

NEURO MORPHIC COMPUTING –HOW MATERIALS AND DEVICES WITH “BRAIN LIKE” ARCHITECTURE ARE REVOLUTIONIZING COMPUTING - AN INTERVIEW WITH DR. ALAIN DIEBOLD

p. 8

CALL FOR PAPERS: 2018 NANOSCIENTIFIC SYMPOSIUM ON SPM AT SUNY POLYTECH p.6

USING PINPOINT™ PIEZOELECTRIC FORCE MICROSCOPY

p. 17

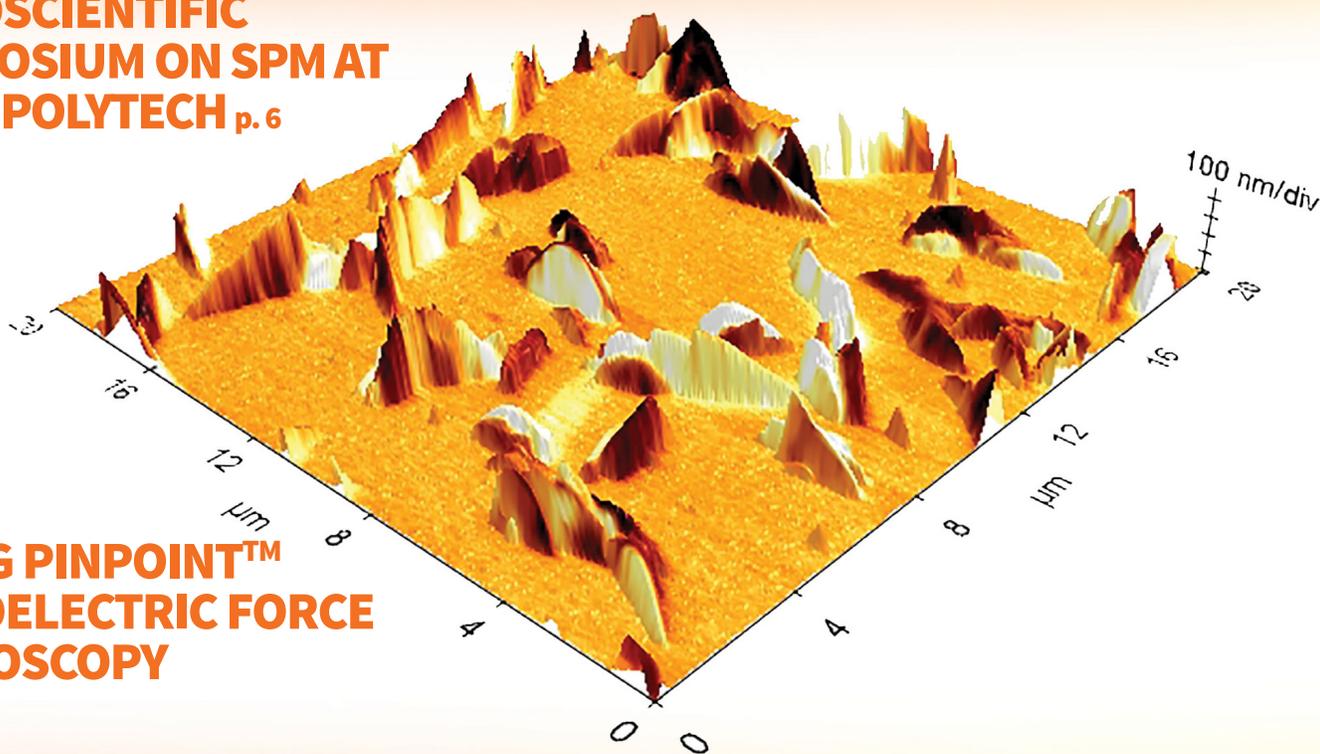
ELECTRICAL CONDUCTIVITY MEASUREMENT OF CARBON NANOTUBES FILM USING CONDUCTIVE PROBE ATOMIC FORCE MICROSCOPY (CP-AFM)

p.10

TRANSFORMING TECHNOLOGY AND MANUFACTURING IN THE AGE OF 3-D PRINTING

p. 20

60 GRAMS FROM ASTEROID BENNU MAY REVEAL THE HISTORY OF OUR SOLAR SYSTEM - AN INTERVIEW WITH DR. MICHAEL DALY p. 13





The Most Accurate Atomic Force Microscope

Park NX10 the quickest path to innovative research

Better accuracy means better data

Park NX10 produces data you can trust, replicate, and publish at the highest nano resolution. It features the world's only true non-contact AFM that prolongs tip life while preserving your sample, and flexure based independent XY and Z scanner for unparalleled accuracy and resolution.

Better accuracy means better productivity

From sample setting to full scan imaging, measurement, and analysis, Park NX10 saves you time every step of the way. The user friendly interface, easy laser alignment, automatic tip approach, and analysis software allow you to get publishable results faster.

Better accuracy means better research

With more time and better data, you can focus on doing more innovative research. And the Park NX10's wide range of measurement modes and customizable design means it can be easily tailored to the most unique projects.

To learn more about Park NX10 or to schedule a demo, please call: +1-408-986-1110 or email inquiry@parkafm.com

www.parkAFM.com



Park
SYSTEMS

TABLE OF CONTENTS

NanoScientific Vol 13 Spring 2018

Message from Editor 5

IN THE NEWS Call for Papers for NanoScientific Symposia on SPM at SUNY Polytech Institute and Technical University Frieberg 6

Feature Article: Neuro Morphic Computing – How Materials and Devices with “brain like” Architecture are Revolutionizing Computing- an interview with Dr. Alain Diebold *Interim Dean at the College of Nanoscale Science at SUNY Polytechnic Institute* 8

Application Note: Electrical Conductivity Measurement of Carbon Nanotubes Film using Conductive Probe Atomic Force Microscopy (CP-AFM) *John Paul Pineda, Gerald Pascual, Byong Kim, and Keibock Lee* 10

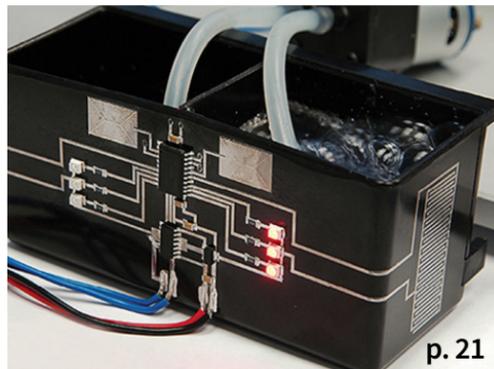
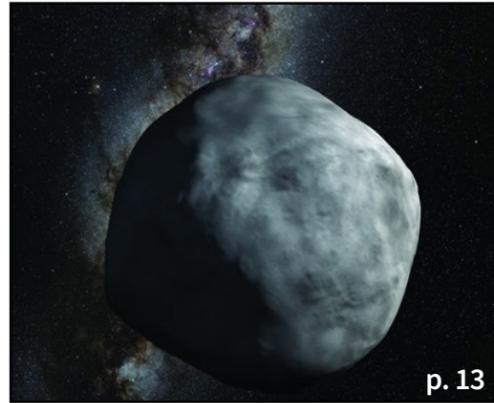
Feature Article: 60 Grams from Asteroid Bennu May Reveal the History of our Solar System- an interview with Dr. Michael Daly, *Associate Professor and York Research Chair in Planetary Science Department of Earth and Space Science & Engineering York University* 13

Application Note: PinPoint Piezoelectric Force Microscopy *Wenqing Shi, Cathy Lee, Gerald Pascual, John Paul Pineda, Byong Kim, Keibock Lee* 17

Feature Article: Transforming Technology and Manufacturing in the Age of 3-D Printing, *Alejandro H. Espera, Jr., MSc, Dr. Rigoberto Advincula, Advincula Research Group, Department of Macromolecular Science and Engineering at Case Western Reserve University* 20

Park AFM Scholarship Winners Two new AFM Scholars Announced 23

Application Note: Using AFM PinPoint™ Nanomechanical Mode for Quantification of Elastic Modulus in Materials Two Orders of Magnitude Faster than Force Volume Spectroscopy *John Paul Pineda, Gerald Pascual, Byong Kim, and Keibock Lee* 26



NANOscientific

www.nanoscientific.org

Keibock Lee, Editor-in-Chief
kei@nanoscientific.org

Deborah West, Content Editor
debbie@nanoscientific.org

Debbie Bishop, Art Director

Gerald Pascual, Digital Media & Advertising Manager
gerald@nanoscientific.org

Published by Park Systems, Inc.
3040 Olcott St. Santa Clara CA 95054
inquiry@parkafm.com, 408-986-1110

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.

We would enjoy hearing from you, our readers. Send your research or story ideas to debbie@nanoscientific.org.

For inquiries about advertising in NANOscientific, please contact Gerald Pascual at gerald@nanoscientific.org.

INSET PHOTO ON COVER:

This image shows a 3D overlay of a piezoelectric response map and topographical data acquired using PinPoint piezoelectric force microscopy (PFM) from an annealed phenanthrene thin film on top of an ITO surface. This material has been a challenging sample to get quality topographical and piezoelectric response data from using conventional SPM methods. The main difficulty is due to the rod-shaped nanostructures on the sample surface being very susceptible to displacement by a scanning probe's tip. The invention of Park's latest PinPoint PFM technique gives researchers both

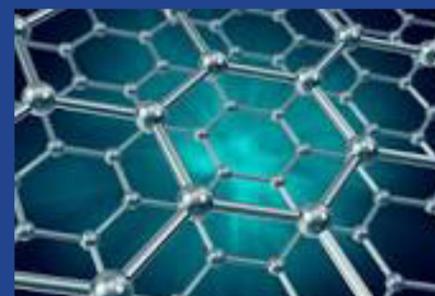
a friction-less imaging technology that overcomes this difficulty and the means to achieve publication-ready image quality in much less time than previously possible with older methods. In this example, not only can we see well-resolved individual rod-shaped phenanthrene structures, but also differences in electrical polarization expressed as differences in contrast (brighter areas showing a positive polarization and darker areas a negative polarization).



Image: NASA

First Test of Graphene in Space-like Applications

Working with Graphene Flagship and European Space Agency, researchers from the Cambridge Graphene Center tested graphene in microgravity conditions for the first time. Using graphene in loop-heat pipes, pumps that move fluid without the need for mechanical parts, a metallic wick was coated in graphene improving efficiency of the heat pipe. Graphene's excellent thermal properties improve the heat transfer from the hot systems into the wick and the porous structure of the graphene coating increases the interaction of the wick with the fluid, and improves the capillary pressure, meaning the liquid can flow through the wick faster. In other experiments researchers looked at use of the material for the improvement of space propulsion or solar sails for fuel-free spacecraft (pictured above) and thermal management systems. The Graphene Flagship, launched by the European Union in 2013 as part of its largest research initiative ever. With a budget of €1 billion, their overall goal is to take graphene from the realm of academic laboratories into European society. www.graphene-flagship.eu



Keibock Lee, Editor-in-Chief

MESSAGE FROM EDITOR

Greetings!

We are excited to bring you our Spring 2018 edition of **NanoScientific** with a tremendous display of how Nanotechnology is quickly advancing science into new realms. Dr. Ennio Capria, Deputy Head of Business Development, IRT NanoElectronics states in his welcome message for the 21st International Conference on Advanced Nanoscience and Nanotechnology to be held in London in June, “Nanoscience is everywhere. Although incredible advances occurred in the last 3 decades, a lot remains to unveil.”

In this issue we unveil one of the most exciting developments in semiconductors, the age of neuromorphic chips that mimic neuro-biological architectures present in the human nervous system. With the ability to learn on-the-fly and process the extreme amounts of data needed to create the 'implanted memory' for human-like machine brains, these chips revolutionize what we know as computer technology. Major companies like IBM have defined cognitive computing as their main business for the future and Intel Labs has developed a neuromorphic research chip, code-named "Loihi," that mimics the functioning of neurons and synapses in the brain. Neuromorphic technology can be used in a wide range of consumer and business products, from driverless cars to domestic robots. In this issue, Dr. Alain Diebold from SUNY Polytechnic gives us an overview of the latest semiconductor revolution and how SUNY is conducting cutting edge research on the material design architecture.

We also present an article on NASA's project OSIRIS-REx — the first-ever sampling mission by NASA to the distant asteroid Bennu. This mission will give us a glimpse into the

formation of our solar system and important discoveries about asteroids, one of the hottest topics in space. NASA is also moving forward with a plan to develop a refrigerator-sized spacecraft capable of deflecting asteroids and preventing them from colliding with Earth and companies like Aten Engineering aim to be first with ideas that could shape the future of asteroid mining.

This issue also talks about another revolution poised to explode in the near term, 3D printing, already transforming industries and becoming 50 percent cheaper and up to 400 percent faster, it could reach \$49 billion as soon as 2025 and already well underway. For example, 3D printed food is already a reality on Earth and in space. Beehex, an American startup, has received a grant from NASA to develop a food 3D printer to allow astronauts to produce their own food during long-term space missions in order to go to Mars.

As always, we feature technical application notes in this issue that highlight new techniques in Nanometrology, the nanoscale imaging that enables scientists to visualize at the atomic scale. In this issue, we showcase Electrical Conductivity Measurement of Carbon Nanotubes and PinPoint Piezoelectric Force Microscopy.

To continue collaboration on the new nanotech innovations world-wide, NanoScientific is hosting NanoScientific Symposia which will feature leading academic and industry presentations and an opportunity to present your research and network with industry leaders. The first NanoScientific Symposium on SPM is Sept 19-20 at SUNY Polytechnic Institute and the second will be hosted by Technical University Freiberg October 10-12. We encourage you to submit an abstract to present at the NanoScientific Symposia and share your amazing Nanoscience discoveries!

For details on these two events go to www.parksystems.com/2018spm for the US and www.parksystems.com/nsfe2018 for Europe.

We would enjoy hearing from you, our readers. Send your research or story ideas to Debbie at Debbie@nanoscientific.org and let us know if you are interested in sponsoring or attending our NanoScientific Symposia. You can also visit our new website at www.nanoscientific.org. We hope you enjoy this issue.

