINGING TOGETHER BRILLIANT MINDS FROM ALL OVER THE WORLD IN A CREATIVE AND STIMULATING ENVIRONMENT ACCELERATING PROGRESS TOWARDS A CONNECTED, SUSTAINABLE FUTURE.**

**IMEC PERFORMS WORLD-LEADING RESEARCH IN NANO-ELECTRONICS AND CREATE GROUNDBREAKING INNOVATION IN APPLICATION DOMAINS SUCH AS HEALTHCARE, SMART CITIES AND MOBILITY, LOGISTICS AND MANUFACTURING, AND ENERGY.**

## ABOUT IMEC

Imec is the world-leading research and innovation hub in nanoelectronics and digital technologies. The combination of their widely acclaimed leadership in microchip technology and profound software and ICT expertise is what makes them unique. By leveraging their world-class infrastructure and local and global ecosystem of partners across a multitude of industries, they create groundbreaking innovation in application domains such as healthcare, smart cities and mobility, logistics and manufacturing, and energy. As a trusted partner for companies, start-ups and universities they bring together close to 3,500 brilliant minds from over 70 nationalities.

Imec is headquartered in Leuven, Belgium and also has distributed R&D groups at a number of Flemish universities, in the Netherlands, Taiwan, USA, China, and offices in India and Japan.

In 2015, Park systems signed a Joint Development Project (JDP) with imec, to develop in-line AFM metrology solutions of future technology nodes including but not limited to surface roughness, thickness, critical dimension (CD), and sidewall roughness. Together, Park and imec are exploring new frontiers of high resolution 3D AFM metrology to address accurate CD, line width

roughness (LWR), line edge roughness (LER) measurements, and sidewall roughness during etch, EPI, film deposition, and lithography processes. The imec and Park Systems partnership is developing new in-line monitoring and analysis methods for semiconductor manufacturers as well as new production protocol for better process development and control, which will result in improved device performance and production yield. The high resolution sidewall information on vertical planar and cylindrical structure by Park's new 3D AFM is having a huge impact on the performance of vertical devices such as FinFET, TFET, STT-MRAM and others.

## Please describe your background and the work you do at imec.

I studied material science and engineering and got a degree on surface preparation and modification in semiconductor. For topic, studying surface interaction between two species is a key topic to understand mechanism of contamination and removal. As semiconductor device scale is getting decrease industry requires small contaminants removal efficiently without minimize material loss and pattern damage. It requires me to measure particle removal force and pattern collapse force in quantitatively. For quantification of those force, particle adhesion and removal forces and pattern collapse force AFM is the only technique to provide the capability and it asked me to study and use AFM since 1999.

## How does imec work with Park Systems?

There are two ways of collaborations. Imec explores highly advanced semiconductor devices and use Park NX3DM at different device architectures to find a suitable solution for their process development and its monitoring. In order to find a best solution, imec and Park Systems develop the software and hardware together. We show the results to the imec partner. Second collaboration would be promoting the application results by publication and presentation at various conferences. For on-time result delivery an engineer from Park Systems works at imec to support all in-line AFM activities.

## How is in-line (3D) AFM a solution provider for semiconductor manufacturing processing?

Actually, I see many potential AFM applications in semiconductor manufacturing process. Many people doubt about AFM capabilities in semiconductor process due to their slow measurement speed and low throughput. Although the speed could not faster than other optic and electron based metrology technique, the value of result is greater than that of other metrology solutions surprisingly. Primary advantage of AFM in semiconductor is true atomic resolution topography measurement without sample damage which other metrology technique cannot. I just start to show some results at conference and the capability of AFM is promising. We are also working on improving the throughput and it should meet industry needs soon. My main role is bridging the semiconductor industry and AFM industry. One of my common questions is why more than 30 year old AFM technique was not used for semiconductor manufacturing. I found a possible answer while working with Park Systems. One of

biggest jobs is explaining semiconductor needs for AFM specialist by finding problems in semiconductor manufacturing in order to prove suitable metrology solutions for semiconductor engineers. There is a need for a paradigm shift and we are actively doing so.

## What features of Park AFM are most useful in semiconductor processing?

I truly emphasize that true non-contact measurement provides a strong advantage on atomic resolution and accuracy with no sample damage. Many imec integration engineers were very surprised at the capability of the Park AFM tool because they never saw such nice information and quality before. They rely on the measurement more and more because of the capabilities. Actually many people do not believe the advantage of non-contact measurement before the tool installation but afterwards they believe it. Long probe lifetime, which is result of non-contact measurement, is significant advantage in use of process monitoring. I think that Park AFM could only handle high density carbon (HDC) probe with 100 nm length and 10 nm diameter for more than 1000 die measurements. It is more than 2000 imaging using single probe.

## What are the latest trends advancing semiconductor wafer production currently?

The latest trend in semiconductor wafers is more and more complex and taller and taller. Device scaling will meet the limit, but the functionality of device increase so the semiconductor device processing trend will go the direction. Many other different materials will be introduced, but it might take a while to study them.

## How do you see semiconductor manufacturing changing in the future?

Well. I am not in the position to give a comment, but according to imec manufacturing environment and discussion with partner companies, machine learning and AI based on fab data might be a trend to improve manufacturing quality and efficiency.



Tae-Gon Kim, senior researcher at in-line inspection and metrology group imec Belgium

**"THE PARTNERSHIP BETWEEN PARK SYSTEMS AND IMEC PROVIDES A CRUCIAL LINK OF SCIENTIFIC COLLABORATION THROUGHOUT THE CHAIN OF SUPPLIERS AND VENDORS IN SEMICONDUCTOR WAFER PRODUCTION CREATING SIGNIFICANT TECHNOLOGICAL ADVANCES IN AFM-BASED INLINE NANOSCALE METROLOGY."  
DR. SANG-IL PARK,  
CEO OF PARK SYSTEMS**

“IMEC EXPLORES HIGHLY ADVANCED SEMICONDUCTOR DEVICES AND USE PARK NX3DM AT DIFFERENT DEVICE ARCHITECTURES TO FIND A SUITABLE SOLUTION FOR THEIR PROCESS DEVELOPMENT AND ITS MONITORING. IN ORDER TO FIND A BEST SOLUTION, IMEC AND PARK SYSTEMS DEVELOP THE SOFTWARE AND HARDWARE TOGETHER.”

TAE-GON KIM,  
SENIOR RESEARCHER AT IN-LINE INSPECTION AND METROLOGY GROUP IMEC BELGIUM



## IMEC TRANSFORMS BELGIUM'S CITY OF ANTWERP INTO LIVING LAB FOR THE INTERNET OF THINGS

In this new smart city, businesses, researchers, and local residents, will experiment with smart technologies that aim to make urban life more pleasant, enjoyable, and sustainable. Hundreds of smart sensors and wireless gateways positioned at carefully selected locations across streets and buildings will transform the city into a true living lab for the Internet of Things (IoT). The long-term objective is to connect thousands of Antwerp citizens with numerous innovative solutions that will considerably improve their quality

of life – by positively impacting mobility and public safety in the city, among other things.

The essence of a smart city is not that it is crammed full of new technology. First and foremost it is a city in which the quality of living is lifted to a new level, fulfilling the practical needs and expectations of the people who live there. To see how this could be realized the city council of Antwerp and a number of committed companies have set up a smart city project, making Antwerp one

of the world's largest living labs of smart city technology. In 2016, the foundations were laid of what may become a benchmark for urban environments worldwide.

The collaborative project will run from 2017 to 2019 and is designed to grow into the largest living lab in Europe for IoT applications. imec will leverage its expertise in sensor, wireless, and microchip technologies to help Antwerp target mobility, security, sustainability, and digital interaction as strategic priorities.

## IN-LINE 3D AFM FOR CRITICAL DIMENSION AND SIDEWALL ROUGHNESS OF SI PHOTONIC WAVEGUIDE AND CORRELATION WITH ITS PROPAGATION LOSS

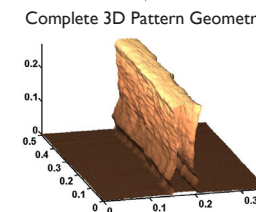
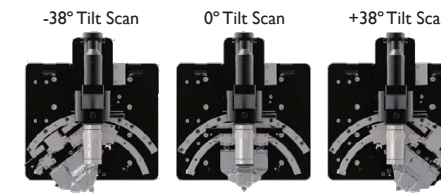
TAE-GON KIM\*, P.VERHEYEN, P. DE HEYN, T.VANDEWEYER, A. MILLER, M. PANTOUVAKI, J.VAN CAMPENHOUT (IMEC), A.-J. JO, S.-J. CHO, S.-I. PARK (PARK SYSTEMS)

imec

Park SYSTEMS

### 3D AFM Measurement Methodology

- Decoupled XY and Z scanner allows to tilt Z scanner head by  $\pm 19^\circ$  and  $\pm 38^\circ$
- Tilt Z scanner head allows probe to access waveguide sidewall
- Completed 3D geometry could be constructed by measuring 3 sides, top, left and right and stitching them together
  - Single sidewall could be characterized as well by single tilt scan