Keibock Lee
Editor-in-Chief



2018 NanoScientific Symposium

Scanning Probe Microscopy

September 19-20, 2018 • SUNY Polytechnic Institute, Albany, NY

CALL FOR PAPERS

THE NANOSCIENTIFIC JOURNAL ANNOUNCES ITS CALL FOR PAPERS FOR THE 2018 NANOSCIENTIFIC SYMPOSIUM ON SCANNING PROBE MICROSCOPY AT SUNY POLYTECHNIC INSTITUTE

Abstract Submission Deadline — June 15, 2018

Park Systems and NanoScientific Publications are proud to announce the

2018 NANOSCIENTIFIC SYMPOSIUM ON SCANNING PROBE MICROSCOPY (SPM)

A new venue for nanoscience researchers, scientists, and engineers to learn about the latest studies being formed using SPM. Keynote speakers from both academia and industry will be on hand to talk about the current cutting-edge work being performed in their laboratories and discuss the headway they have made with SPM in some of the hottest fields and topics in nanoscience today.

Do not miss your chance to join this great opportunity to learn and network with some of the best and brightest in materials characterization!

The first day, Wednesday, September 19, will be composed of keynote speakers and presenters on a variety of topics including the following:

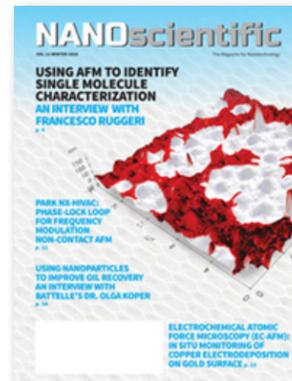
- 2D and other nanomaterials
- Polymers and composites
- Electronics, magnetics, and photonics
- Sustainable energy applications
- Semiconductor and MEMS process and fabrication
- Analytical chemistry
- Biology, biomedicine, and other life sciences

The evening networking event will include cocktails and hors d'oeuvres.

The second day, Thursday, September 20, will focus on hands-on programming:

A theory and practical class on AFM with access to live systems at the Park Nanoscience Center at SUNY Polytechnic Institute.

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



**REGISTER ONLINE TODAY AT:
<http://parksystems.com/spm2018>**

*Sponsored by Park Systems & NanoScientific Magazine at the Park Nanoscience Center
At SUNY Polytechnic Institute 257 Fuller Road Albany NY*



IMPORTANT DEADLINES

Registration for the conference is open until September 30

Abstract / image submission is open until June 30



2018 NanoScientific Forum Europe
Scanning Probe Microscopy (SPM)

NSFE 2018

10-12 October, 2018
TU Bergakademie Freiberg, Germany

Sponsored by Park Systems, TU Bergakademie Freiberg & NANOScientific Magazine



CALL FOR PAPERS

The NanoScientific Journal announces its Call for Papers for the 2018 NanoScientific Forum on Scanning Probe Microscopy (SPM) in Europe (NSFE 2018) at the Technical University Freiberg October 10-12, 2018.

TU Bergakademie Freiberg (TU Freiberg), Institute of Mechanical Process Engineering and Mineral Processing host of the 1st NanoScientific Forum Europe 2018 (NSFE 2018) will give a special session during the scientific program on nanobubbles, which is a part of the flagship project of TU Freiberg and Helmholtz Institute Freiberg for Resource Technology. The special session on nanobubbles will cover the influence of nanobubbles in engineering processes like melt filtration (CRC 920, a flagship project of TU Freiberg) and flotation (SPP2045, TU Freiberg and Helmholtz Institute Freiberg for Resource Technology).

This 2 Day Event will include lectures by renowned AFM researchers, Instrument workshops on Park Systems AFMs, including basic and advanced measuring techniques as well as tips and tricks, how to obtain stunning AFM data.

Wednesday Evening: Fusing Science & People - Conference Gala Dinner

Thursday Evening: Discovering Natural Treasures - terra mineralia Tour & Party

CONFERENCE TOPICS

Application:

- Geoscience and sustainable energy applications
- Polymers and composites
- Nanoelectronics, photonic and photovoltaic applications
- Nanomaterials and Life Science
- Special Session nanobubbles

Method:

- Nanomechanical and Electrical Characterization
- Characterization Techniques in Aqueous Solution
- Advanced Imaging

NANOScientific Publications announces the **2018 NANOSCIENTIFIC SYMPOSIUMS ON SCANNING PROBE MICROSCOPY (SPM)** new venues for nanoscience researchers, scientists, and engineers to learn about the latest studies being formed using SPM. Keynote speakers from both academia and industry will talk about cutting-edge work being performed in their laboratories and the hottest topics in nanoscience today - sponsored by NanoScientific and Park Systems. Poster and Oral Presentation Opportunities, submit your abstract today.

NanoScientific Symposium on SPM in Europe – Oct. 10-12, 2018 at Frieberg University
www.parksystems.com/nsfe2018

NanoScientific Symposium on SPM in US – Sept 19-20 at SUNY Polytechnic Institute
www.parksystems.com/2018spm

**REGISTER ONLINE TODAY AT:
<http://parksystems.com/nsfe2018>**

*Sponsored by NanoScientific, Park Systems & Technical University Freiberg
October 10-12, 2018*



NEUROMORPHIC COMPUTING – HOW MATERIALS AND DEVICES WITH “BRAIN LIKE” ARCHITECTURE ARE REVOLUTIONIZING COMPUTING-

AN INTERVIEW WITH DR. ALAIN DIEBOLD INTERIM DEAN AT THE COLLEGE OF NANOSCALE SCIENCE AT SUNY POLYTECHNIC INSTITUTE AND NATHANIEL CADY, PHD, ASSOCIATE PROFESSOR OF NANOBIO SCIENCE SUNY POLYTECHNIC INSTITUTE

Caption: Dr. Alain Diebold with students at SUNY Polytech Institute. Dr. Diebold's group has continuously been at the cutting-edge of nanometrology since its inception. Other research interests include materials characterization, metrology and materials science at the nanoscale, and semiconductor metrology. The group is also investigating the impact of substrate interactions on the complex refractive index of graphene. Another research area is the imaging and characterization of nano-scale structures using electron microscopy. Simulation of transmission electron microscopy (TEM) and scanning TEM (STEM) imaging is more important than ever as aberration corrected microscopes are introduced.



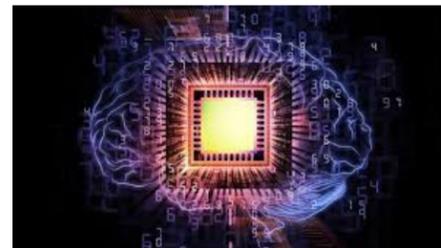
Dr. Alain Diebold is Interim Dean at the College of Nanoscale Science at SUNY Polytechnic Institute, Empire Innovation Professor of Nanoscale Sciences, and Executive Director, Center for Nanoscale Metrology. Dr. Diebold earned his BS in chemistry from Indiana University-Purdue University, and holds the PhD in Statistical Mechanics of Gas-Solid Surface Scattering earned at Purdue University. He is Associate Editor of the IEEE Transactions on Semiconductor Manufacturing as well as the Metrology Section of Future FAB International. A frequent presenter at international conferences, Dr. Diebold has been named a Fellow of both the International Society for Optics and Photonics (SPIE) and the American Vacuum Society (AVS).

Since 2005, Dr. Diebold has presented at over 45 leading conferences on Frontier Metrology

and Characterization for Nanoelectronics. He is recognized industry-wide as a leader in cutting edge research on advanced metrology methods to improve nanoelectronics.

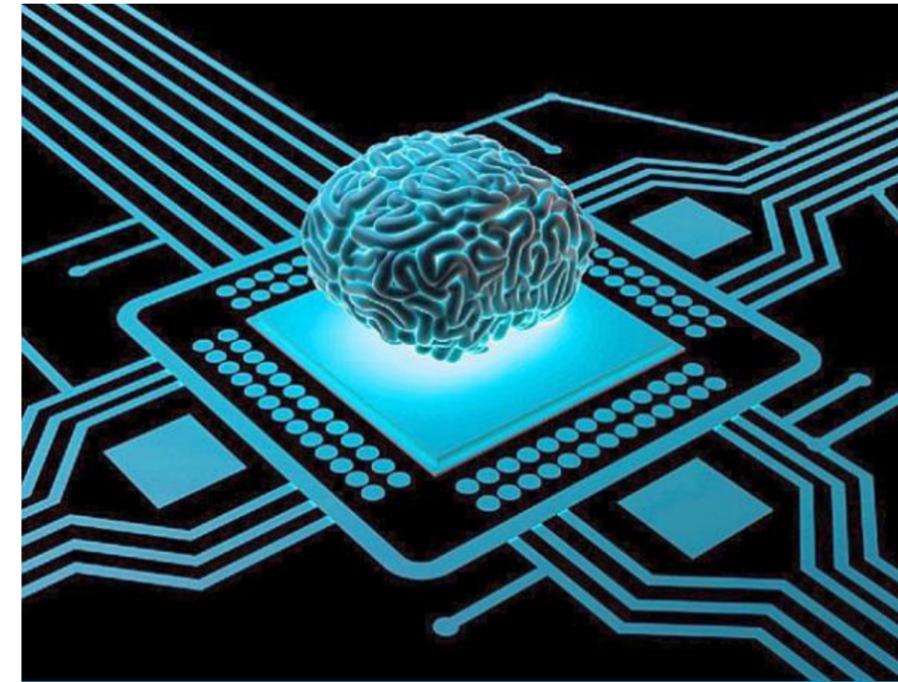
Dr. Diebold has published close to a hundred research papers and made over 40 presentations world-side. He has established a long career in nanometrology and nanoscale semiconductors and has done continuous work for decades with industry to collaborate on the development of new methods and technology.

His books include *Handbook of Silicon Semiconductor Metrology*. He is co-editor of *Frontiers of Metrology and Characterization for Nanoelectronics, Characterization and Metrology for ULSI Technology, Semiconductor Characterization: Present Status and Future Needs, Analytical and Diagnostic Techniques for Semiconductor Materials, Devices, and Processes*.



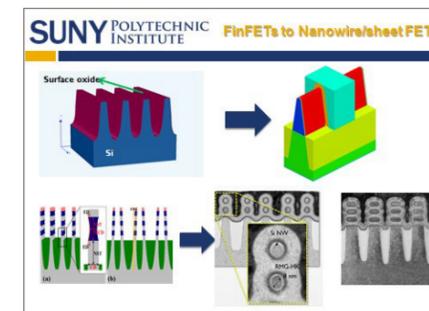
Neuro Morphic Computing – How Materials and Devices with “brain like” Architecture are Revolutionizing Computing

Nanometrology Advances and Challenges
When asked what the latest in new designs for more complex device structures and advanced new materials is, Dr. Diebold explains that Nanowire type transistors fabricated from multi-layer thins are emerging as the news technology. In the past, finfet was made from silicon. Silicon nanogratings with fin-like nanogroove arrays have been used in



“WE ARE DEVELOPING A NEW FORMAT OF NEUROMORPHIC HARDWARE, LEVERAGING UNIQUE PIECES OF HARDWARE TO BUILD A HYBRID SYSTEM,” SAID DR. CADY. “THEY ARE LIKE ELECTRONIC DEVICES THAT MIMIC SYNAPSES IN THE BRAIN.” THE GOAL OF THE RESEARCH IS TO HAVE A BETTER ABILITY TO DO THINGS AUTONOMOUSLY SUCH AS AUTONOMOUS CARS FOR INSTANCE AND DEVELOP SPECIALIZED HARDWARE THAT REDUCES POWER USAGE COMPARED TO THE TRADITIONAL SOFTWARE APPROACH.

nanoelectronics to build field effect transistors (FinFETs), which have attracted enormous attention due to their superior electronic properties. They can also be used in photonic systems to achieve desired linear and nonlinear optical functionalities.



But more recently multi-layer Silicon Nanowire and Silicon Nanosheets with gates all around the transistors and a lot of 3D structure at being researched. “It is very difficult to do the metrology,” explains Dr. Diebold. “And it is hard to predict when or if this will be commercialized.” There are metrology challenges with Nanowires is the Dielectric gate, spaces, and wires. There are advances in all of the typical nanometrology tools especially TEM. One of the key factors in today’s nanometrology research is automated TEM analysis dual column; it is becoming more prevalent as a critical new advancement due to much higher throughput characterization. One of the most significant future trends in the semiconductor industry as Dr. Diebold looks into the future are automated TEM analysis that feed into hybrid metrology. “The goal of Hybrid Metrology is to use the measurement information from multiple methods to improve

3D determination of feature shape and dimensions,” explains Dr. Alain Diebold. AFM enables the determination of surface and sidewall roughness and feature line shape and is often used in conjunction with TEM, CD-SEM, and Scatterometry in Hybrid Metrology.

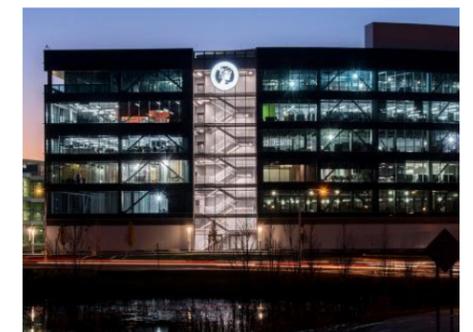
The von Neumann architecture is replaced with neuro morphic architecture

The semiconductor industry is definitely going in a new direction explains Dr. Diebold. He does not see the future of silicon chips

continuing to scale the way they have for the last twenty years. “Silicon chips for the future are going to be more functional, computing like the brain,” he explains. “Neuro Morphic computing is a new way of computing with different architecture to act neuromorphic.”

At SUNY, research on neuro morphic computing is well underway. In Jan of 2016, Dr. Nathaniel Cady at Associate Professor of Nanobioscience at SUNY Polytechnic Institute was awarded \$1.2 million from the Air force Research Lab as part of a \$2.4 million grant in collaboration with the University of Tennessee, Knoxville enabling the fabrication and testing of a dynamic, adaptive neural network based on memristors, cutting edge technology which could one day lead to faster, more energy efficient and powerful computer capabilities.

The extra computing power of neuromorphic computing comes at a time when industry has hit a limit and won’t be able to continue to make the leaps in speed and density they did over the past decades. “Neuro computing augments the chip by building unique hardware



that adds functionality,” explains Cady.

The neuromorphic computing market is poised to grow rapidly over the next decade to reach approximately \$1.78 billion by 2025. For the Air Force grant, they are creating unique, non standard materials for neuro synapses. Professor Cady said that he does not characterize what they are doing as AI (artificial intelligence). “The end game is to create a whole brain, by starting with a small set of neurons,” said Cady.