#### Measurable parameters

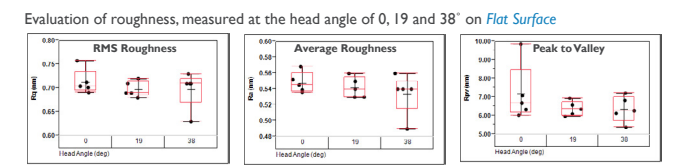
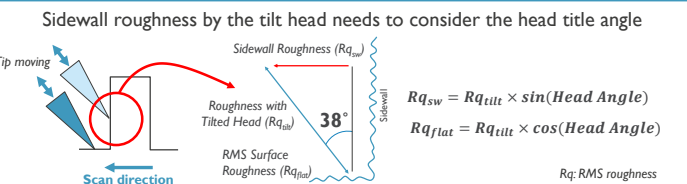
- Height
- CD/LER/LWR
- Sidewall Slope Angle
- Sidewall Roughness

### In-line 3D AFM at imec



- Tool: NX3DM, Park Systems
- 24/7 operational at 300 mm P-line
- Fully automated for 300 mm wafer
- Scanning System
  - True non-contact measurement
  - Minimum probe-sample damage
  - Long tip lifetime and good reliability
- Scanner Specification
  - XY scan area:  $100 \times 100 \mu\text{m}^2$
- Fully automated tool
  - Automatic Tip Exchanger
  - Fab Automation (SECS/GEM)

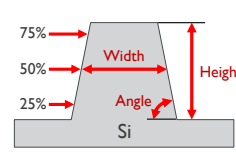
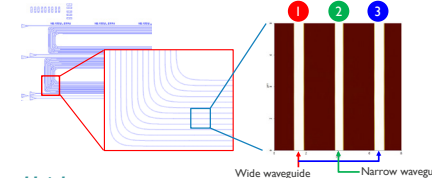
### Sidewall Roughness Characterization



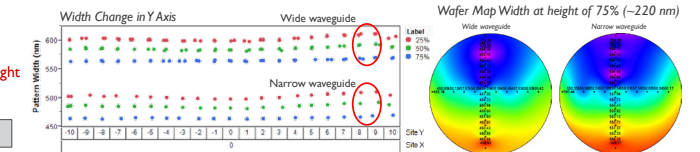
Tilted measurements show a good agreement with roughness value at 0°.

### Waveguide 3D Geometry and its Sidewall Roughness

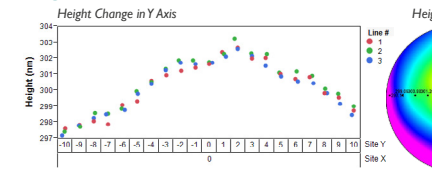
#### Experimental Structure



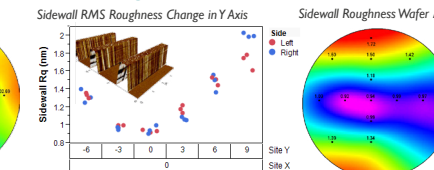
#### Waveguide Width



#### Height

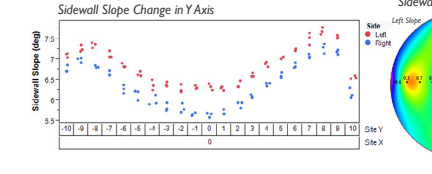


#### Sidewall Roughness

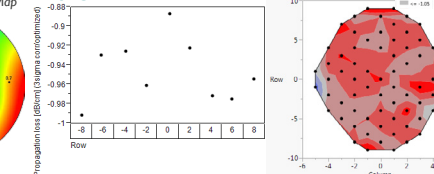


- Width of narrow and wide Si waveguide were captured accurately
- No significant change of waveguide width in Y axis was observed
- The height of Si waveguide at the center is higher than at the edge
- Clear sidewall slope change at different location was observed
- The sidewall slope offset between left and right sides might be caused by the offset of tilt angle of head, which could be minimized by calibration of tilt head angle
- Similar sidewall RMS roughnesses were measured on both left and right
- The sidewall slope offset does not impact on sidewall roughness value and the offset can be neglected
- Waveguide with rough sidewall shows propagation loss increase
- Larger waveguide shows lower impact on roughness and lower propagation loss characteristics

#### Sidewall Slope



#### Propagation Loss



**In-line 3D AFM could accurately measure 3D geometry of Si waveguide as well as its sidewall roughness. Not only sidewall roughness characteristic but also its dimension could correlate with propagation loss of their waveguide**

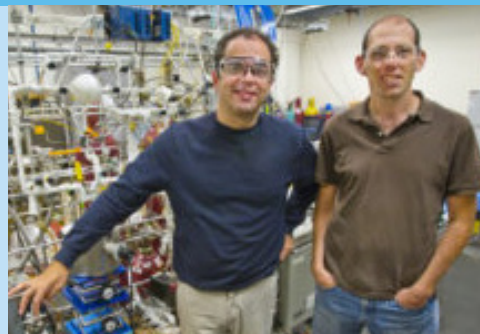
Above Poster Presentation from International Conference on Frontiers of Characterization and Metrology for Nanoelectronics (FCMN) March, 2017  
In-line 3D AFM for Critical Dimension and Sidewall Roughness of Si Photonic Waveguide and Correlation with Its Propagation Loss

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# MEETING ENERGY NEEDS OF THE 21ST CENTURY BY IMAGING NANOPARTICLES



## AN INTERVIEW WITH ELAD GROSS, SENIOR LECTURER, INSTITUTE OF CHEMISTRY HEBREW UNIVERSITY OF JERUSALEM



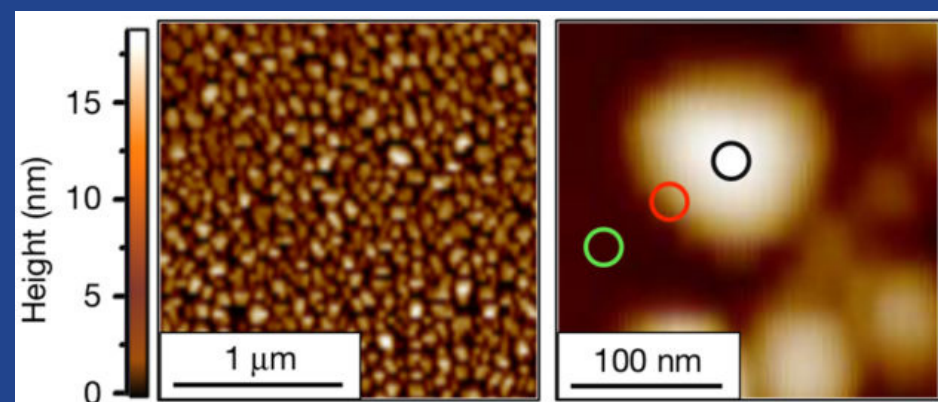
**“IN OUR EXPERIMENTS THE TIP OF THE ATOMIC FORCE MICROSCOPE IS USED AS AN ANTENNA THAT LOCALIZES AND ENHANCES THE INFRARED LIGHT.”**

Dean Toste, left, of Berkeley Lab and UC Berkeley, and Elad Gross, right, of the Hebrew University of Jerusalem, led a study of site-specific chemical reactivity on tiny platinum and gold particles at Berkeley Lab's Advanced Light Source. (Credit: Roy Kaltschmidt/Berkeley Lab)

Dr. Elad Gross completed his B.Sc. studies in Chemistry, in excellence, at the Hebrew University of Jerusalem, Israel. He then pursued his Ph.D. with Prof. Micha Asscher at the Hebrew University and a postdoctoral appointment with Prof. Gabor A. Somorjai at UC Berkeley.

Dr. Gross joined the faculty of the Hebrew University at 2015. Dr. Gross' research group focuses on understanding the basic elements that control and guide catalytic process by using cutting edge spectroscopy measurements.

His research focus is Heterogeneous catalysis, an essential technology for the formation of environmentally friendly, alternative feedstock. He has a multidisciplinary group aimed at elucidating the mechanisms that governs catalytic processes in order to prepare highly controllable catalysts suited for energy-needs of the 21st century society. To uncover the full potential of heterogeneous catalysis for energy applications, we employ a bottom-up approach in which we design the properties of catalysts in order to activate designated bonds within a reactant molecule. Structure-reactivity correlations within the catalytic systems are analyzed with advanced in-situ spectroscopy in order to unfold the dynamic processes that shape the catalytic reaction.



**Researchers combined a broad spectrum of infrared light, produced by Berkeley Lab's Advanced Light Source (ALS), with an atomic force microscope to reveal different levels of chemical reactivity at the edges of single platinum and gold nanoparticles compared to their smooth, flat surfaces.**

*From a collection of nanoscale platinum particles, left, researchers homed in on the chemistry occurring in different surface areas of individual nanoscale platinum particles like the one at right, which measures about 100 billionths of a meter across. Researchers found that chemical reactivity is concentrated at the edges of the particles (red circle at right), with lesser activity in the central area (black circle). This image was produced by an atomic force microscope. (Credit: "High-spatial-resolution mapping of catalytic reactions on single particles," Nature, Jan. 11, 2017)*

**CO-LEADER F. DEAN TOSTE SAID, “WE CAN NOW DIRECTLY IDENTIFY THE IMPORTANT ROLE OF SURFACE DEFECTS IN ACTIVATING INDUSTRIALLY RELEVANT CATALYTIC PROCESSES”.**

### 1. What questions are you trying to answer in catalyst research?

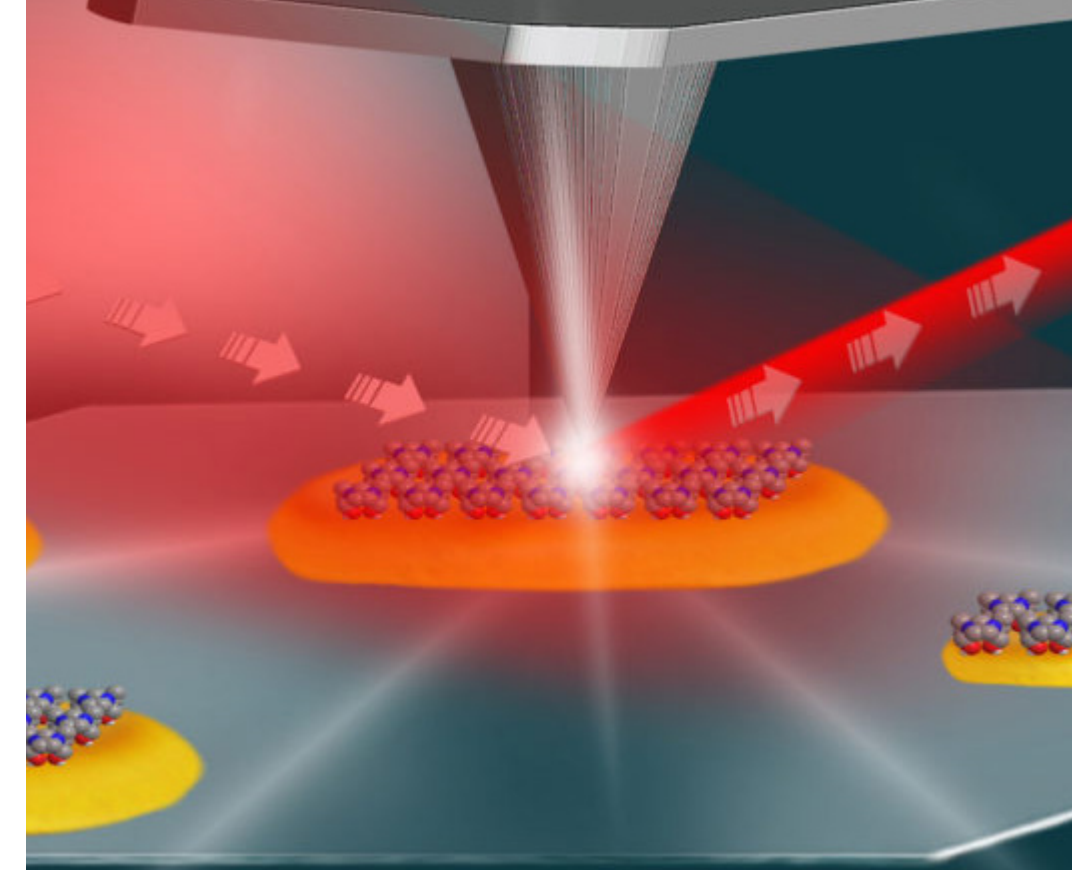
In our work we address fundamental questions in catalysis research and try to answer them by using specifically-designed model systems and advanced spectroscopy measurements. Specifically, in the published work we wanted to identify the active sites on the surface of single catalytic particles. To achieve this goal, we prepared a model system in which chemically active molecules were anchored to the surface of Pt nanoparticles. The chemical reactivity of these surface-anchored molecules was probed with high spatial resolution IR nanospectroscopy measurements.

### 2. What did you discover about how the atomic structure of nanoparticles impacts their function as catalysts in chemical reactions?

We identified, based on IR nanospectroscopy measurements, that surface defects are key element in activating catalytic processes. We demonstrated that surface sites which are located at the perimeter of the catalytic nanoparticles and are characterized with high density of surface defects are more catalytically-active than surface sites which are located on the center of the particles and have lower density of surface defects.

### 3. Can you explain what you discovered about the jagged edges and defects that shows they are actually beneficial?

We discovered that areas with high density of surface defects, such as particles' edges, can better activate catalytic processes. Metal atoms at defect sites have lower number of



*This illustration shows the setup for an experiment at Berkeley Lab's Advanced Light Source that used infrared light (shown in red) and an atomic force microscope (middle and top) to study the local surface chemistry on coated platinum particles (yellow) measuring about 100 nanometers in length. (Credit: Hebrew University of Jerusalem)*

neighboring metal atoms and therefore will strongly interact with reactant molecules, leading to higher catalytic reactivity. The importance of surface defects in activating catalytic reactions is well known in catalysis research. However, up to now, the structure-reactivity correlation was not documented on single particles.

Catalysts can improve the rate of chemical reactions and allow them to be more efficient while staying unchanged in the process, and are involved in the manufacture of a range of industrial products, including fuel, fertilizers and plastics. This study, reported in Nature [Wu et al. Nature (2017) DOI: 10.1038/nature20795], used a new spectroscopic approach to detect chemical processes at a nanoscale resolution, increasing our knowledge of how the atomic structure of nanoparticles impacts their function as catalysts in chemical reactions. Being aware of the exact level of energy that's required to trigger chemical reactions is key to optimizing reactions – as co-leader Elad Gross states, “This technique has the ability to tell you not only where and when a reaction occurred, but also to determine the activation energy for the reaction at different sites”.

### 4. How might your research help make chemical processes "greener"?

Our research helped to identify the crucial role of surface defects in activation of catalytic reactions, providing guidelines for preparation of optimized catalysts. These optimized catalysts, that will have higher density of surface defects, will be more efficient in activating catalytic processes, thus requiring less energy for activating the catalytic reactions and will also reduce the amount of undesired byproducts which can be formed during less-efficient catalytic process.

### 5. How is the Advanced Light Source at Berkeley Lab used in your research?

In the experiments that we conducted we used a unique setup that was assembled in the Advanced Light Source and combines the high brightness of the Advanced Light Source and Atomic Force Microscope.

### 6. Why is Atomic Force Microscopy in your research?

In our experiments the tip of the Atomic Force Microscope is used as an antenna that localizes and enhances the infrared light. Therefore, by combining the high-brightness infrared source of the synchrotron and the atomic force microscope we were able to track chemical process on the surface of single nanoparticles with a spatial resolution of ~20 nm.



## WHAT IS THE ADVANCED LIGHT SOURCE AT BERKELEY? WHAT PROJECTS DO THEY WORK ON?



The Advanced Light Source (ALS) is a specialized particle accelerator that generates bright beams of x-ray light for scientific research. Electron bunches travel at nearly the speed of light in a circular path, emitting ultraviolet and x-ray light in the process. The light is directed through about 40 beamlines to numerous experimental endstations, where scientists from around the world ("users") can conduct research in a wide variety of fields, including materials science, biology, chemistry, physics, and the environmental sciences. Operation of the ALS is funded by the U.S. Department of Energy, Office of Basic Energy Sciences. It cost \$99.5 million to build.

The wavelengths of the synchrotron light span the electromagnetic spectrum from infrared to x-rays and have just the right size and energy range for examining the atomic and electronic structure of matter. These two kinds of structure determine nearly all the commonly observed properties of matter, such as strength, chemical reactivity, thermal

and electrical conductivity, and magnetism. The ability to probe these structures allows us to design materials with particular properties and understand biological processes inscrutable to visible light.

Most recently, advances in accelerator design have made it possible to construct diffraction-limited storage rings (DLSRs), where the electron beam size is comparable to the wavelength of the light emitted. According to preliminary studies, a DLSR upgrade of the ALS (ALS-U) modifying the ALS magnetic lattice and injection process would vastly improve brightness, coherence, and resolution. On September 27, 2016, ALS-U received "critical decision zero" (CD-0) status from the U.S. Department of Energy, the first milestone in a path forward that will allow a 23-year-old facility to maintain world leadership in soft x-ray science for at least another 20 years.

The upgrade project, known as ALS-U, will boost the brightness, focus, and other

properties of the light beams produced at the ALS.

The ALS is a type of synchrotron: It creates and accelerates powerful electron beams that emit light as they travel around bends. This light, which ranges from infrared to X-ray wavelengths, is channeled to dozens of beamlines, where it is used to explore a variety of sample types at microscopic scales for experiments that span from materials science and chemistry to environmental science and biology.

*The Advanced Light Source is a U.S. Department of Energy (DOE) Scientific User Facility supported by the Director, Office of Science, Office of Basic Energy Sciences and operated for the DOE Office of Science by Lawrence Berkeley National Laboratory.*



## PARK INDUSTRY NEWS

# PARK SYSTEMS

## WORLD LEADER MANUFACTURER OF ATOMIC FORCE MICROSCOPES ANNOUNCES OPENING OF EUROPEAN HEADQUARTERS IN HEIDELBERG, GERMANY

(April 18, 2017 Santa Clara, CA) Park Systems, world-leader in atomic force microscopy (AFM) recently announced the opening of their European Headquarters in Heidelberg, Germany and appointment of Ludger Weisser as General Sales Manager. Park Systems is a publicly traded company listed on KOSDAQ since 2015 where they received "AA" from two separate rating agencies on advanced technologies, becoming the first company listed that year through the special technologies IPO program. Dr. Sang-il Park CEO and founder of Park Systems worked as an integral part of the group at Stanford University that first developed AFM technology.

"We are very excited to open our European Headquarters and to create closer relationships with our valued customers in Europe," states Dr. Sang-il Park, Park Systems Founder and CEO. "The appointment of Ludger Weisser as European General Sales Manager demonstrates our commitment to provide best-in-class technical sales support and technology collaboration with our customer to provide unique customer-driven Atomic Force Microscopy solutions."