Dr. Cady who holds a PhD in microbiology from Cornell University specializes in research at the interface of nanotechnology and biology. Dr. Cady’s research uses inspiration from natural, biological systems to guide the design, formulation, and/or fabrication of unique systems and technologies.

ELECTRICAL CONDUCTIVITY MEASUREMENT OF CARBON NANOTUBES FILM USING CONDUCTIVE PROBE ATOMIC FORCE MICROSCOPY (CP-AFM)

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

Abstract

Electrical conductivity measurement is an effective approach to describe how a material behaves for certain applications, ranging from energy storage and energy conversion devices, to interconnections in molecular electronics and nanometer-sized semi-conductor devices. A technique known as Conductive Probe Atomic Force Microscopy (CP-AFM) is a powerful technique that provides accurate nanoscale measurement and mapping of relative difference in electrical conductivity of advanced materials such as CNTs film. Several characterization techniques were introduced in the past decade to study these materials, however, the majority of these can only measure a limited electrical properties range. In this study, Park NX20 equipped with CP-AFM was used to investigate 3 different materials with a wide range of electrical conductivity. The data acquired in this experiment clearly demonstrates the ability of this technique in measuring a wide range of electrical conductivity and differentiating surfaces of materials covered with various types of conductive materials, with the use of a logarithmic current amplifier integrated in the system.

Introduction

Carbon Nanotubes (CNTs) have attracted a great deal of scientific attention and industrial interest world-wide due to its unique electrical behavior [1, 2]. A number of different applications ranging from energy storage and energy conversion devices, to interconnections in molecular electronics and nanometer-sized semi-conductor devices were demonstrated over the past decade [3, 4]. CNTs can behave

as metallic or semi-conducting materials depending on the arrangement of their atoms, their chirality (degree of twist), as well as their sizes (diameter and length) [1, 2]. The electrical conductivity of CNTs plays a major role in describing its behavior and its implication to science and electronics. Therefore, it's critically important to utilize a technique that can effectively measure the electrical conductive properties of these new materials. However, due to their fragile characteristics and nano-scale dimensions, measuring their local properties have become a great challenge to many researchers and device engineers [5, 6]. There are several methods that were introduced to characterize these materials, and the most common are scanning tunneling microscopy (STM), transmission electron microscopy (TEM) and focused ion beams (FIBs). [5, 7, 8] However, some of these techniques are destructive, some have a limited measurement and property characterization modes, others require high vacuum environment. One of the most powerful tools that was designed to overcome these problems is Conductive Probe Atomic Force Microscopy (CP-AFM). This technique provides both electrical properties and topography at the same time, first by monitoring the current flowing between the conductive tip and sample, and the latter by monitoring the cantilever deflection as the tip scans over the sample surface.

In this study, Park NX20 equipped with CP-AFM was used to investigate 3 different materials namely: 1) glass, 2) silver and 3) CNTs film. The results acquired in this experiment clearly demonstrate the ability of this technique to accurately measure a wide range of electrical

conductivity of advanced materials such as CNTs film. In addition, this technique can be used effectively to differentiate regions covered with various types of conductive materials.

Experimental Sample and Probe

The sample that was investigated in this experiment is a CNTs film made of soot of carbon nanotubes that were deposited on a glass substrate to form a thin film layer. Subsequently, an Ag electrode is patterned into the surface to make an electrical contact. A conductive diamond coated probe (NANOSENSORS™ CDT-CONTR) with a nominal force constant of $k = 0.5 \text{ N/m}$ and resonance frequency of $f = 20 \text{ kHz}$ was utilized in the entire test.

CP-AFM Experimental Conditions

The CNT film sample was investigated using Park NX20 AFM system under ambient air conditions to perform a $45 \times 45 \mu\text{m}$ scan outputting to an image size of 256×256 pixels. Figure 1 shows the principles of CP-AFM. The topography and electrical properties of the sample can be acquired simultaneously during operation. The topography data is acquired by monitoring the deflection signal of the cantilever as the conductive tip scans the sample surface in contact. On the other hand, electric conductivity is acquired by measuring the electrical current passing in between the conductive probe and the sample, produced by applying a bias voltage in between the two. The electrical conductivity is measured through an electric current amplifier. In this experiment, the bias voltage that was used is positive 0.3V , since the optimum current

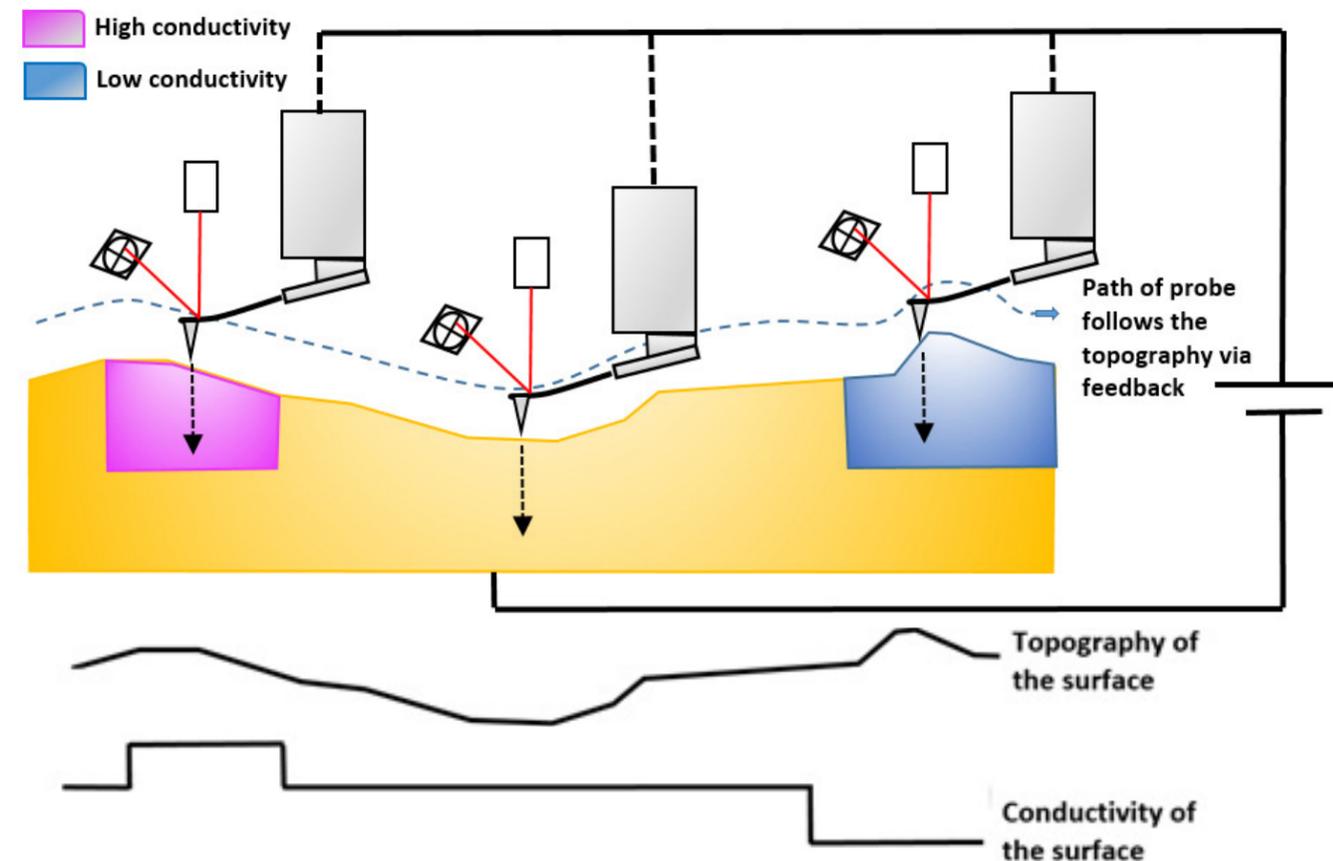


Figure 1. Principles of Conductive Probe AFM. This diagram demonstrates that feedback control from the AFM controller allows this technique to acquire both surface topography and conductivity data simultaneously. The topography data is acquired by monitoring the deflection signal of the cantilever, while electric conductivity is acquired by measuring the tunneling current using current amplifier.

distribution was observed using this value. Generally, the current flow is acquired by a current amplifier and then processed into an image. The Park NX20 is equipped with internal current amplifier with variable gain of 106-1012 V/A, however, since the sample consist of different materials with wide range of electrical conductivity from nearly non conducting $0 \mu\text{A}$ to relatively well conducting $23.18 \mu\text{A}$, a logarithmic amplifier adaptable to this range was utilized in the experiment.

Result and Discussion

The acquired images were analyzed using XEI software developed by Park Systems which mapped the acquired signals to a color table. For topography image, the intensity of the shading correlates to the surface height variation with extremely bright and dark areas having the highest and lowest height regions. Figure 2 shows the topography and current image of the CNTs film sample. The topography data clearly shows that the $45 \mu\text{m}$ by $45 \mu\text{m}$ scanned surface of the sample is composed of areas with various heights which can be

divided into 3 regions. It can be observed that the region with the lowest height has a relatively smooth surface, suggesting that this region is glass substrate. On the other, the two other regions have relatively rough surface, suggesting that these regions are the CNTs and Ag materials. To confirm this, the surface roughness of each regions were calculated using XEI software. The measured surface roughness of the glass substrate was approximately 1.48 nm , while the other two regions have around 14.25 and 14.71 nm . Since the CNTs and Ag has almost the same features, differentiating these two materials will be difficult by simply looking at the topography data.

The current image clearly differentiates the material composition of the sample and it also shows that the surfaces of the sample is divided into 3 regions. The areas with highest conductivity are represented by the red color map, while areas with lower conductivity are represented by the green color map, and for the areas that are non-conductive are represented by brown color map. Among the

3 materials composition of the CNTs film, Ag has the highest conductivity, while CNT has 2nd to the highest, and the glass has the lowest conductivity. (9)

If one were to analyze the corresponding line profile of topography and current image, the region with the highest height in the topography image is the region with the highest conductivity in the current image which is known to be the Ag materials, while the region with 2nd to the highest height and conductivity is the CNTs film, and the lowest is the glass substrate. The quantitative results of conductivity in terms of measured current values are shown in Table 2. The average measured conductivity of Ag is approximately $23.56 \mu\text{A}$, while for CNT is $0.98 \mu\text{A}$, and for glass is $0 \mu\text{A}$. The results were plotted in Figure 3 to better analyze the data. The standard deviations of the electrical conductivity results were also calculated. The glass sample has the lowest standard deviation with $\text{Std} = 0 \text{ A}$, while Ag sample has the highest standard deviation with $\text{Std} = 8$.

Sample	Current (μA)	Std (μA)
Glass	0	0
CNT	0.98	0.94
Ag	23.56	8

Table 2. Measured mean and standard deviation (Std) of current.

Summary

The topography and electrical conductivity of a CNTs film have been characterized using CP-AFM with a Park NX20 AFM system. The data collected in this experiment reveals that this technique can provide qualitative and quantitative information for electrical characterization of advanced materials. Moreover, the results demonstrate that this technique is an effective mean in measuring wide range of electrical conductivity and differentiating surfaces of materials covered with various types of conductive materials, with the use of a logarithmic current amplifier integrated in the system. Overall, the technique described in this study will successfully help researchers and device engineers with key electrical parameters information to better understand the behavior of certain material with unique properties such as CNTs.

Reference

1. Y. Xue, *Experimental Study of Electrical Conductivity of Carbon Nanotube, Nanofiber Buckypapers and Their Composites*.
2. P. Collins, et al., *The Electronic Properties of Carbon Nanotubes*.
3. J. Brito, et al., *Nanostructured films from phthalocyanine and carbon nanotubes: Surface morphology and electrical characterization. Journal of Colloid and Interface Science, Volume 367, Issue 1, 1 February 2012, Pages 467-471*.
4. J. Maklin, *ELECTRICAL AND THERMAL APPLICATIONS OF CARBON NANOTUBE FILMS*
5. Z. Wang, et al., *Measuring physical and mechanical properties of individual carbon nanotubes by in situ TEM. Journal of Physics and Chemistry of Solids 61 (2000) 1025-1030*.
6. M. Li, et al., *Analysis of variance on thickness and electrical conductivity measurements of carbon nanotube thin films. Published 19 July 2016 • © 2016 IOP Publishing Ltd, Measurement Science and Technology, Volume 27, Number 9*.
7. K. Lee, *Failure analysis and the innovative PinPoint™ conductive AFM*
8. G. Homyak, et al., *TEM, STM and AFM as tools to study clusters and colloids. Micron, Volume 29, Issues 2-3, April-June 1998, Pages 183-190. https://doi.org/10.1016/S0968-4328(97)00058-9*
9. A. Helmenstine, *Table of Electrical Resistivity and Conductivity*.