The opening of the European Headquarters will provide support for the vast network of highly skilled Park AFM distributors already established throughout Europe. Park Systems European distributors include Schaefer Technologie GmbH in Germany, Elexience in France, LOT-Quantum Design in the UK, ST Instruments B.V. in BeNeLux, Gambetti Kenologia Srl in Italy, and Biometec Tecnologia y Sistemas, s.a in Spain. To find a distributor location for Park AFM near you, go to: <http://www.parkafm.com/index.php/company/locations>

Ludger Weisser brings over twenty years of experience in management positions with Atomic Force Microscopy companies,

including Director of European Sales with Asylum Research and Managing Director at Atomic Force F&E GmbH, an advanced metrological instrumentation organization for the European research community. His extensive background includes a degree in Physics from the University of Heidelberg, university research on polymer surfaces and work with Digital Instruments, later acquired by Veeco where he was a sales manager.

"The fast-paced European scientific and industrial markets continue to make significant advancements in all areas of nanotechnology," says Ludger Weisser, General Sales Manager for Europe. "Park Systems has a long history as a leader in atomic force microscopy, providing unsurpassed nanoscale advances in continuous pursuit of the latest AFM innovations." The Park Systems European Headquarters launches a focused effort to develop close relationships with European thought leaders within the scientific and manufacturing communities and develop products that meet their needs.

Park Systems is a leading innovator in nanoscale microscopy and metrology and continues to invest in the development of emerging technology. They now have offices in Korea, United States, Japan, Singapore and Europe. As the nanoscale microscopy demands more effective AFM technology, Park Systems is focused on revolutionary breakthroughs for the best AFM products offered in both science and industry. 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™).



### LUDGER WEISSER, PARK SYSTEMS NEWLY APPOINTED EUROPEAN GENERAL SALES MANAGER AT THE PARK SYSTEM EUROPEAN HEADQUARTERS IN HEIDELBERG, GERMANY

#### ABOUT PARK SYSTEMS

Park Systems is a world-leading manufacturer of atomic force microscopy (AFM) systems with a complete range of products for researchers and industry engineers in chemistry, materials, physics, life sciences, and semiconductor and data storage industries. Park's products are used by over a thousand of institutions and corporations worldwide. Park's AFM provides highest data accuracy at nanoscale resolution, superior productivity, and lowest operating cost thanks to its unique technology and innovative engineering. Park Systems Corporation is headquartered in Suwon, Korea with its Americas headquarters in Santa Clara, California. Park's products are sold and supported worldwide with regional headquarters in the US, Korea, Japan, and Singapore, Europe and distribution partners throughout Europe, Asia, and America. Please visit <http://www.parkafm.com> or call 408-986-1110 for more information.

# FAST IMAGING USING PARK NX10 ATOMIC FORCE MICROSCOPE

**MINA HONG, CHARLES KIM, GERALD PASCUAL, BYONG KIM, AND KEIBOCK LEE TECHNICAL MARKETING & APPLICATIONS, PARK SYSTEMS**

## INTRODUCTION

The Atomic Force Microscope (AFM), a powerful nanotechnology tool, has been widely utilized in the characterization of materials' topography and properties for decades since it was first developed in the 1980s. The advantages of AFM over more traditional forms of microscopy are significant: (1) there are little requirements for sample preparation, (2) it can characterize materials in a non-destructive manner, (3) it can be used to run both bulk data acquisition of multiple samples as well as highly-intensive, single-sample nanoscale investigations and (4) AFM can be runsamples in both air as well as under in-liquid conditions. In all, AFM can be a very powerful technique for doing an in-situ study such as the observation of a material's surface topographical changes and particle transportation during the reaction processes. However, to keep track of the topography changes and particle transportation, the AFM needs to be able to image as fast as possible. Motivated by this need, scientists and engineers from Park Systems have been pushing themselves very hard to improve their existing AFM systems to scan as fast as possible without the loss of resolution or the need of any additional setup.

Nowadays, with Park's NX series of AFM systems and high frequency cantilevers, one can generate AFM images at the expected high resolution and increased scan speed. All Park NX series AFMs are built for Zscanner feedback speed and optimized Zservo control so that an ultra high frequency cantilever is all that is

needed to carry out high-speed AFM imaging. Dubbed Fast Imaging, Park's solution to the need for expediting AFM image generation enables the operator to perform quick and accurate imaging of sample surfaces that have a variation in feature heights ranging from single nanometers to tens of nanometers.

## EXPERIMENTS

The Park NX10 AFM was used to perform a fast imaging operation with the ultra-short cantilevers (USC) supplied by NanoWorld. USC has a spring constant of 3N/m and a resonance frequency of 2MHz. Three commercial products were imaged to test the ease of use and accuracy of the AFM's fast imaging capability. All experiments were run under non-contact mode using the Park Smart Scan AFM operation software.

The first sample imaged was Pentacene film. Pentacene is a polycyclic aromatic hydrocarbon consisting of five linearly-fused benzene rings. The rings are connected to form a structure that has surface height differences of roughly 10 nanometers. It is a highly conjugated compound that can be used as an organic semiconductor. The second sample that was run was a triangular aluminum production pattern. The third sample tested was Celgard, a material used as a separator and filtration membrane in porous lithium ion batteries as well as in several medical applications. The surface of Celgard has a high variation of feature heights, on the order of tens of nanometers. All samples were imaged with a speed increase from 1Hz to 10 Hz.

## RESULTS AND DISCUSSIONS

Combined with the Park SmartScan AFM operation software, fast imaging on the Park NX10 AFM system is as user-friendly as the AFM's already highly accessible basic non-contact mode. The high productivity, high resolution, and high performance of this technique were well exhibited from the sample measurements to be discussed.

As a promising organic semiconductor for film and device applications, Pentacene and the growth of defects and grain boundaries on it are of great interest to researchers. AFM is the main analysis technique to study the characteristics of this material [1]. Figure 1 clearly exhibits the Pentacene surface features such as the holes (defects) and the monolayer structures. With the scanning speed increased from 1Hz (Figure 1a) to 10Hz (Figure 1b), there is no obvious difference between these observed features. We believe with a scanning speed of 10Hz, the changes in the Pentacene's surface topography can be well tracked in environments that promote real growth or dissolution.

Celgard is perhaps the most structurally complex of the three samples scanned with fast imaging. Its structure comes from it being a porous material that is made by stretching isotactic polypropylene [2]. Celgard's topography is composed of areas with drawn fibrils and areas with undrawn crystalline lamella (Figure 3). Due to the height variation and the fact that the fibrils are freely suspended, the fibrils are very challenging to track for conventional techniques. Figure 3 provides us proof that the details of the surface structure were captured perfectly even at the speed of 10 Hz.

## CONCLUSION

As demonstrated, the topographies of Pentacene, an aluminum projection pattern, and celgard were efficiently and accurately imaged using the Park NX10 AFM system. All Park Systems NX series AFMs can perform fast imaging without any additional required hardware except for high frequency cantilevers. We believe the easy to use fast imaging from Park Systems will successfully provide researchers with an accelerated method to acquire in-situ, real-time critical material surface topography and property change information to help them acquire a better understanding of their samples at the nanoscale.

## REFERENCE

- [1] J. Am. Chem. Soc., 2005, 127 (33), pp 11542-11543
- [2] <https://www.asylumresearch.com/Gallery/CypherFastScanning/FastScan1.shtml>

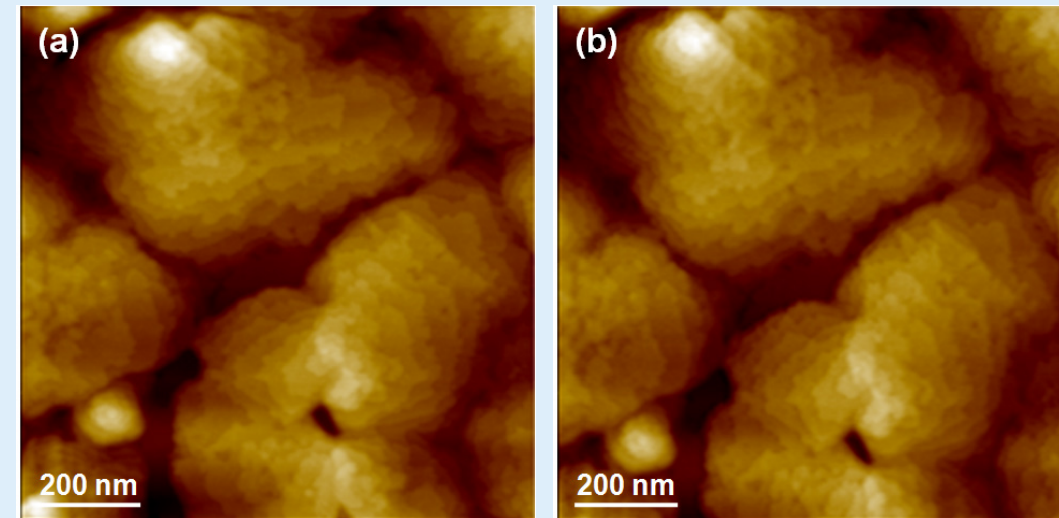


Figure 1. Topography images of pentacene film imaged at (a) 1Hz and (b) 10Hz.

Scan size:  $1\mu\text{m} \times 1\mu\text{m}$ , image size: 512px  $\times$  512px.

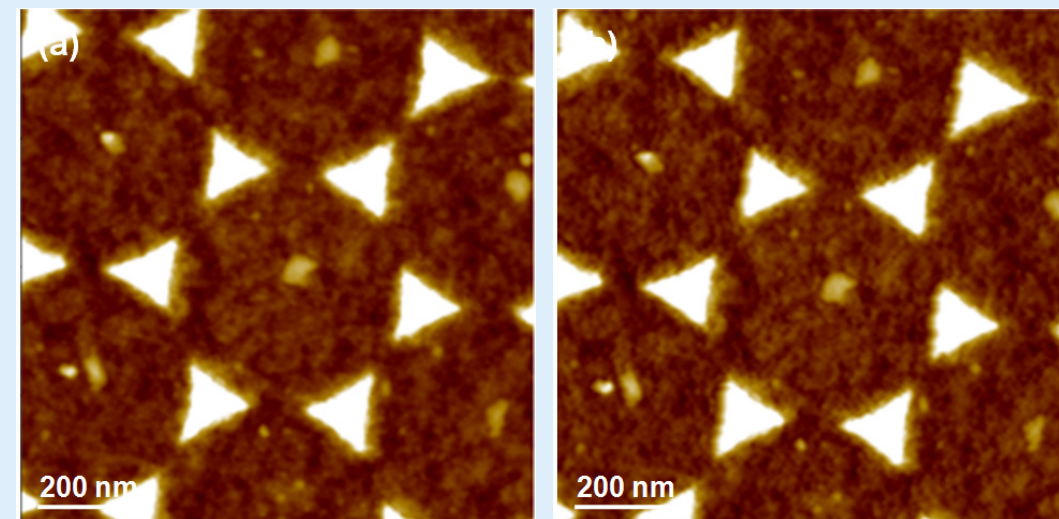


Figure 2. Topography images of triangular aluminum production patterns imaged at (a) 1Hz and (b) 10Hz. Scan size:  $1\mu\text{m} \times 1\mu\text{m}$ , image size: 512px  $\times$  512px.

In Figure 2, the sharp edges of the 15nm high triangular aluminum production pattern can be clearly revealed at both low and high speed without sacrificing any image quality.

The faster 10 Hz imaging speed allows one to see at a quality equal to the image produced at 1 Hz.

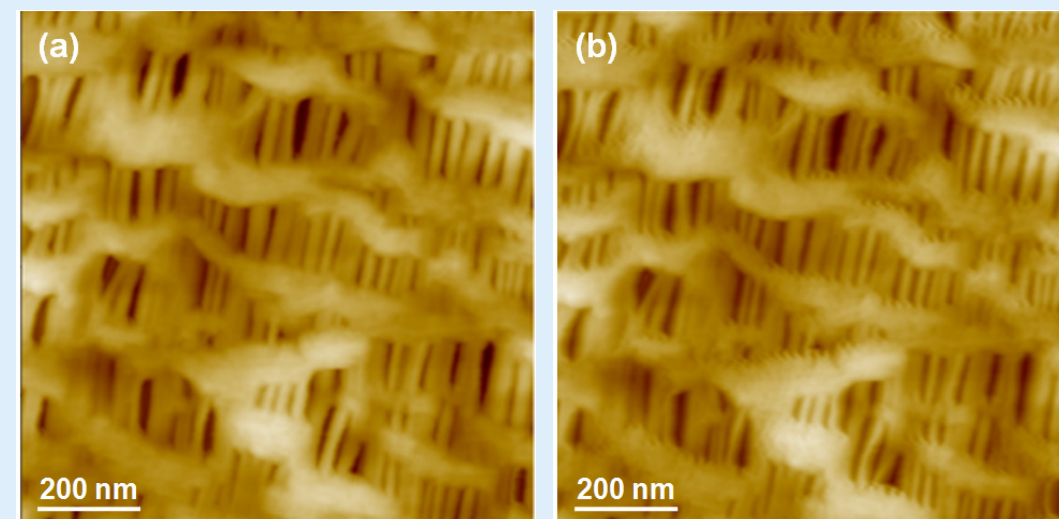


Figure 3. Topography images of Celgard imaged at (a) 1Hz and (b) 10Hz.

Scan size:  $1\mu\text{m} \times 1\mu\text{m}$ , image size: 512px  $\times$  512px.

# PARK SYSTEMS ANNOUNCES PARK AFM SCHOLARSHIP AWARD WINNERS

“WE ARE DELIGHTED TO OFFER FINANCIAL INCENTIVE TO PARK AFM SCHOLARS WHO ARE PIONEERING NEW RESEARCH METHODOLOGIES IN NANOTECHNOLOGY AT LEADING ACADEMIC INSTITUTIONS WORLDWIDE,” STATED KEIBOCK LEE, PARK SYSTEMS PRESIDENT. “OUR CONTINUED MISSION IS TO SUPPORT THE PROMOTION OF SHARED KNOWLEDGE AMONGST INTERDISCIPLINARY TEAMS OF SCIENTISTS AND ENGINEERS TO ADVANCE NANOSCALE DISCOVERIES.”

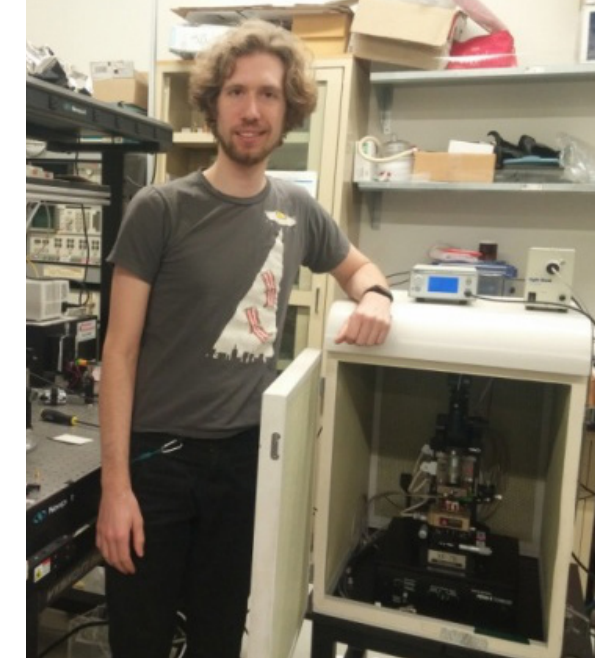


Park Systems Park AFM Scholarship Award is eligible to postdoctoral students or researchers working in nanotechnology research using Park AFM. As progress for nanotechnology research and development advances at an unprecedented rate, universities world-wide offer degree programs in nanotechnology. Park Systems, world-leading manufacturer of Atomic Force Microscopes is offering two monetary scholarships to promote the education of future scientists and engineers in a number of nanoscale research areas that require advanced nano microscopy for analysis and to promote shared research findings and methodologies amongst researchers.

Below and on the following pages are the first Park AFM Scholarship Award Winners. For more information on the Park AFM Scholarship, go to: <http://www.parkafm.com/index.php/medias/programs/park-afm-scholarship>



*Park NX 10 The most accurate and reliable atomic force microscope for small sample research using the world's only true non contact AFM.*



## SCOTT JOHNSTON

is a graduate student in the applied physics department at Stanford University. He currently works on scanning microwave impedance microscopy and its application to new materials and devices. Systems of interest include ferroelectrics, photovoltaics, and phase change materials.



## PATRICK GALLAGHER

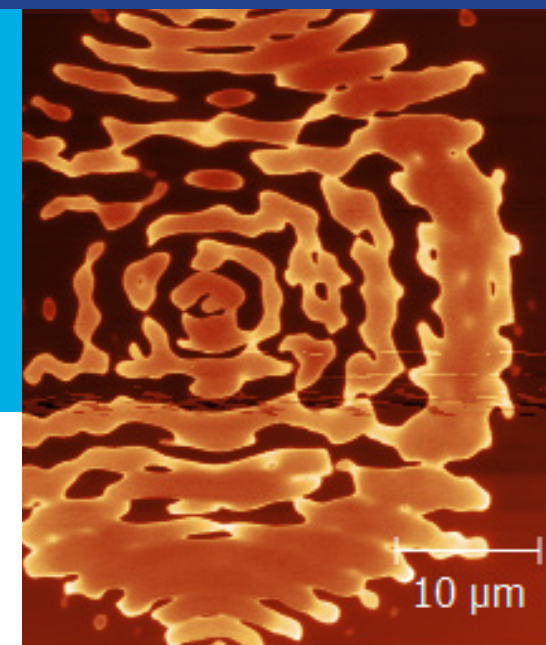
received his PhD from Stanford in 2017 and is now a postdoctoral fellow at the Kavli Energy Nanoscience Institute at UC Berkeley. He studies two-dimensional systems of electrons, which can form on surfaces, at interfaces, or in atomically thin materials. Patrick's work has most recently focused on graphene, but past work has involved electron systems in strontium titanate and gallium arsenide. Experimental techniques include terahertz spectroscopy, optical spectroscopy, electrical measurement, and scanning probe microscopy.

“I'VE FOUND THE PARK XE-100 TO BE A VERSATILE AND CONVENIENT PLATFORM FOR AFM AND STM. ITS LOW VERTICAL VIBRATION WAS PARTICULARLY USEFUL FOR MY STUDY OF SELF-ASSEMBLY ON GRAPHENE, AS THE MEASURED TOPOGRAPHIC VARIATION OF INTEREST WAS OFTEN LESS THAN 1 ANGSTROM.”

### Self-assembly of environmental adsorbates on graphene and other 2D materials Abstract:

Graphene sheets on atomically flat substrates are expected to be flat. Yet recent studies of nominally flat graphene using high-resolution atomic force microscopy have revealed an apparently corrugated surface: topography scans show large-scale periodic structures of stripes whose period is 4 nm and whose amplitude is less than a nanometer. I will present scanning probe and optical measurements that show that these stripes are self-assembled environmental adsorbates, the chemical identity of which is still under study. This self-assembly appears to be common on 2D materials, as the same phenomenon occurs on hexagonal boron nitride sheets, and 4 nm-periodic stripes were recently observed on molybdenum disulfide by another group. I will discuss the impact of the self-assembled stripes on the frictional, optical, and electronic properties of graphene samples.