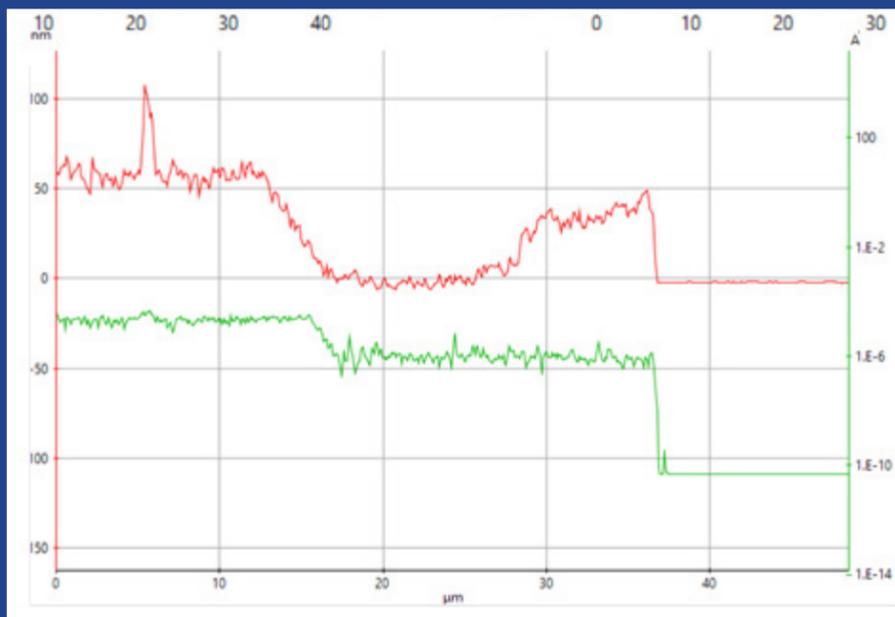
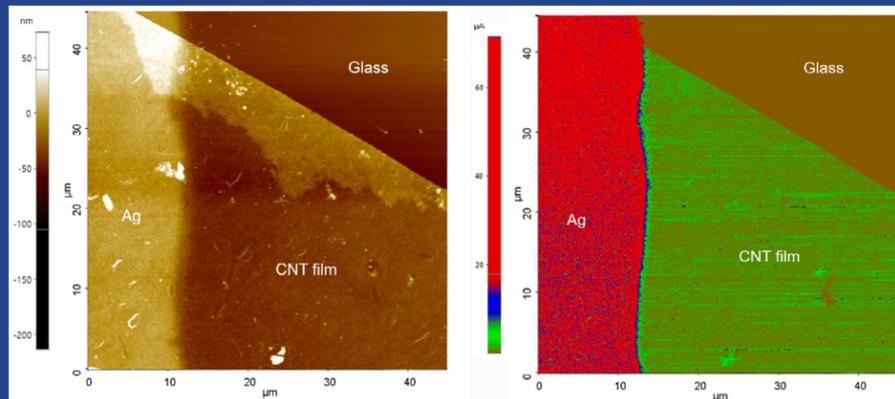


Figure 2. Topography (top-left) and current image (top-right) acquired from CNTs film sample. Scan size: $45 \times 45 \mu\text{m}$, image size: 256×256 pixels., and line and b.) Current image acquired from the CNTs film sample. Topography line profile (red line, y-axis on left) and current line profile (green, y-axis on right).

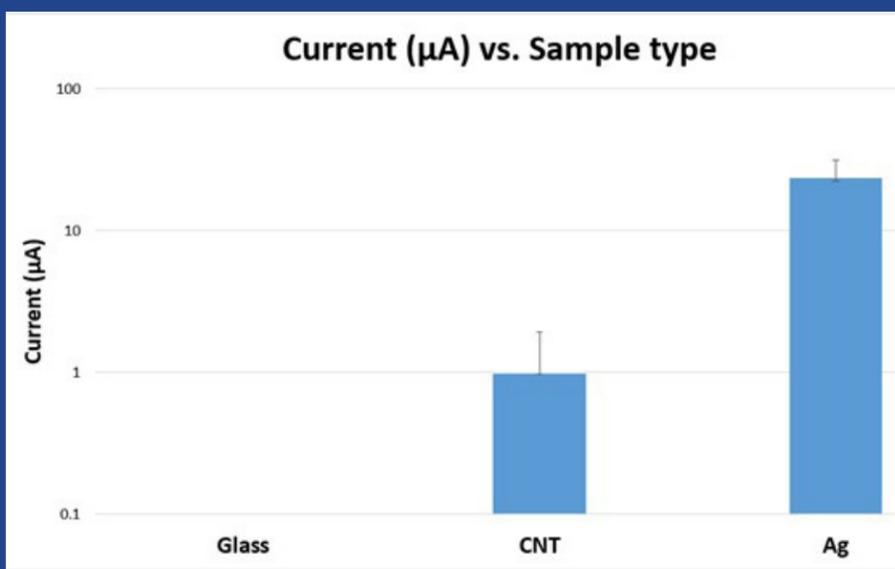


Figure 3. Plotted value of measured mean standard deviation of current.

FEATURE INTERVIEW

60 GRAMS FROM ASTEROID BENNU MAY REVEAL THE HISTORY OF OUR SOLAR SYSTEM

Artist concept of OSIRIS-REx at Bennu, Photo Credit: NASA

As of this week, OSIRIS-REx has travelled over 1.5 billion km since its launch in Sept. 2016. It is currently 47.7 million km from Earth and has a little over .5 billion km left to travel until it reaches the asteroid Bennu.



"THE 60 GRAMS OF PRIMITIVE MATERIAL WE COLLECT FROM BENNU CAN GIVE US CHEMICAL INDICATORS OF LIFE ON OTHER WORLDS"

-Dr. Daly, who leads the science contribution of Canada's OSIRIS-REx Laser Altimeter (OLA) to the NASA New Frontiers mission that was launched in September 2016 toward asteroid 101955 Bennu - the first B-type asteroid to be visited by a spacecraft.

Professor Michael Daly is the York University Research Chair in Planetary Science. He recently received the honor from the International Astronomical Union of having the asteroid 1999 UW25 renamed as (129973) Michaeldaly. He was also the Canadian Aeronautics and Space Institute's 2016 W. Rupert Turnbull lecturer who is selected for his/her association with some significant achievement in the scientific or engineering fields of aeronautics, space-associated technologies or their application. This honor recognized Dr. Daly's contribution to Canadian planetary science mission contributions.

Dr. Daly is currently leading the science contribution of Canada's OSIRIS-REx Laser Altimeter (OLA) to the NASA New Frontiers mission that was launched in September 2016 toward asteroid 101955 Bennu — the first B-type asteroid to be visited by a spacecraft. A scanning laser altimeter, the OLA instrument will measure the range between the OSIRIS-

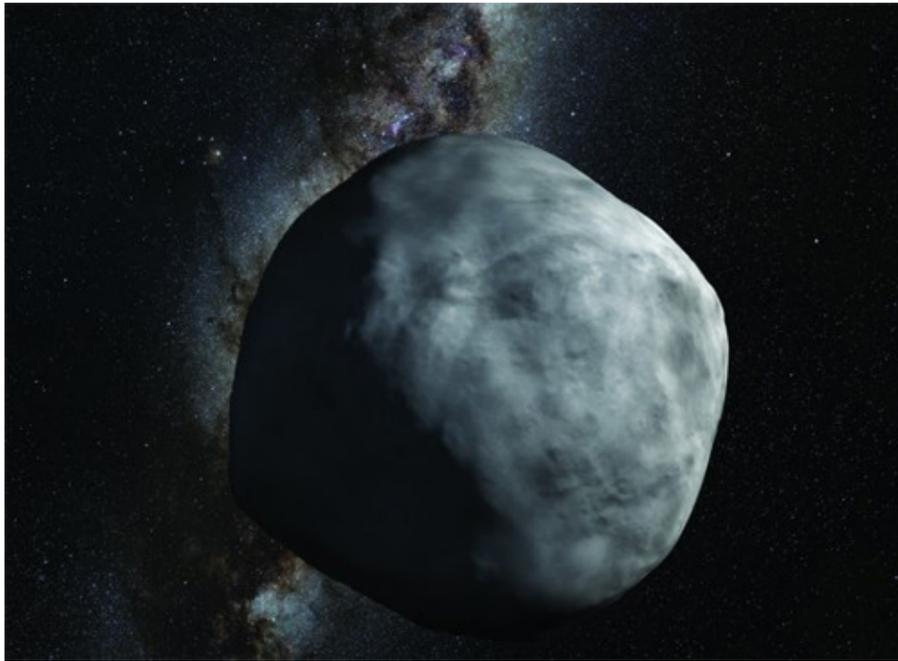
REx spacecraft and the surface of Bennu, to produce digital terrain maps of unprecedented spatial scales for a planetary mission. He also works in the area of deep-UV Raman spectroscopy as well as time-resolved laser-induced fluorescence. He is the PI for a new \$3.5M Canadian planetary science laboratory focused on the simulation and understanding of planetary surface processes as well as developing instruments and improving analysis techniques for planetary science in-situ investigations. Initial investigations will focus on Mars, asteroids and comets.

He is also the Undergraduate Program Director for York's unique Space Engineering and Space Science Programs and has been the acting director for the Centre for Research in Earth and Space Science. Prior to joining York University, he initiated and led the engineering of Canada's first instruments to operate on Mars. These were a two-wavelength atmospheric

lidar that observed snowfall on Mars as well as a temperature and pressure measurement instrument. Dr. Daly has also been the engineering lead for a variety of space-flight cameras including the design of the cameras in the DEXTRE robot's end-effectors aboard the International Space Station. He was awarded a Tier 2 York Research Chair in Planetary Science which enabled his participation in NASA's OSIRIS-REx mission to the near-Earth asteroid Bennu and the return of Canada's first sample of material from another solar system.

60 Grams from Asteroid Bennu May Reveal the History of our Solar System

The asteroid Bennu. OSIRIS-REx — the first-ever sampling mission by NASA to the distant asteroid Bennu — was successfully launched into space on Sept. 8, 2016 from Cape Canaveral Air Force Station in Florida. The mission will revolutionize our understanding of asteroids



The asteroid Benu. Image: NASA'S Goddard Space Flight Center, Greenbelt, Maryland

and the origins of the universe. Benu, which is about 500 meters in diameter, is of particular interest because it is one of the most potentially hazardous asteroids presently identified, with a small chance of hitting Earth in the 22nd century. In Oct. 2018, a 60 gram sample will be collected from Benu and returned to Earth on Sept. 24, 2023. The sample collection, a five-second sampling contact is challenging, but this small sample can tell us about the formation of our solar system. "Benu will help us understand what organic components might be present on the asteroid," said Professor Mike Daly, who is leading the science contribution of Canada's OSIRIS-REx Laser Altimeter (OLA) for this mission. "Bringing a sample back enables us to use instruments a large magnitude better to analyze not just for today but also for the future." Examining Benu could help us to understand more about the solar system and our origin.

OSIRIS-Rex Laser Altimeter (OLA), the most sophisticated laser altimeter ever to visit an asteroid. OLA and the Canadian science team are funded by the Canadian Space Agency. (OSIRIS-REx is the acronym for a the spacecraft's complicated moniker, namely the Origins, Spectral Interpretation, Resource Identification, Security-Regolith Explorer Spacecraft.)

As of this week, OSIRIS-REx has travelled over 1.5 billion km since its launch in Sept. 2016. It is currently 47.7 million km from Earth and has a little over .5 billion km left to travel until it reaches the asteroid Benu. Once the sample arrives back to Earth, a team of scientists world-wide will gain incredible knowledge from it. About 20 percent of the returned Benu

specimen will be studied by the OSIREX-REx team, while four percent is given to Canada for providing the mission's laser altimeter. A half-percent goes to Japan under cooperative agreement with that country's Hayabusa asteroid missions. And three-quarters of the sample will be set aside for future study by instruments not yet invented.



Tim Haltigin, Senior Mission Scientist - Planetary Exploration at the Canadian Space Agency and a member of the OSIRIS-REx mission team answered several questions regarding the OLA instrument and the topographical data it will provide.

Can you tell us about the OLA instrument and how it works?

The OSIRIS-REx Laser Altimeter, or OLA, is a lidar instrument that will scan the entire surface of asteroid Benu to create a high-resolution three-dimensional (3D) map. OLA was contributed to the mission by the Canadian

Space Agency, was built by MacDonald Dettwiler and Associates (MDA), and has a science team led by York University.

Lidars work very similarly to radars, but use light instead of radio waves. A lidar operates by firing short laser pulses that reflect from a target and are subsequently detected by an internal receiver. By very precisely measuring the return transit time of the laser pulse, it can calculate the distance to the target. Repeating these measurements over numerous locations on the asteroid surface allows the user to build up a full 3D model.

To do so, OLA will rely on its two separate transmitters. The High Energy Laser Transmitter (HELT) will be used to scan from distances 1-7.5 km from the asteroid surface, and the Low Energy Laser Transmitter (LELT) will provide rapid measurements at shorter ranges (225m to 1 km).

What kind of topographical data will it be able to provide?

OLA data will be used to create a global shape model at a resolution of approximately one point every 7 cm. Given that Benu is approximately 500 m in diameter, this translates to over 150 million individual topography measurements covering the asteroid's surface.

The global shape model will be used to understand the current state and evolution of the asteroid as well as to provide contextual information for the mission's scientists to interpret geological data collected by other instruments on board the spacecraft, and also to help select candidate locations to retrieve the sample. As the spacecraft gets closer to Benu to further investigate these candidate sampling sites, OLA will be used to produce even higher resolution maps with topography measurements spaced less than 5 cm apart.

Is the OLA used on any other space or earth based missions?

OLA, itself, is a unique instrument. However, its concept has been drawn from previous terrestrial and space examples. Amongst others, airborne lidar systems are widely used on Earth for geological, archeological, agricultural, and ecological applications. Moreover, they can be adapted for use in vision systems for purposes such as robotic mining vehicles.

The OLA system is based on MDA's heritage design of a scanning lidar system flown on the US Air Force Research Laboratories XSS-11 mission. The system was augmented utilizing heritage derived from NASA's Phoenix Mars Lander mission, where a version of the HELT was flown as part of the MET instrument, also built by MDA.



EARLY EARTH Caption: This is an artist's concept of the young Earth being bombarded by asteroids. Scientists think these impacts could have delivered significant amounts of organic matter and water to Earth. The early solar system was quite chaotic. Giant impact craters throughout the inner solar system indicate there may have been a "late heavy bombardment" by asteroids approximately 4.1 billion to 3.8 billion years ago, right around the origin of life on Earth. This asteroid bombardment may have been a significant source of organic matter and water for the early Earth. Credits: NASA's Goddard Space Flight Center Conceptual Image Lab

“THE PROBABILITY THAT THE ORBITS OF BENNU AND EARTH COULD COINCIDE IN A COLLISION IS CURRENTLY 1 IN 2,700 SOMETIME BETWEEN THE YEARS 2175 AND 2193. ONCE WE GAIN A BETTER UNDERSTANDING OF THE YARKOVSKY EFFECT FROM OUR CHARACTERIZATION OF BENNU, WE WILL BE ABLE TO BETTER PREDICT THE TRAJECTORY OF BENNU AND OTHER ASTEROIDS WITH THE POTENTIAL TO COME CLOSE TO EARTH.”

- Dr. Tom Zega, Professor of planetary materials science at the University of Arizona and member of the OSIRIS-REx science team



Dr. Tom Zega, Professor of planetary materials science at the University of Arizona and member of the OSIRIS-REx science team explains the kind of tests being done on the returned sample and the probability of Benu colliding with Earth.

When the sample is returned to Earth, what kind of tests will be done and what primarily will the team be looking for?