References: P. Gallagher et al. Nature Commun. 7, 10745 (2016). P. Gallagher et al. in prep (2016).



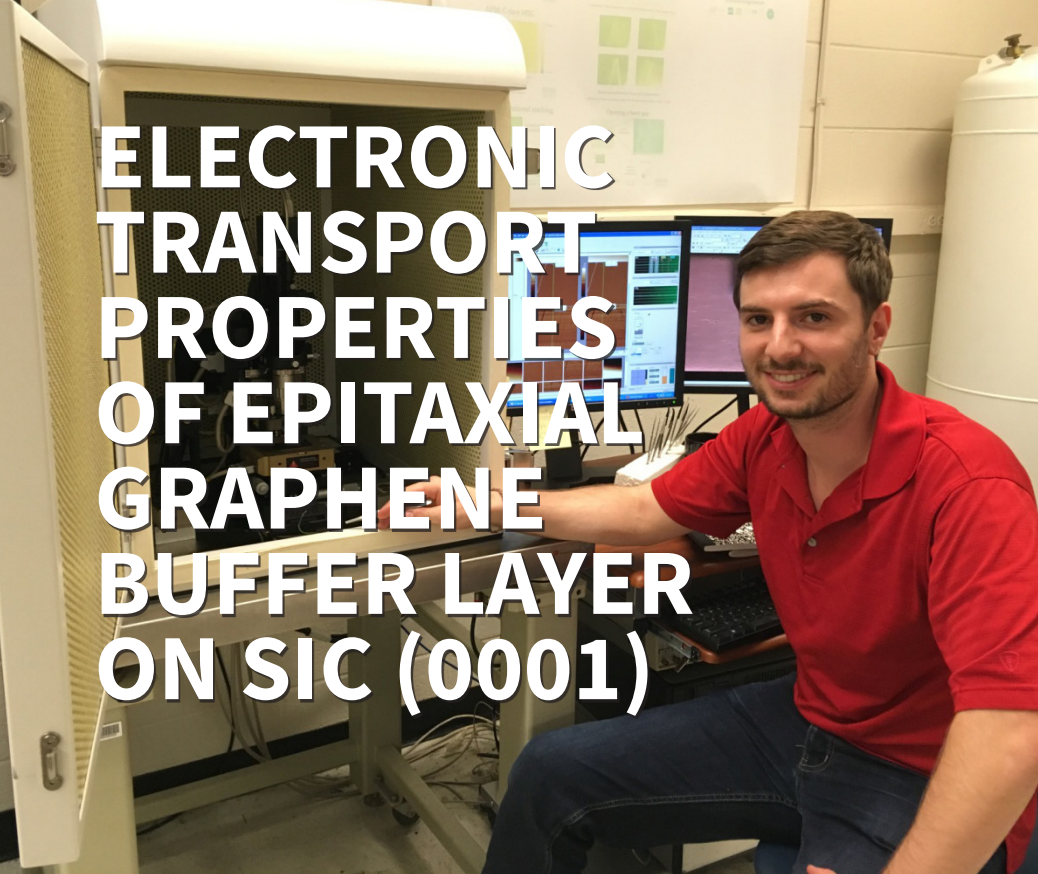
“OUR PARK AFM GIVES US THE STABILITY AND PRECISION THAT WE NEED TO PUSH THE BOUNDARIES OF ULTRA-SENSITIVE NANOSCALE IMPEDANCE MEASUREMENTS.”  
AFM SCHOLARSHIP WINNER SCOTT JOHNSTON IN FRONT OF THE PARK AFM

### How do you think your research will impact society in a positive way?

I hope that the insights we gain from probing the nanoscale electronic properties of applied materials will lead to improvements in their performance. This could include better surface acoustic devices from ferroelectrics with application to wireless communications, better phase change memory for computing, and of course better photovoltaics.

THIS IS A "GST" IMAGE OF THE PHASE CHANGE MATERIAL GESBTE. THIS "SAW" IMAGE IS OF SURFACE ACOUSTIC WAVE RESONANCES IN A FERROELECTRIC DOMAIN, AND IS FROM THE PRESENTATION ENTERED FOR THE PARK AFM SCHOLARSHIP.

# ELECTRONIC TRANSPORT PROPERTIES OF EPITAXIAL GRAPHENE BUFFER LAYER ON SiC (0001)



## JEAN-PHILIPPE TURMAUD

was born near Lyon, France in 1990. Growing up on the country side and regularly escaping into the Alps, he developed a curiosity towards nature, weather phenomena and science in general. Learning more about basics physics in high school lead him to choose a higher education in engineering and physics. He graduated with a Master of science in engineering physics from Grenoble Institute of Technology and the Royal Institute of Technology in Stockholm. Now, he is working on getting his PhD in Physics from Georgia Tech where his research focuses on the electronic properties of different forms of epitaxial graphene on silicon carbide.

Park AFM Scholarship Winner, Jean-Philippe Turmaud currently working on his PhD in Physics from Georgia Tech where his research focuses on the electronic properties of different forms of epitaxial graphene on silicon carbide. He characterizes samples by Raman spectroscopy, AFM, XPS and ARPES to confirm their structural properties and produce electronic devices on single SiC terraces.

**"OUR PARK AFM ALLOWS US TO ANSWER CRUCIAL QUESTIONS ABOUT THE STRUCTURE OF THE SURFACE OF OUR MATERIAL: DID WE PRODUCE A SURFACE COVERED WITH GRAPHENE? WHAT IS THE SIZE OF THE GRAPHENE RIBBONS WE ARE GOING TO INVESTIGATE? IS THERE ANY KIND OF CONTAMINATION? WHAT IS THE ROUGHNESS OF THE SURFACE?" JEAN-PHILIPPE TURMAUD**

The confinement control sublimation is used to produce high quality epitaxial graphene on SiC for nanoelectronics. We report here on the experimental investigation of the first graphene layer grown on SiC(0001) (the buffer layer). The buffer layer is a semiconducting form of graphene, with a gap in the density of state previously probed by ARPES and STM measurement. We characterize our samples by Raman spectroscopy, AFM, XPS and ARPES to confirm their structural properties and produce electronic devices on single SiC terraces. The temperature and electrical field dependence of the bulk conductivity of the buffer layer are investigated and the effects of contacts and gas adsorption are considered. The observed behavior seem to be related to the known structural periodicity of the buffer layer.

### How do you think your research will impact society in a positive way?

There is no doubt that today's society is profoundly shaped by our use of technology. From renewable energy production to self driving cars, the need for cheap but efficient functional materials is constantly increasing. The development of the silicon industry was only possible through years of research on the fundamental and practical properties of semiconductors. In the same way, materials of future application need the same fundamental knowledge of their properties. My research is oriented towards that goal. It focuses on understanding the electronic transport in epitaxial graphene, a promising candidate as a successor to silicon in many applications. My project aims to bring light onto limiting factors of electrical conduction in novel two dimensional materials like graphene, to eventually overcome them and produce competitive devices.

### What is the best or most useful part of using Park AFM for your research?

Prior to building devices with epitaxial graphene on silicon carbide, it is essential to know the structure of the surface of our material: did we produce a surface covered with graphene? What is the size of the graphene ribbons we are going to investigate? Is there any kind of contamination? what is the roughness of the surface? Our Park AFM allows us to answer those crucial questions. The most powerful mode we use is the Lateral Force Microscopy. It allows us, with excellent resolution to measure the local friction of the surface and discriminate between a bare silicon carbide surface, one layer of graphene and two or more layers of graphene, which all have different friction coefficients. We can then build our devices accordingly.



## DR. XIAMING CHEN

Dr. Xiaming Chen received his B.S. and M.S. degree in Mechanical Manufacturing and Automation from Xi'an Jiaotong University in China, and Ph.D. degree in Mechanical Engineering from State University of New York. Dr. Chen's research focuses on investigating the mechanical properties of carbon and boron nitride nanotubes and their polymer nanocomposites, part of a broad effort to develop next-generation, light-weight and high-strength multifunctional engineering materials, particularly for aerospace applications. In collaboration with NASA and the National Institute of Aerospace, and financially supported by the Air Force Office of Scientific Research, he tackles very challenging problems and has made several breakthroughs using state-of-the-art nanomechanical testing techniques. His research findings help to better understand the mechanical strength of nanotube structures and the local stress transfer on the nanotube-polymer interfaces, both critical for design and optimization of innovative nanotube-based material systems. He has published nineteen articles and has two book chapters to his credit. He has made more than 20 conference presentations and holds one patent. Dr. Xiaming Chen received several awards and honors from State University of New York at Binghamton, National Science Foundation and American Society of Mechanical Engineers.

Here we present an in situ electron microscopy nanomechanical study of nanotube-polymer interfaces between individual CNTs/BNNTs and polymers in conjunction with atomistic simulations. By pulling out individual nanotubes from polymer films inside a high resolution electron microscope, the nanomechanical measurements capture the shear lag effect on nanotube-polymer interfaces. Our nanomechanical measurements

reveal that BNNTs can form much stronger binding interfaces with polymers than comparable CNTs and that the interfacial strength of BNNT-epoxy interfaces is higher than that of BNNT-PMMA interfaces. The observed superior load transfer capacity of BNNT-polymer interfaces is ascribed to both the polarized nature of B-N bonds and the high bonding potentials of B and N atoms, which are supported by molecular dynamics (MD) simulations. The findings contribute to a better understanding of the local load transfer on the tube-polymer interface and the tube's reinforcing mechanism. In addition, the extraordinary load transfer capacity of BNNT-polymer interfaces suggests that BNNTs are excellent reinforcing nanofiller materials for light-weight and high-strength polymer nanocomposites.