We will do a lot of different types of measurements. Generally, we are interested in what materials compose the sample, their textures, their spatial relationships to one another, their crystal chemistry and atomic structures. We will use light, ion, and electron microscopy in addition to mass spectrometry to image the sample, measure its elemental composition, and its isotopic composition. Those measurements will be conducted at scales ranging from centimeters down to the atomic level. Data types will include optical images including reflected and transmitted light, element and isotopic maps in two dimensions, spectra of various types, and high-resolution atomic-scale images.

Some of the instruments we will use include:

SEM = scanning electron microscopy for high-resolution imaging and chemical analysis of the sample.

EMPA = electron microprobe analysis for measuring the chemical composition of the

sample. This gives us quantitative information on material stoichiometry and two-dimensional chemical maps of the sample.

SIMS = secondary ion mass spectrometry for measuring isotopic composition of the sample.

FIB-SEM = for high-resolution imaging, chemical analysis, microstructural analysis, and in situ site-specific extraction of regions of interest in the sample.

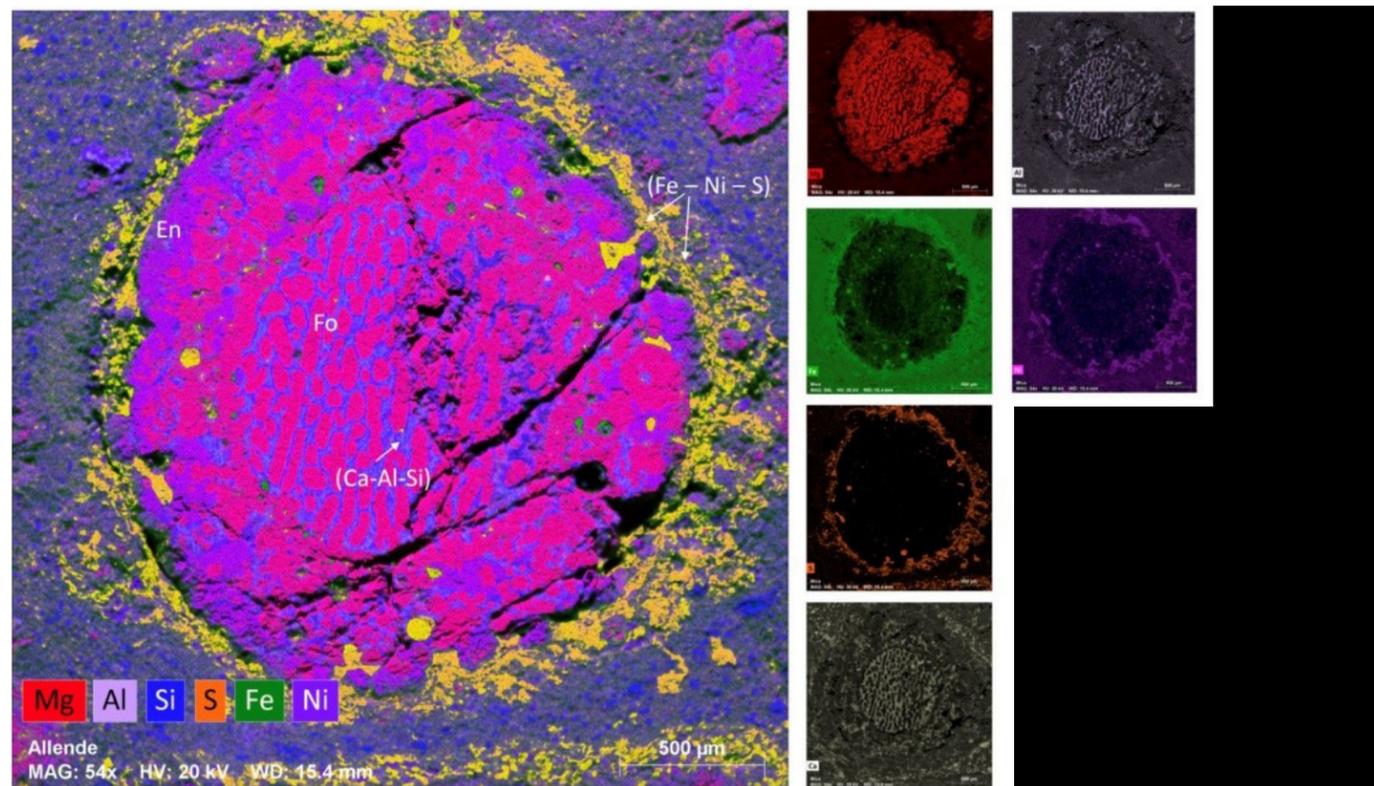
TEM = transmission electron microscopy for highly detailed atomic-resolution analysis and crystal-chemical analysis of the sample.

There will be other instruments used including different types of spectrometers and some others, but these are some of the major types of instruments we will use and analyses we will do.

Is there any update on the possible collision of Benu with Earth?

The probability that the orbits of Benu and Earth could coincide in a collision is currently 1 in 2,700 sometime between the years 2175 and 2193. Luckily, the science we learn from OSIRIS-REx will help us better understand the hazards posed by asteroids and how to mitigate them. One of OSIRIS-REx's goals is to study the Yarkovsky Effect, a force caused by the emission of heat from a rotating object that can slightly change its orbit. These orbit changes make it difficult to predict the path of a small, potentially hazardous asteroid over time.

Once we gain a better understanding of the Yarkovsky Effect from our characterization of Benu, we will be able to better predict the trajectory of Benu and other asteroids with the potential to come close to earth.



Bits of dust are flash heated to molten rock and solidify to become chondrules — some of the building blocks of the solar system. Chondrules clump together via electrostatic and gravitational forces to become asteroids and planets. Chondrules may make up a large part of the material in Benu. By analyzing the sample collected from Benu, the OSIRIS-REx team will be able to examine some of the most pristine material to be found anywhere in the solar system. Benu may also harbor organic material from the young solar system. Organic matter is made of molecules containing primarily carbon and hydrogen atoms and is fundamental to terrestrial life. The analysis of any organic material found on Benu will give scientists an inventory of the materials present at the beginning of the solar system that may have had a role in the origin of life. “The biggest advantage we have now is we can examine much smaller portions of the sample with modern instrumentation with multiple analysis pathways such as morphology and biology and we can separate out very small components of the sample from a nanotechnology standpoint,” said Dr. Daly. “By bringing a sample back to Earth, we can access more information than we could on a space craft and we can analyze the sample with future instrumentation not yet discovered.”

How was Benu chosen?

The goal of the OSIRIS-REx mission is to collect a sample from an asteroid and bring it back to

Earth. But just how did the OSIRIS-REx team choose Benu from the over 500,000 known asteroids in the Solar System?

Proximity to Earth

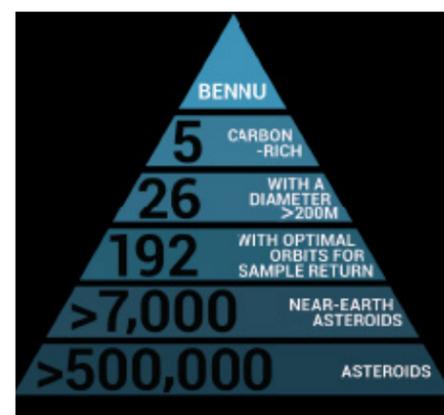
The closest asteroids to Earth are called Near-Earth Objects (NEOs). As the name suggests, NEOs are objects that orbit within 1.3 AU of the Sun. (1 AU = the distance between Earth and the Sun, or ~93 million miles) For a sample return mission like OSIRIS-REx, the most accessible asteroids for a spacecraft to reach are located between 1.6 AU and 0.8 AU. The ideal asteroid has an Earth-like orbit with low eccentricity and inclination. At the time of the mission’s asteroid selection in 2008, there were over 7,000 known NEOs, but only 192 had orbits that met these criteria. Benu Selection

Size

Asteroids with small diameters rotate more rapidly than those with large diameters. With a diameter less than 200 meters, an asteroid spins so rapidly that the loose material on its surface (regolith) can be ejected from it. The ideal asteroid has a diameter larger than 200 m so that a spacecraft can safely come into contact with it and collect a sufficient regolith sample. This size requirement reduced the number of candidate asteroids from 192 to 26.

Composition

Asteroids are divided into different types based on their chemical composition. The most



primitive asteroids are carbon-rich and have not significantly changed since they formed nearly 4 billion years ago. These asteroids contain organic molecules, volatiles, and amino acids that may have been the precursors to life on Earth. Of the 26 asteroids left on the list, only 12 had a known composition, and only 5 were primitive and carbon-rich.

From these 5 asteroids, Benu was selected. Benu is a B-type asteroid with a ~500 meter diameter. It completes an orbit around the Sun every 436.604 days (1.2 years) and every 6 years comes very close to Earth, within 0.002 AU. Benu’s size, primitive composition, and potentially hazardous orbit make it one of the most fascinating and accessible NEOs ... and the ideal OSIRIS-REx target asteroid.

Park
SYSTEMS

PINPOINT PIEZOELECTRIC FORCE MICROSCOPY

APPLICATION
NOTE

WENQING SHI, CATHY LEE, GERALD PASCUAL, JOHN PAUL PINEDA, BYONG KIM, KEIBOCK LEE

PARK SYSTEMS INC., SANTA CLARA, CA USA

Abstract

Electromechanical coupling in materials is a key property that provides functionality to a variety of applications, including sensors, actuators, IR detectors, energy harvesting and biology. Most materials exhibit electromechanical coupling in nanometer-sized domains. Therefore, to understand the relationships between structure and function of these materials, characterization at nanoscale is required. This electromechanical coupling property can be directly measured in a non-destructive manner using piezoelectric force microscopy (PFM), a mode that comes standard in all Park atomic force microscopes (AFMs). Here in this application note, we developed a novel technique termed as PinPoint™ PFM and demonstrated the application of PinPoint PFM in the characterization of annealed phenanthrene film. In addition, the performance of PinPoint™ PFM and conventional PFM was compared, and enhanced resolution was observed in both topography and PFM quad signal via PinPoint™ PFM.

Introduction

Piezoelectric effect, is an intrinsic material property, in which the application of an electric field leads to thickness changes and/or shearing of the material. This unique electromechanical coupling property has been employed in a wide range of applications ranging from medical imaging and energy harvesting, to actuators and sensors.¹ Example of piezoelectric materials include crystals (i.e., quartz), biological materials (i.e., DNA, bones and proteins) and man-made materials such as synthetic ceramics (barium titanate and zinc oxide) and some organic thin films.³

Driven by the developing nanotechnology and the increasing demands for miniaturization of electronic devices, characterization of piezoelectric effect at micro- and nanoscale has attracted significant interest.

Piezoelectric force microscopy (PFM), also termed as dynamic-contact electrostatic force microscopy (DC-EFM) by Park, is an atomic force microscopy (AFM) based method that allows for high-resolution imaging, quantification and manipulation of piezoelectric materials at micron- and nanometer-length scale. Conventional PFM is usually performed in contact mode, and concurrent topographic imaging and piezoresponse measurements is obtained.

In conventional PFM operation, an electrically-biased conductive tip is brought in contact with the surface of a piezoelectric material. Through application of an AC modulation to the conductive tip, the piezoelectric response of the material can then be measured by tracking the deflection of the cantilever as a result of sample’s local expansion or contraction based on the applied electric field. As these surface displacement are often very small with a low signal-to-noise ratio, and, thus, a lock-in amplifier is used for the detection of the amplitude and phase of the piezoelectric response signal. In terms of the AC bias frequency selection, a frequency that’s much lower compared to the cantilever’s resonance frequency is used. In addition, a DC bias can be applied to the sample to switch the domains of the piezoelectric material. Since the atomic force microscopy’s photodiode is position-sensitive, piezoelectric force microscopy can also identify the direction of electrical polarization in piezoelectric or ferroelectric domains. There are two modes of PFM imaging: vertical PFM (VPFM) and lateral PFM (LPFM), which are sensitive to domains polarized out-of-plane and in-plane, respectively.² (Figure 1)

In vertical piezoelectric force microscopy, in the presence of piezoelectric domains that point out-of-plane or perpendicular to the sample surface (Figure 1a-b), the cantilever will deflect vertically with respect to the sample surface in response to the applied electric field. Therefore, the PFM signal will

appear bright for domains that point upward and dark for domains that point downward.

In lateral piezoelectric force microscopy, in the presence of in-plane piezoelectric domains that’s parallel to the surface, a displacement shearing on the surface will occur. As a result, a torsional displacement of the cantilever will be induced, which in turn will be captured by the position sensitive photo detector as a lateral deflection. (Figure 1c-d)

In this application note, piezoelectric force microscopy is performed utilizing the newly-developed PinPoint™ mode by Park Systems as opposed to the conventional contact mode. Performance comparison of PinPoint™ PFM and conventional PFM was carried out on annealed phenanthrene film, and improved resolution was observed in both topography and piezoelectric response signal with PinPoint™ PFM.

In PinPoint™ PFM mode, the AFM probe monitors its feedback signal, approaches toward the sample surface until a predefined force threshold point is reached, measures the Z scanner’s height, then the AFM probe is rapidly retracted away from the surface to a user-defined height. The XY scanner stops during the piezoelectric response acquisition, and the probe-substrate contact time is controlled to allow sufficient time for quality data acquisition (Figure 2). PinPoint PFM allows higher spatial resolution with optimized piezoelectric response measurement over different sample surface. PinPoint PFM was designed to replace the conventional contact PFM and is an enhanced design that eliminates the problems of the tip wearing out during contact mode topography and diminished contact time. The PinPoint™ PFM technology has proven to solve all of the shortcomings of conventional PFM including quick tip wear, degradation of resolution, low signal to noise ratio, and poor reproducibility of data.