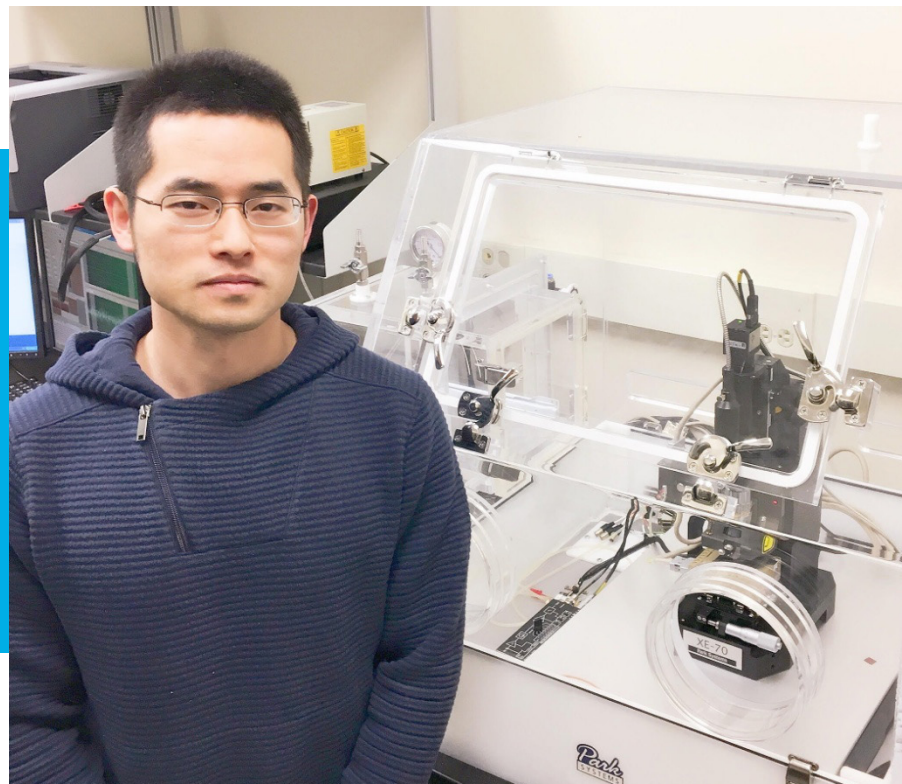
### 1. How do you think your research will impact society in a positive way?

The research objective of this project is to investigate the mechanical properties of carbon nanotubes (CNTs) and boron nitride nanotubes (BNNTs) and their polymer composites by using multi-scale experimental approaches. This study will focus on the quantitative experimental characterization of (1) mechanical properties of CNTs and BNNTs in three structural forms (i.e. individual tubes, thin-bundles and yarns) and the interfacial binding strength of the respective tube-tube interactions; and (2) mechanical properties of CNT and BNNT-based polymer composites and the interfacial shear strength of the respective tube-polymer interfaces. Our proposed nanotube-reinforced polymer composite study will employ epoxy and polyimide as matrix materials because they are widely used for structural applications in aerospace industries. The elastic moduli and yield

strengths of CNT and BNNT nanostructures and the respective tube-tube and tube-polymer interfacial strengths will be characterized by using the state of the art in-situ scanning electron microscopy (SEM) and atomic force microscopy (AFM) mechanical characterization techniques. Both of our proposed nanoscale experimental techniques uniquely enable the high-resolution concurrent measurements of the applied load and the mechanical response of the nanostructure under a variety of testing conditions (e.g. tensile, peeling and pull-out tests). The mechanical properties of CNT and BNNT-based yarns and thin-film polymer composite will be characterized by using micro/meso-scale tensile testing techniques. Using our multi-scale experimental platforms, we will systematically investigate the effects of harsh environments (e.g. high temperature and strong radiation) on the tube-tube interfacial strength and mechanical properties of CNTs and BNNTs, and the effect of the surface functionalization on the tube-polymer interfacial stress transfer and mechanical properties of CNT and BNNT-reinforced polymer composites. The impact of this project includes significant advances of the nanoscale mechanical characterization technique and our knowledge of the mechanical properties of CNTs and BNNTs in various structural forms and the interfacial strength of the respective tube-tube and tube-polymer interactions. This study will provide critical insights into the role of the interfacial interaction in the mechanical properties of CNTs and BNNTs and their polymer composites, and will directly contribute to the optimal design, modeling and manufacturing of novel multi-material and multi-functional light-weight high-strength materials systems, which are critically demanded for many aerospace and automobile industries applications.

### 2. What is the best or most useful part of using Park AFM for your research?

As a leading provider of atomic force microscopy, Park systems provides powerful functions and tools for nanoscale research and engineering. The most useful part of Park system in my research is Lateral Force Microscopy (LFM), which not only provides accurate topographic measurements, but also gives the surface frictional information of our nanoscale research samples. When the AFM cantilever scan the sample, the cantilever can move even cut the sample with specific normal load and scanning velocity, meanwhile the morphology and lateral force will be recorded. By using LFM, we already successfully investigate the dynamic and frictional properties of carbon nanotubes, boron nitride nanotube, graphene and boron nitride nanosheets.



CHNEGLIN YI

Chneglin Yi is a Research and Teaching Assistant at the Department of Mechanical Engineering Thomas J. Watson School of Engineering and Applied Science State University of New York and is a PhD Candidate.

**HIS RESEARCH FOCUS IS ON EXPERIMENTAL NANOMECHANICS OF NOVEL 1D AND 2D NANOSTRUCTURES, TESTING THE MECHANICAL PROPERTIES AND INTERFACES, INVESTIGATE THE FUNDAMENTAL MECHANICAL PROPERTIES OF CARBON NANOTUBES (CNTS) AND BORON NITRIDE NANOTUBES (BNNTS) AND THEIR METAL COMPOSITES BY USING MULTI-SCALE EXPERIMENTAL APPROACHES.**

**Nanomechanical Folding and Unfolding of Graphene on Flat Substrate**

Graphene is a type of two-dimensional nanostructure with extraordinary physical properties, and is promising for a number of applications. Due to its ultra-thin characteristics, graphene can easily fold under external stimuli such as mechanical forces. The substantial local deformation in folded graphene has a prominent influence on its electrical properties. Understanding and ultimately having a good command of the mechanical deformation in folded graphene is of importance to the design and manufacturing of graphene origami and its functional mechanical and electrical properties. The study focuses on investigating the local folding and unfolding behaviors of few-layer graphene sheets by using atomic force microscopy (AFM) techniques. The bending rigidity of few-layer graphene and the interlayer shear interaction during the graphene folding process are studied. The results reveal that the bending stiffness of two to six layers graphene follows a square-power relationship with its thickness. The study demonstrates that it is a plausible venue to qualify the pure bending stiffness of graphene through measuring its self-folding conformation on flat substrates. The nanomechanical measurements also reveal that individual graphene sheets can be mechanically folded in a buckling delamination mode, which leads to accordion-shape self folded graphene on flat substrates. This work is useful to better understand the structural and mechanical properties of graphene, and in

the pursuit of its applications, in particular, as programmable nanoscale origami structures.

**How do you think your research will impact society in a positive way?**

With the development of nanotechnology, nanomaterial has been widely studied by lots of research groups. But the fundamental research still remains limited. I think our research is quite important for the applications of nanomaterials (Carbon nanotubes, Graphene, etc). Since during the manufacture process of graphene based devices, especially in aerospace and automobile industries, folding and unfolding phenomenon cannot be avoided. In our study, we quantitatively explored the folding and unfolding process by using atomic force microscopy (AFM) techniques. The research findings are useful to the study of active and controllable folding of graphene and in the pursuit of graphene origami with complex geometries.

**What is the best or most useful part of using Park AFM for your research?**

For our research, Park AFM is not only used for topographic images of sample, but also worked as a nano-manipulator. So the availability of accurate force control is quite important for us. With the powerful software (XE1), we can take advantage of Park AFM to applied normal load on the sample in order to fold and unfold the graphene and record the corresponding topography and lateral force profiles, which is the key data for our study.



JING FU

Jing Fu received her Master's degree (2011) in Environmental Science & Engineering from East China University of Science and Technology, where she specialized in alkaline fuel cells. After graduation, she worked with Volvo Cars Corporation as a chassis design engineer (2011-2014). Jing is now pursuing her Ph.D. in Chemical Engineering under the supervision of Prof. Zhongwei Chen at the University of Waterloo.