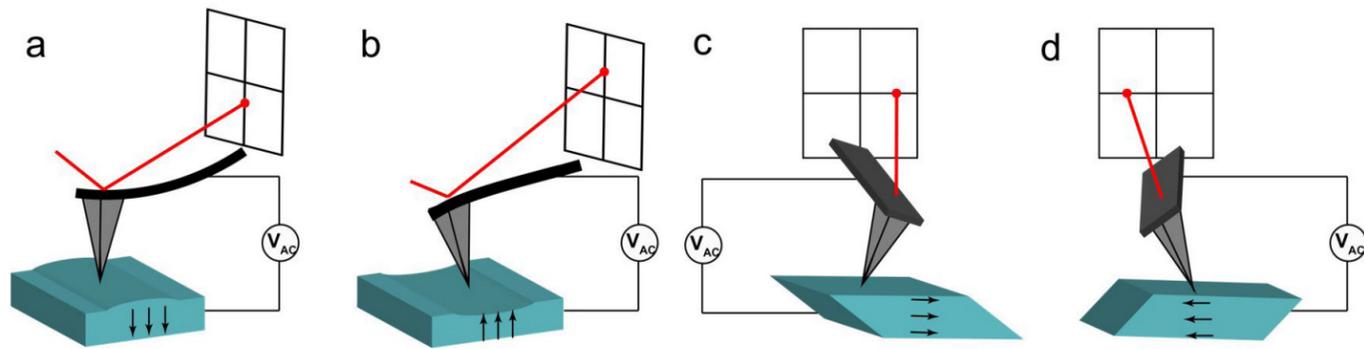


Figure 1. A schematic representation of (a-b) vertical and (c-d) lateral PFM. The AFM laser shows vertical deflections which correspond with (a) downward or (b) upward out-of-plane electrical polarization. In lateral PFM, the cantilever will exhibit torsion in response to (c-d) lateral in-plane polarization directions. Black arrows indicate the direction of polarization vector in each case assuming that the relationship between polarization and crystal orientation is conserved.

Experimental

A Park NX10 AFM was used to image the annealed phenanthrene surface, and the topography signal and PFM quad signal were acquired in both conventional PFM imaging and PinPoint™ PFM imaging. In conventional PFM imaging, a NSC36-C (nominal spring constant $k = 0.6$ N/m and resonance frequency $f = 65$ kHz) coated with Cr and Au on both the front and back side was used in conventional PFM imaging. The nominal radius of the tip curvature is ~ 25 nm. Scan size was $20 \mu\text{m} \times 20 \mu\text{m}$. Scan rate was 0.2 Hz. The AFM tip was biased with AC potential with an amplitude of 4.5 V and a frequency of 17 kHz. No external bias was applied to the sample during imaging. The force set point used in the experiment was 6.84 nN.

In PinPoint™ PFM imaging, a conductive NANOSensorSTPointProbe® Plus-Electrostatic Force Microscopy (PPP-EFM) cantilever (nominal spring constant $k = 2.8$ N/m and resonance frequency $f = 25$ kHz) coated with Ptlr5 on both the front and back sides was used in PinPoint™ PFM imaging. The nominal radius of the tip curvature is ~ 25 nm. Scan size was $20 \mu\text{m} \times 20 \mu\text{m}$. Same as the conventional PFM experiment, a AC potential with an amplitude of 4.5 V and a frequency of 17 kHz was applied to the tip, and no DC potential was applied to the sample. The force set point used in the experiment was 196.8 nN. The retract height was $0.3 \mu\text{m}$. The retract/approach speed was $20 \mu\text{m/s}$. The contact time between the probe and the sample surface was controlled at 1 ms.

Results and Discussion

In Figure 3, the topography and the piezoelectric response maps of the annealed phenanthrene thin film on ITO surface imaged with both conventional PFM and PinPoint™ PFM are shown. The images obtained via conventional PFM are shown in Figure 3a (topography) and Figure 3b (piezoelectric response), and the images

collected by PinPoint PFM are shown in Figure 3c (topography) and Figure 3d (piezoelectric response).

From the topography images (Figure 3a and 3c), the annealed phenanthrene polymer was resolved under both imaging modes and observed to be rod-shaped features with pointy ends. The height of polymer rods was measured to be ranging from 50 to 400 nm, while the width of the polymer rods was measured to be between several hundred of nm to a couple of μm . It is noteworthy that the quality of the topography image obtained via PinPoint PFM is significantly improved compared to that taken with conventional

PFM mode. In Figure 3c, under PinPoint PFM imaging conditions, the annealed phenanthrene polymer were well-distinguished from the ITO substrate. However, in Figure 3a, under conventional PFM imaging conditions, image artifacts can be seen throughout the entire scan area, indicating that the probe was scratching on the surface repeatedly. Of note, for conventional PFM measurement, our initial attempt was to use PPP-EFM ($f = 2.8$ N/m) as the probe, which is the same as the one used in the PinPoint PFM measurements. However, the probe was constantly scratching on the surface and the image quality was very poor. A cantilever with smaller force constant, NSC36-C ($f = 0.6$ N/m) was used instead, in hope to

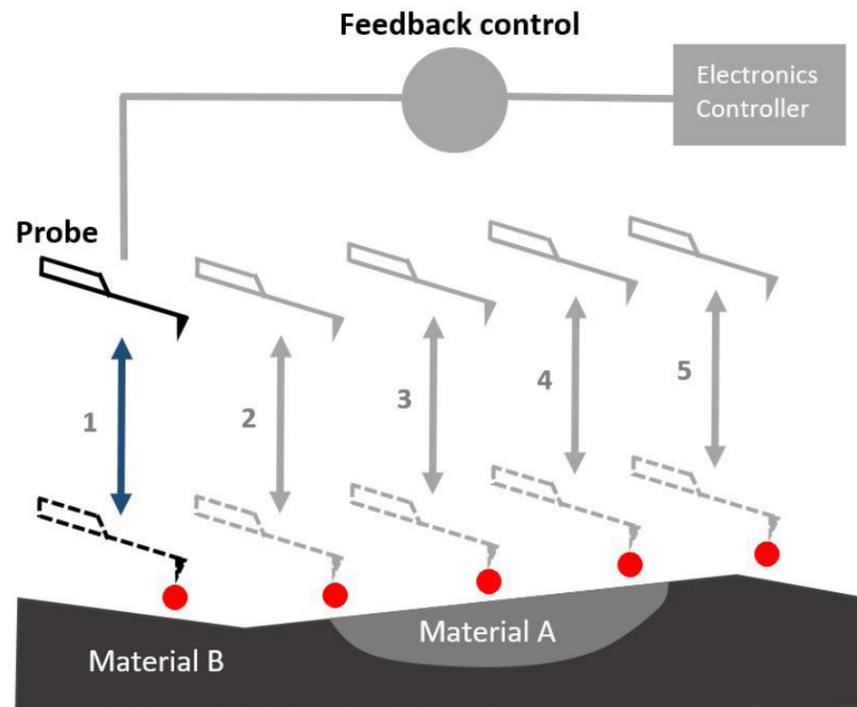


Figure 2. A schematic representation of PinPoint mode operation. The probe approaches towards the sample surface until a pre-defined force threshold is reached, then the Z scanner height is recorded. The XY scanner stops and the piezoelectric response is measured. Then the probe retracts away from the surface and move to the next pixel. The process repeats to collect the topography map and the piezoelectric response map.

alleviate the scraping on the surface. Albeit the NSC36-C has a smaller force constant compared to that of PPP-EFM, the probe was still dragging on the surface and resulted in imaging artifacts as seen in Figure 3a. In PinPoint PFM, on the other hand, since the probe was retracted away from the surface to a safe height at every pixel prior to approach towards the surface, the scratching between the probe and the underlying substrate was effectively eliminated, leading to significantly improved imaging quality.

Similar results were observed in the piezoelectric response images (Figure 3b and Figure 3d). By comparing the results of the two techniques, one can easily determine that PinPoint PFM has better performance compared to conventional PFM in the detection of piezoelectric response as the image obtained under PinPoint PFM was much better compared to that taken with conventional PFM mode.

Figure 4a and 4b are hysteresis curves for the PFM amplitude and PFM phase signals of the phenanthrene film. These hysteresis curves were obtained by measuring the piezoelectric response at a specific location of the sample while applying the sample bias from -1.5 V to $+1.5$ V. The hysteresis curves provide localized information with respect to the switching properties of piezoelectric material. In Figure 4a, a characteristic “butterfly” shape that is similar to the ideal strain versus bias curve was observed in the amplitude signal. In addition, the coercive voltage, which is a measure of ability to withstand an external electric field without depolarization, is ~ 0.3 V. In Figure 4b, the phase hysteresis loop is shown, which is the typical response of a ferroelectric material.

Conclusions

Here in this application note, imaging performance of PinPoint PFM mode and conventional PFM mode was compared on phenanthrene film annealed on ITO surface. The PinPoint PFM mode introduced by Park Systems has proven to offer significantly-improved quality in both topography and piezoelectric response measurement. The advanced PinPoint mechanism eliminates the frictional force between the probe and the substrate, enabling concurrent high-resolution topography and piezoelectric response mapping of the surface. In addition, the response in strain (PFM amplitude) and polarization (phase) as a function of applied voltage was examined to obtain insights regarding material characteristics such as coercive voltage and hysteresis. Taken in total, PinPoint PFM mode is the ideal approach to characterize and quantify localized piezoelectric response at nanoscale, with maximized tip life and sample originality as a result of minimal frictional force between tip and sample.

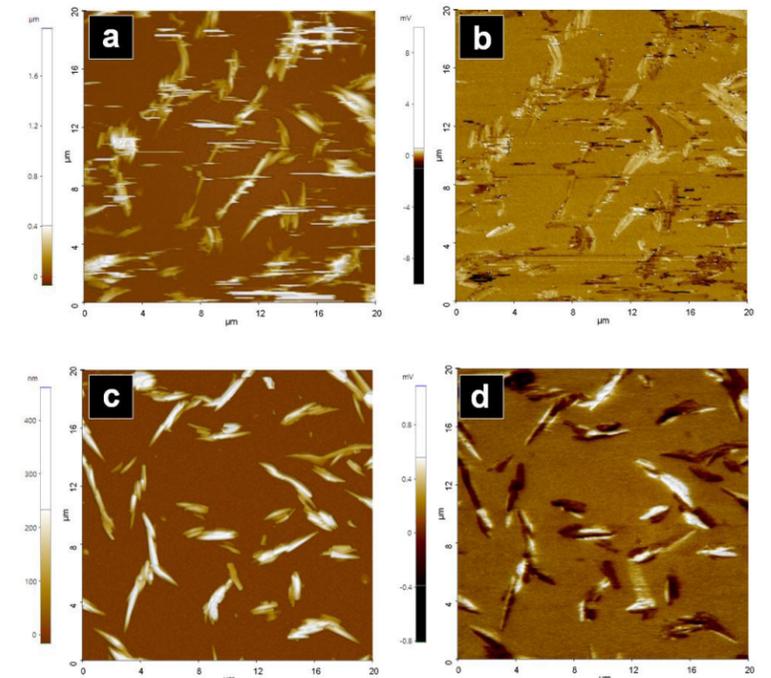


Figure 3. a) Topography image taken with conventional PFM mode; b) PFM quad image taken with conventional PFM mode; c) Topography image taken with PinPoint PFM mode; d) PFM quad image taken with PinPoint PFM mode. Scan size: $20 \mu\text{m} \times 20 \mu\text{m}$.

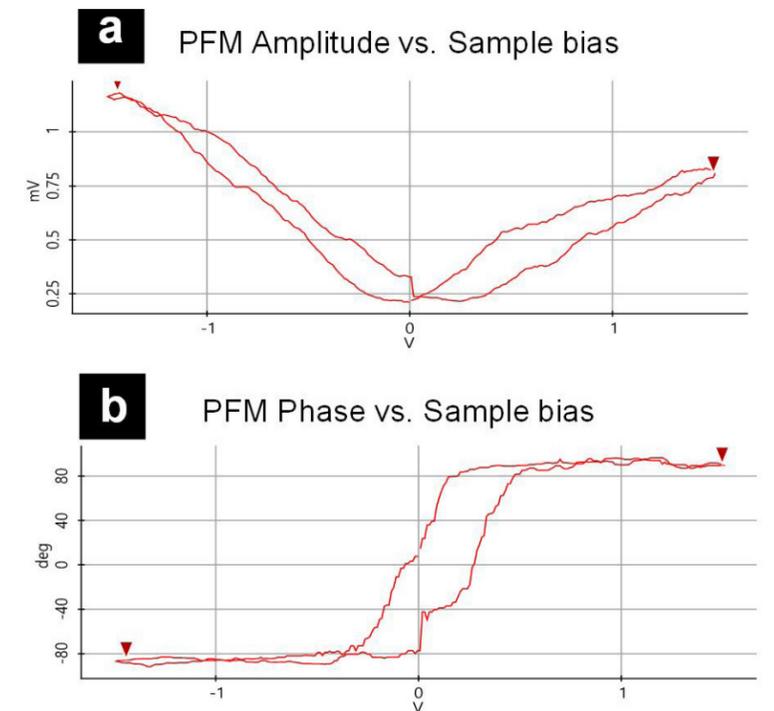


Figure 4. Hysteresis curves for the amplitude (a) and phase (b) signal taken with the phenanthrene polymer film.

References

- Ikeda T. Piezoelectricity. Oxford university press; 1990.
- Soergel E. Piezoresponse force microscopy (PFM). J Phys D Appl Phys. 2011;44(46):464003.
- Vijaya MS. Piezoelectric Materials and Devices: Applications in Engineering and Medical Sciences. CRC Press; 2012.

TRANSFORMING TECHNOLOGY AND MANUFACTURING IN THE AGE OF 3-D PRINTING

ALEJANDRO H. ESPERA, JR., MSC.

“PARK ATOMIC FORCE MICROSCOPY (AFM) PLAYS AN IMPORTANT ROLE IN OUR 3-D PRINTING PROJECTS IN THE LABORATORY. THIS POWERFUL TOOL IS CAPABLE OF LOOKING AT THE SURFACE PROFILE OF 3-D PRINTED OBJECTS CREATED BY DIFFERENT 3-D PRINTING TECHNOLOGIES IN NANOMETER RANGE.”

The 3-D printing industry is being hailed as the next industrial revolution, poised to transform every industry and innovate new ones. New studies point to the 3-D printing industry as one of the fastest growing in our economy. In 2014, the 3-D printing industry was estimated to be at \$4 billion. With new predictions that 3-D printing will be 50 percent cheaper and up to 400 percent faster, the industry could reach \$49 billion by 2025.

But how fast are manufacturers adapting to the new technology, which requires investment and retraining? Experts predict that 93 percent of manufacturers will expand their use of 3-D printing for production parts in the next three to five years and sixty percent of manufacturers expect 3-D printing use to at least double in two to five years.

3-D Printing as a technological feat that started as hobbyists' innovative way of building things has grown into a well-accepted additive manufacturing (AM) technology. It has now penetrated various manufacturing industries such as in electronics manufacturing, bioengineering, food fabrication, and in almost anything we can think of.

3-D printing of electronics

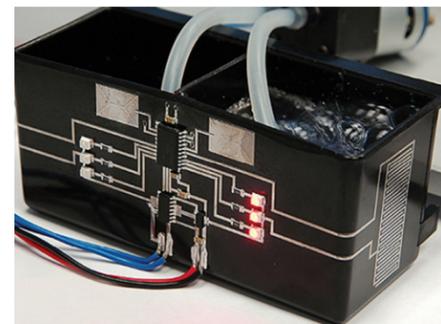
Innovation has paved the way for 3-D printing technologies in the field of electronics fabrication. With the advances in creating new control systems, techniques such as inkjet and aerosol printing are primarily used for fabricating homogeneous structural electronics. 3D printing has shown a great potential in the electronics fabrication industry as well when it comes to fabricating multiple layer complex electronics.

Can 3D printing potentially become the most viable method for mass producing electronics? For now, incorporating the traditional pick-and-place method of building electronics with the latest 3-D printing technologies could be a workable strategy. In this case, to be able to reduce the whole volume of a conventionally-manufactured device, the space occupied and the materials used to build it should also be reduced. In the future, it becomes more and more practical to accurately embed electronic components with conductive interconnects within a three-dimensional substrate as an approach for a more robust and space-saving model.

“According to the 2016 Markets and Markets analysis report, printed electronics, in general, was considered to have a market value of 3.13 billion US dollars at the end of 2015 with a potential to reach 12.10 billion US dollars in the coming decade. But for 3D-printed electronics alone, it was valued 20 million US dollars in 2015 according to Harrop, the director of IDTechEx research firm. Asia Pacific market accounts the largest piece of the market in 2016 where the centers of electronics manufacturing are located in China, South Korea, Japan, and India; therefore, advancing the demand for printed electronics in the said region. Europe, on the other hand, holds the majority of research and development activities, and countries such as UK, Netherlands, and Finland are directing the printed electronics research in the region.”

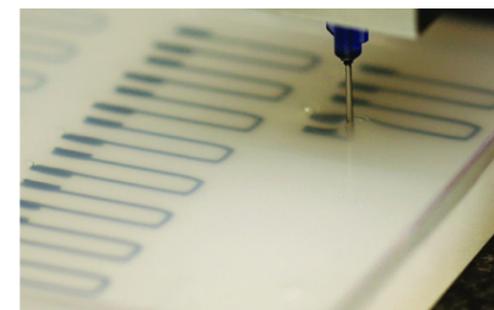
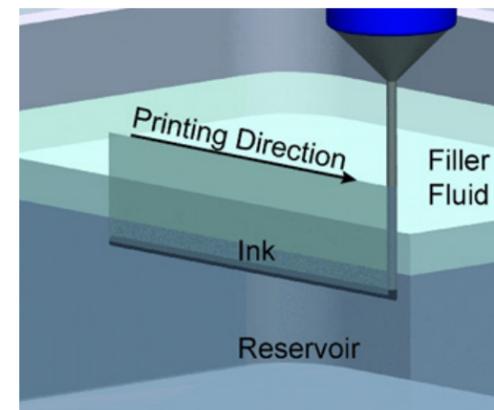
Optomec, the company that pioneered Aerosol Jet printing, has envisioned the idea of mass production of electronics using integrated 3-D printing approach. While most of the aforementioned companies are outsourcing some of their machine parts (e.g. print heads), Optomec manufactures their own using open systems design for compatibility. The print heads are made compatible with most CNC machines, making it adaptable to any integrated 3-D printing systems for any given production settings.

Taking a unique strategy, their machines can readily utilize commercially available inks rather than solely using their proprietary formulation. Their primary design considerations are focused on printing specialized antennas and sensors. They have printed 3-D sensor for detecting expansion or shrinkage of a turbine. They have also printed interconnects on integrated circuits (ICs) that serve as an alternative to wire bonding process. Aerosol Jet technology is considered superior to existing traditional manners of printing. For instance, it is capable of printing antennas directly on any given surface shape, which is called conformal printing, without the need to use injection molding and laser-based directed printing. With the abovementioned features, Optomec's Aerosol Jet system is viewed as a key player in the realization of mass production of 3-D printed electronics.



A three-faced 3D-printed sensing circuit that consists of a plastic tank with circuitry on it was printed by Optomec Aerosol Jet and OEM Neotech Services

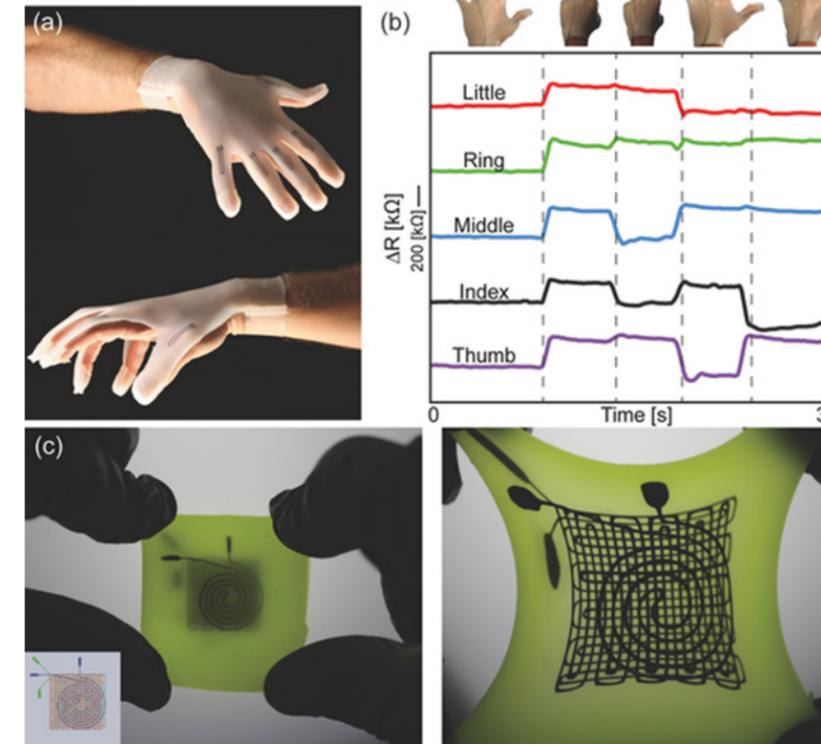
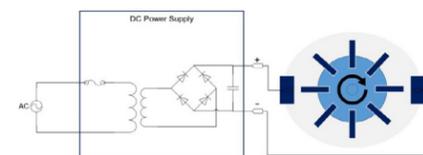
In the area of electrical engineering, attempts have been made to replicate the physical characteristics of metals by 3-D printing functionalized high-performance polymers for applications that require the rigidity and the electrical properties of metallic materials. In this case, electric motors, which are basically made of metallic parts, typically have the abovementioned properties to be able to effectively and efficiently transform electrical to mechanical energy or vice versa. We have successfully demonstrated in our laboratory a metal-free 3-D printed electrostatic motor



Conductive polydimethylsiloxane (PDMS) has been successfully 3D printed by using a unique embedded 3D printing (e-3DP) method, as shown. a) A photograph of a glove with embedded strain sensors produced by e-3DP. b) Electrical resistance change at different hand gestures. c) A three-layer strain and pressure sensor in the unstrained state (left) and stretched state (right).

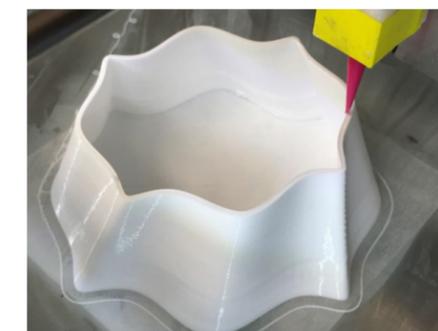
successfully demonstrated in our laboratory a metal-free 3-D printed electrostatic motor made of polyamide-12 and reduced graphene oxide powders using selective laser sintering technology. By applying DC voltage in the electrodes, the rotor is able to spin at a rate proportional to the electric field created at the electrodes, thus, this is also called capacitor motor. With the transition to building an object with near-metallic properties using 3-D printing and functionalized materials, 3-D printing technology and novel material preparation have really set another standard in the field of manufacturing process.

The photo shows a spinning 3-D printed electrostatic motor. The rotor blades and the electrodes were made of processed conductive



PA12 with reduced GO while the base was printed using pure PA12.

The photo shows a spinning 3-D printed electrostatic motor. The rotor blades and the electrodes were made of processed conductive PA12 with reduced GO while the base was printed using pure PA12.



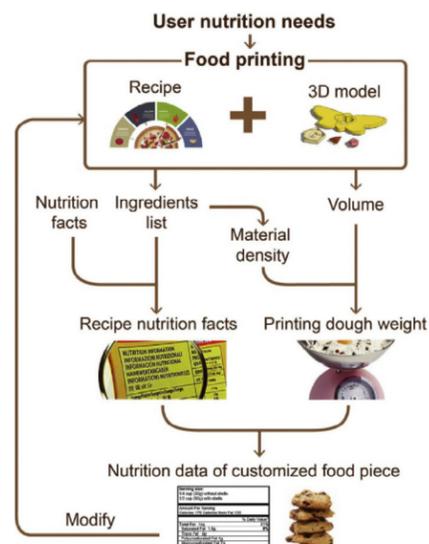
3-D printing of silicone

Printing of paste mixtures or viscous solutions will become a trend in the future along with the need for building freedom for complex shapes and geometries in manufacturing flexible materials. A broad array of functionalities has been demonstrated by flexible electronics. This unique category of electronics has already been playing an important role in manufacturing solar cells, displays and LEDs, sensors, and thin-film transistors. Flexible electronics has

been a trend due to its application in flexible displays and wearable electronics.

Conductive polydimethylsiloxane (PDMS) has been successfully 3D printed by using a unique embedded 3D printing (e-3DP) method, as shown. a) A photograph of a glove with embedded strain sensors produced by e-3DP. b) Electrical resistance change at different hand gestures. c) A three-layer strain and pressure sensor in the unstrained state (left) and stretched state (right).

Park Atomic force microscopy (AFM) plays an important role in our 3-D printing projects in the laboratory. This powerful tool is capable of looking at the surface profile of 3-D printed objects created by different 3-D printing technologies in nanometer range. One example is accurately profiling the width of the extruded strands and the height of the layers of a 3-D printed silicone adhesive via high-resolution paste extrusion technique. This measures the consistency of the resolution set all throughout the 3-D printing process. Moreover, shrinkage and other defects can also be examined which can be correlated to the build parameters of the 3-D printers. This helps identify significant adjustments of parameters for optimizing the build process and reduce defects found in the 3-D printed objects. Unlike other microscopy techniques, AFM does not need treatment of the sample that may cause damage to



Food fabrication

A rising technology in food fabrication called extrusion-based food printing uses the same mechanism as paste extrusion printing but is specifically intended for building food products using edible materials in a layer by layer manner. Researches have been conducted in 3-D printing food products with the ability to have nutritional control. The 3-D printing technique has been correlated to the produced food texture, and the tunability of nutritional content in the food product, based on 3-D printing extrusion mechanisms. 3-D printing in this field has truly revolutionized the way food is designed and fabricated.

Schematic of 3-D printing of food with nutritional control.

Sustainability

3-D printing market is seen to grow 500% in the next 5 years, reaching around \$16.2 billion by this year given the rise of 3-D printer and materials sales worldwide. "It has now moved from a new and much-hyped, but largely unproven, manufacturing process to a technology with the ability to produce real, innovative, complex and robust products." Thus, it is one of the fastest evolving markets to date. There's no question on its potential success due to its high level of customization, its ability to suppress replacement buying, curbing down material waste in manufacturing process, the idea of free sharing of digital designs online, and lessening carbon emissions by eliminating the need for transporting actual products. However, the big question is: how about its sustainability? The flip side would be: power consumption will increase with respect to production, there will be rise of single-use plastic production and usage, there will be introduction of strategized obsolescence of products, possible intellectual property theft due to replication of designs, and the dramatic increase of product consumption will happen,

which possibly raise ethical issues on the environment and on creating products and their actual use (e.g. guns, food restrictions, etc.). With the observance of circular economy as a basis for writing energy and waste management policies, initiatives around the world are being concretized towards reducing plastic wastes by recycling them with the aid of 3-D printing technologies. Although, the future is still unclear for a technology in its infancy stage, there are no assurances. Scientists and lawmakers are working hand in hand towards realizing sustainable development for 3-D printing. Time will come that additive manufacturing will surpass traditional manufacturing. When this time comes and the 3-D printing technology comes to an age, us – main consumers – are expected to take responsibility for the lives of the future generation, not just for the sustainability of the technology itself.

IN 2014, THE 3-D PRINTING INDUSTRY WAS ESTIMATED TO BE AT \$4 BILLION. WITH NEW PREDICTIONS THAT 3-D PRINTING WILL BE 50 PERCENT CHEAPER AND UP TO 400 PERCENT FASTER, THE INDUSTRY COULD REACH \$49 BILLION BY 2025.