**HER CURRENT RESEARCH INTERESTS INCLUDE THE DEVELOPMENT OF ADVANCED NANOSTRUCTURED ELECTRODE MATERIALS AND SOLID-STATE ELECTROLYTES FOR FLEXIBLE RECHARGEABLE METAL-AIR BATTERIES.**

**Flexible bifunctional oxygen electrode through morphological emulation of human hair array for rechargeable zinc air batteries**

Zinc-air batteries have a huge weight advantage over comparable types and significantly improve energy density. Many researchers have sought highly efficient nanosized oxygen electrocatalysts for better battery performance and rechargeability, but the potential benefits of those catalysts are lost significantly by depositing physically on limited surfaces of the air electrodes. Inspired by the growth and morphology of the human hair, we have designed an electrically rechargeable, nanoarchitected air electrode that morphologically emulates human hair array. This hair-like array, consisting of nanoassemblies involving two-dimensional mesoporous Co3O4 nanopetals in one-dimensional carbon nanotubes, is supported vertically on a flexible stainless-steel mesh (Co3O4-NCNT/SS). The morphology of the hair-like nanoassemblies was well characterized by AFM, SEM and TEM techniques. Using the Co3O4-NCNT/SS air electrode, a solid-state zinc-air battery is able to deliver a high energy density of 847.6 Wh kg-1, accompanied with excellent cycling stability over 600 h. In addition to the pronounced electrochemical performance, the superior mechanical flexibility of the Co3O4-NCNT/SS electrode allows its use in smart-wearable electronic application.

**1. How do you think your research will impact society in a positive way?**

Batteries are a hugely important technology. Modern life would be impossible without them. Conventional approaches to powering mobile electronics have predominantly focused on maximizing capacity in rechargeable batteries intended as internal components in rigid products. New innovation and approaches to energy storage are required to meet the expanded physical and safety requirements of new flexible and thin form factor applications that are intended for new use cases, such as wearable electronics and on body medical devices. The flexible zinc-air battery technology we developed is essential to make electronics systems truly flexible while maintaining electrical functions. The unique feature of the nanostructured air electrode is key to robust flexibility and high energy density. Thus, we should expect battery technology advances to be one of the cornerstone enablers for new functionality and product design in thin, flexible, lightweight, and low cost electronics.

**2. What is the best or most useful part of using Park AFM for your research?**

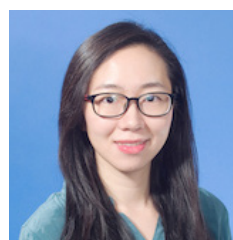
Park AFM is a very useful tool to analyze structural geometry and to acquire nanoscale morphology of my electrode material. Of particular use is the in-liquid imaging technology that AFM provides. This technique allows for the study of the electrochemical reaction analyses of the three-dimensional nanostructured electrode material in liquid electrolyte directly.

# PARK SYSTEMS INTRODUCES PARK NX12 FOR UNSURPASSED AFFORDABLE HIGH RESOLUTION NANOSCALE IMAGING REQUIRED FOR ADVANCED ANALYTICAL CHEMISTRY AND ELECTROCHEMISTRY RESEARCH

Park NX12, an affordable versatile platform for analytical chemistry and electrochemistry researchers and multi-user facilities. Park NX12 features a versatile Inverted Optical Microscope (IOM) based SPM platform for SICM, SECM, and SECCM in addition to Atomic Force Microscopy for research on a broad range of materials from organic to inorganic, transparent to opaque, soft to hard.



**PARK SYSTEMS, WORLD-LEADING MANUFACTURER OF ATOMIC FORCE MICROSCOPES (AFM) JUST ANNOUNCED NEW PARK NX12, AN AFFORDABLE VERSATILE PLATFORM FOR ANALYTICAL CHEMISTRY AND ELECTROCHEMISTRY RESEARCHERS AND MULTI-USER FACILITIES. PARK NX12 FEATURES A VERSATILE INVERTED OPTICAL MICROSCOPE (IOM) BASED SPM PLATFORM FOR SICM, SECM, AND SECCM IN ADDITION TO ATOMIC FORCE MICROSCOPY FOR RESEARCH ON A BROAD RANGE OF MATERIALS FROM ORGANIC TO INORGANIC, TRANSPARENT TO OPAQUE, SOFT TO HARD.**



*"We just purchased Park NX12 because we wanted a high quality research grade, easy to use, versatile, and high resolution AFM/SICM/STM platform,"*

commented Prof. Yixian Wang. *"The Park NX12 was the only comprehensive platform that could perform all SPM techniques (AFM/STM/Pipette based SICM) while also utilizing IOM, for the nanoscale measurement flexibility we needed. We are happy with a very affordable product with so many features and such accurate data."* The Yixian Wang Laboratory at Cal State LA is doing leading edge research on a wide variety of topics including major factors governing the catalyst activity of gold-based catalysts, membrane of individual living cells in real time studying Parkinson's disease and plasmonic imaging of surface electrochemistry of single gold nanowires and are experts at developing new detection protocols.

Park NX12 combines NX10 base with NX-Bio's XY stage that mounts on an inverted optical microscope and supports all of the available modes and options for NX10 with enhanced optics. The Inverted Optical Microscope (IOM) feature is designed to work with transparent samples using a pipette based technique and utilizes PinPoint™ mode in liquid for nanomechanical characterization.

*"Park NX12 is vastly superior to existing solutions by offering the best value research grade high-end IOM based SPM platform, guaranteeing researcher's highest resolution pipette based measurements,"* commented Keibock Lee, President Park Systems. *"Ideally suited for multi user facilities, the unique modular design is a versatile platform designed for further development."*

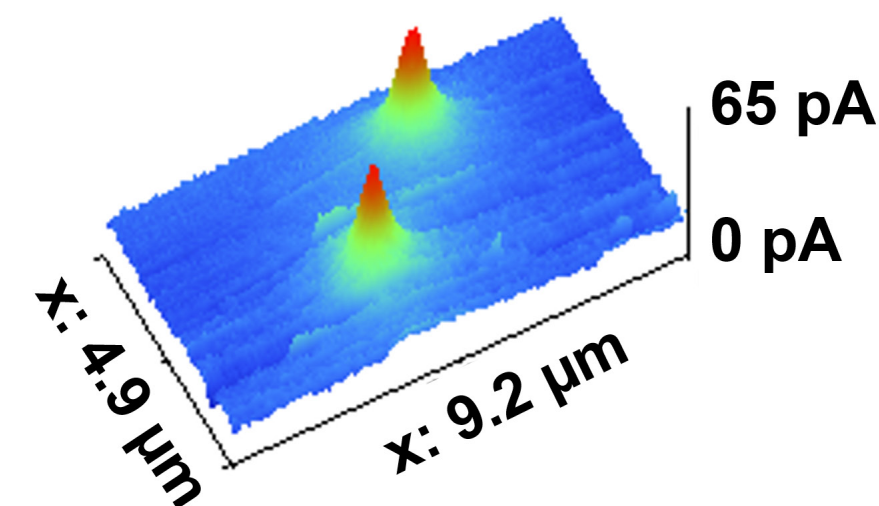
Park NX12 is suited for advanced research on materials such as membranes, organic devices and electronics, and biological and pathological samples, there are outside institution inquiries for characterization of

biomedical devices and materials. In addition, Park NX12 offers an affordable research grade AFM solution for young professors working on biomechanical and pathological studies.



*"Our multi user research lab uses Park NX-Bio and can see the strong advantages in the versatile and comprehensive design of Park NX12,"* commented Prof. Lane Baker, James F. Jackson Associate Professor of Chemistry Indiana University. *"Park Systems is a customer centric manufacturer who carefully listens to their customers needs and develops new products based on customer input."* The Baker Group at Indiana University studies electrochemical methods for analysis and imaging focused on applications of nanopores for the development of chemical and biochemically selective membranes, sensor development and electrochemical imaging.

Park NX12 also solves the challenges in material characterization for clean and renewable energy applications and sensors by providing enough resolution of existing SECM techniques for high resolution detection of interfacial transport and surface chemistry. It also has studios handling of for the pipette probes used in SECM characterization and similarly transparent materials such as nanopore membranes (for fuel cells) and biomembranes (for sensors). Park NX12 is ideal for many industry research applications including electro and analytical chemistry, battery and ESS (Energy storing system), nano pore structure, water purification & treatment, hydrogel study and fluorescence tagging, electrophysiology, neurochemistry, biomimetic, tissue engineering, and biophysics. Park NX12 offers ease of use in both air and liquid, a solution and platform for pipette based SPM techniques (e.g. SICM, SECM, SECCM), and an AFM/optics solutions with broad optical access to the scanning probe and has all of the standard features of Park's line of Atomic Force Microscope products including SmartScan™, revolutionary point-and-click fully automated AFM software.



Description: A polyimide (PI) membrane with track-etched nanopores was mounted on a diffusion cell separating a top chamber and a bottom chamber. Top chamber contained 100 mM KCl. Bottom chamber contained 100 mM KCl + 5 mM Ruthenium hexamine (an electrochemically active molecule). Ruhex molecules will then diffuse from the bottom chamber to the top chamber by the concentration gradient. In SICM topography, two nanopores can be clearly seen. The molecular flux of Ruhex is detected by SECM, as evident by the increased Faradaic current over the nanopores.

**Park Systems is a world-leading manufacturer of atomic force microscopy (AFM) systems with a complete range of products for researchers and industry engineers in chemistry, materials, physics, life sciences, and semiconductor and data storage industries. Park's products are used by over a thousand of institutions and corporations worldwide. Park's AFM provides highest data accuracy at nanoscale resolution, superior productivity, and lowest operating cost thanks to its unique technology and innovative engineering. Park Systems was founded in 1997 and holds over 32 patents related to AFM technology, including True Non-Contact Mode™ using decoupled XY and Z scanners, PTR measurements for data storage application, NX-Bio technology using Scanning ion conductance microscopy (SICM) on live cell, 3D AFM, and fully automated AFM operation software (SmartScan™). Please visit <http://www.parkafm.com> or call 408-986-1110 for more information.**

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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



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