References

B. Cook, B. Tehrani, J. Cooper, S. Kim and M. Tentzeris, "Integrated printing for 2D/3D flexible organic electronic devices," *Handbook of Flexible Organic Electronics*, pp. 199-216, 2015.
 G. Weidner, *Handbook of Nanofabrication*, Elsevier, 2009.
 R. Mosses and S. Brackneridge, "A novel process for the manufacturing of advanced interconnects," *Circuit World*, vol. 29, no. 3, pp. 18-21, 2003.
 D. Zhao, T. Liu, Z. Lin, M. Zhang, R. Liang and B. Wang, "Fabrication and characterization of aerosol-jet printed strain sensors for multifunctional composite structures," *Smart Mater. Struct.*, vol. 21, no. 11, 2012.
 B. Lu, D. Li and X. Tian, "Development Trends in Additive Manufacturing and 3D Printing," *Engineering*, vol. 1, pp. 85-89, 2015.
 Y. Shirasaki, G. Supran, M. Bawendi and V. Bulović, *Nature Photonics*, vol. 7, pp. 13-23, 2012.
 E. Macdonald, R. Salas, D. Espalin, M. Perez, E. Aguilera, D. Muse and R. Wicker, "3D Printing for the Rapid Prototyping of Structural Electronics," *IEEE Access*, vol. 2, pp. 234-242, 2014.
 E. Macdonald, "Integrating stereolithography and direct print technologies for 3D structural electronics fabrication," *Rapid Prototyping Journal*, 2012.
 J. Lewis and B. Ahn, "Three-dimensional printed electronics," *Nature*, vol. 518, pp. 42-43, 2015.
 L. Teschler, "Your next circuit design could be fabricated on a printer," 13 November 2015. [Online]. Available: <http://www.powerelectronicstips.com/your-next-circuit-design-could-be-fabricated-on-a-printer/>. [Accessed 22 June 2017].
 M. Clinch, "3-D printing market to grow 500% in 5 years," 1 April 2014. [Online]. Available: <https://www.cnn.com/2014/04/01/3-d-printing-market-to-grow-500-in-5-years.html> [Accessed 29 March 2018].
 L. Federico-O'Murchu, "How 3-D printing will radically change the world," 11 May 2014. [Online]. Available: <https://www.cnn.com/2014/05/09/will-3-d-technology-radically-change-the-world.html>. [Accessed 29 March 2018].
 A. Mitchell, "3D Printing: Adding a Sustainable Dimension to Modern Life," February 2018. [Online]. Available: <https://en.reset.org/knowledge/3d-printing-adding-sustainable-dimension-modern-life-02262018>. [Accessed 29 March 2018].



Alejandro H. Espera, Jr. is Assistant Professor, Electronics Engineering Department, School of Engineering and Architecture, Ateneo de Davao University, Philippines and is currently a PhD

student at the College of Engineering at Virginia Tech. He was a research fellow in 2017 at the Advincula Research Group, Department of Macromolecular Science and Engineering at Case Western Reserve University, led by Dr. Rigoberto Advincula.



Dr. Rigoberto Advincula is Professor with the Department of Macromolecular Science and Engineering at Case Western Reserve University and Research Professor at the Department

of Chemistry and Department of Chemical Engineering at the University of Houston and the current editor-in-chief of MRS Communications. Dr. Advincula is the recipient of numerous awards including Fellow of the American Chemical Society, Herman Mark Scholar Award of the Polymer Division, and Humboldt Fellow. He did Post-doctoral work at the Max Planck Institute for Polymer Research and Stanford University and has published over 380 papers, including 195 peer-reviewed publications, 11 patents (and pending), and has co-edited the book on Functional Polymer Films, Polymer Brushes, and New Trends in Polymer Science.

For more information go to: <http://www.rcapoly.net/>

Polymers and 3D Printing Event in Houston

Dr. Advincula is hosting a two-day Polymers and 3D Printing Conference Nov. 1-2, 2018 in Houston TX. This conference will connect real-world applications with basic technology covering many topics including the fundamentals of polymers, 3D printing technology, characterization techniques, coatings and finishes for 3D Printing, thermo-mechanical evaluation and 4D Printing and beyond. This will be a two-day Conference-Training meeting and premiere networking event that will draw participants from many in the industry: chemical companies, academia, coatings experts, formulators, and applicators. For information on registration go to

www.poly3d.org

Attn: students & postdocs!



2018 Park AFM Scholarship

Park Systems, the world's leading manufacturer of atomic force microscopes (AFM) invites all researchers worldwide to apply to become Park AFM Scholars and receive a research scholarship. Park AFM Scholarship Awards are open to undergraduate or postdoctoral students working in nanotechnology research either already using Park AFM or who have research they would like to do with a Park AFM and need help getting access to equipment. Through this program, Park Systems has offered assistance to many researchers who qualify as Park AFM Scholars by matching them with one of thousands of nanoscience shared user facilities to perform their research using Park AFMs.

"WE NOT ONLY OFFER FINANCIAL INCENTIVE TO PARK AFM SCHOLARS WHO ARE PIONEERING NEW RESEARCH METHODOLOGIES IN NANOTECHNOLOGY AT LEADING ACADEMIC INSTITUTIONS WORLDWIDE, BUT MOST IMPORTANTLY ARE GIVING THEM ACCESS TO OUR PARK ATOMIC FORCE MICROSCOPES," STATED KEIBOCK LEE, PARK SYSTEMS PRESIDENT. "WE WILL CONTINUE TO ADVANCE NANOSCALE DISCOVERIES THRU THIS PARK AFM SCHOLARSHIP PROGRAM WORLDWIDE."

The Park AFM Scholarship Award is open to postdoctoral researchers and graduate students working in nanotechnology research using Park AFM. As progress for nanotechnology research and development advances at an unprecedented rate, universities worldwide offer degrees in fields working with nanotechnology. Park Systems, world-leading manufacturer of atomic force microscopes, is offering a \$500 USD monetary scholarship to promote the education of future scientists and engineers in a number of nanoscience research areas that require advanced nanoscale microscopy for sample analysis and observation and to promote shared research findings and methodologies amongst their peers.

PARK SYSTEMS PARK AFM SCHOLARSHIP AWARDS

PROGRAM CURRENTLY OPEN TO ALL REGIONS
AROUND THE WORLD

TWO NEW PARK AFM SCHOLARSHIP WINNERS ANNOUNCED



JINGSHAN DU

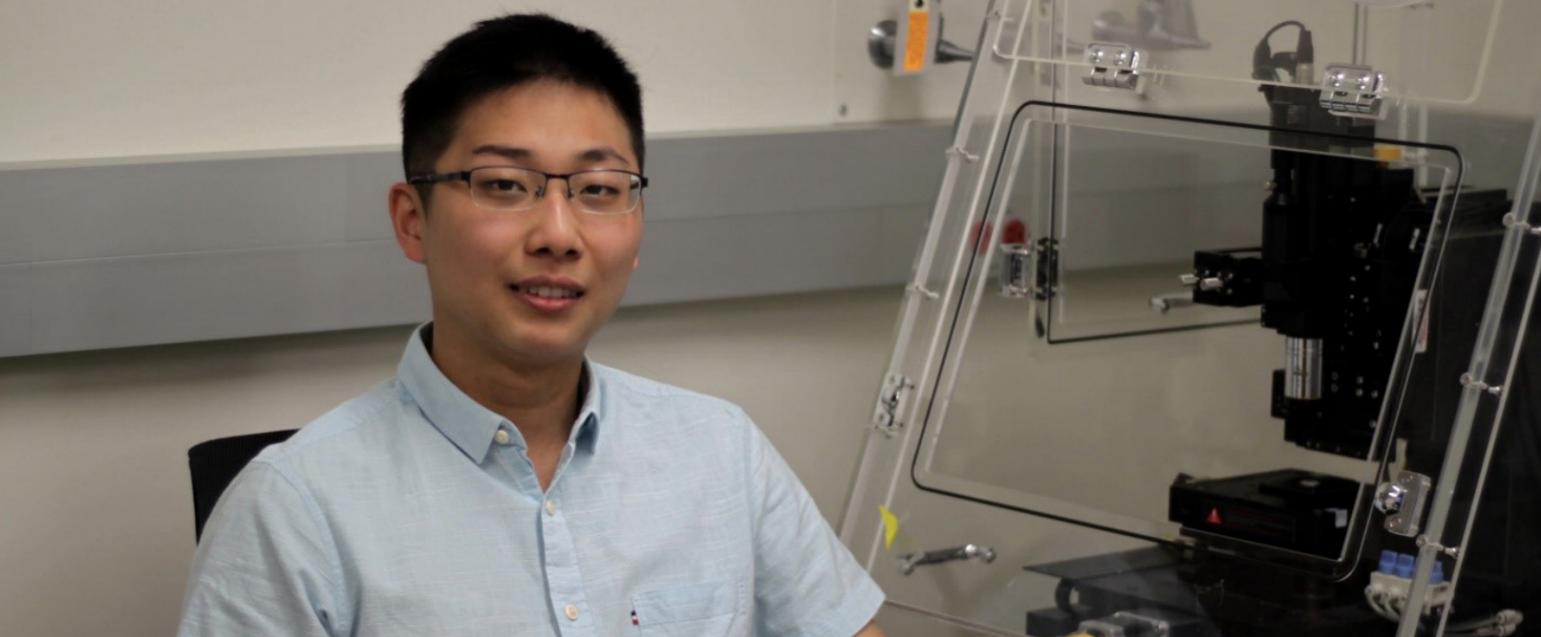
WEBSITE: [HTTP://DUJINGSHAN.TK/](http://DUJINGSHAN.TK/)

"I USE THE PARK AFM TO GENERATE NANO REACTORS ON ELECTRON-TRANSPARENT SUBSTRATES. THE PRECISE CONTROL OF SCANNING PROBE POSITION IN THREE DIMENSIONS ALLOWED ME TO DEPOSIT NANOSCALE FEATURES OF FLUIDS, SUCH AS POLYMER SOLUTIONS, ONTO FILMS THAT ARE ONLY FEW NANOMETERS THICK."

Jingshan Du is currently a graduate student in the Department of Materials Science and Engineering, Northwestern University. Jingshan Du received his B.S. in Engineering: Materials Science and Engineering from Chu Kochen Honors College, Zhejiang University in China, where he worked with Prof. Deren Yang and Prof. Hui Zhang on the colloidal synthesis of hybrid nanomaterials. Before joining Northwestern, he spent his senior year in Prof. David A. Weitz's group at Harvard studying the radiation-induced transformation of nanoparticles using in situ TEM. His current research includes the structures and structural transformation of multicomponent nanoparticles. He was a recipient of a Ryan Fellowship from Northwestern, a Chu Kochen Scholarship from Zhejiang University, and a CSST Award from UCLA.

Please summarize the research you do and explain why it is significant?

Nanoparticles consisting of multiple elements and phases have shown great potentials in enabling multifunctionality and tuning the functionality of each component for various applications, ranging from plasmonics to catalysis. My research focuses on understanding how different elements and phases construct a nanoparticle, evolve in environments, and exhibit interesting properties. Correlative electron microscopy provides multidimensional information on the structures and structural evolution of the complex nanoparticles generated by scanning probe block copolymer lithography (SPBCL). These studies may not only shine light on fundamental chemical physics in nanoscale systems, but also help the design of high performance and multifunctional nanomaterials such as catalysts, sensors, and medicines.



Jingshan Du pictured above with Park AFM at Northwestern University where he uses the unique nanolithography module in Park AFM to rapidly generate nanoreactor arrays on an electron microscopy-compatible substrate.

of high performance and multifunctional nanomaterials such as catalysts, sensors, and medicines.

How might your research be used?

From a basic science perspective, my study provides insights into how multiple elements, phases, and materials interact and evolve in nanoparticles in different environments. This allows us to look at complex multicomponent nanoparticles in a new way, as complex systems, and start to understand their behaviors that stem from fundamental chemical physics. We also invest significant efforts into studying their properties such as plasmonics and catalysis. These multicomponent nanoparticles, with the desired composition and structure, may be able to enable some exciting new applications such as cascade catalysis.

Why is the Park AFM important for your research?

My research focuses on interfacing nanopatterning techniques and electron microscopy into a platform for investigating complex nanoparticle systems. These systems, including multicomponent, multiphase nanoparticles, and their interfaces with fluids and gases, are generally difficult to prepare in a systematic and combinatorial way. I use the Park AFM to generate nano reactors on electron-transparent substrates. The precise control of scanning probe position in three dimensions allowed me to deposit nanoscale features of fluids, such as polymer solutions, onto films that are only few nanometers thick.

What features of Park AFM are the most beneficial and why?

The unique nanolithography module in environments. This allows us to look at complex multicomponent nanoparticles in a new way, as complex systems, and start to understand their behaviors that stem from fundamental chemical physics. We also invest significant efforts into studying their properties such as plasmonics and catalysis. These multicomponent nanoparticles, with the desired composition and structure, may be able to enable some exciting new applications such as cascade catalysis.

Why is the Park AFM important for your research?

My research focuses on interfacing nanopatterning techniques and electron microscopy into a platform for investigating complex nanoparticle systems. These systems, including multicomponent, multiphase nanoparticles, and their interfaces with fluids

and gases, are generally difficult to prepare in a systematic and combinatorial way. I use the Park AFM to generate nano reactors on electron-transparent substrates. The precise control of scanning probe position in three dimensions allowed me to deposit nanoscale features of fluids, such as polymer solutions, onto films that are only few nanometers thick.

What features of Park AFM are the most beneficial and why?

The unique nanolithography module in Park AFM allows us to rapidly generate nanoreactor arrays on an electron microscopy-compatible substrate. Compared to other sample preparation methods that result in randomly located nanoparticles, the regular arrays generated on Park AFM enables us to index and trace back to each particle repeatedly. This makes correlative characterization, ex situ, and in situ studies using electron microscopy possible on the individual nanoparticle level.

PARK AFM SCHOLARSHIP PROGRAM ACCEPTING SUBMISSIONS

Park Systems is continuing their successful Park AFM Scholarship Program in 2018. To be eligible:

- 1) The awardee must be a graduate student or postdoctoral researcher currently enrolled/affiliated with a research university, national laboratory, or governmental agency.
- 2) The research being presented must include meaningful data acquired using a Park AFM instrument. This data can

either be the sole data being discussed in the presentation or be in conjunction with data acquired with other types of tools.

Park Systems will offer assistance to researchers who need a facility to perform their research using Park Atomic Force Microscope by matching them with one of their shared nano facilities. For more information on the Park AFM Scholarship program, go to: <http://www.parkafm.com/index.php/medias/programs/park-afm-scholarship>



HOSSEIN ROKNI DAMAVANDI TAHER

“THE NOVEL DESIGN OF THE PARK XE-70 AFM COUPLED WITH HIGHLY USER-FRIENDLY INTERFACE SOFTWARE XEP HAS MADE IT POSSIBLE TO SIMULTANEOUSLY IMAGE SAMPLE SURFACES WITH ATOMIC-SCALE RESOLUTION, PRECISELY MANIPULATE NANO-OBJECTS DOWN TO SINGLE MOLECULES AND ACCURATELY MEASURE THE PHYSICAL PROPERTIES OF INTEREST THROUGH A COMPREHENSIVE RANGE OF INNOVATIVE MODES SUPPORTED BY THE PARK AFM (E.G., IMAGING MODE, ELECTRICAL MODE, NANOMECHANICAL MODE, ETC.)”

Hossein Rokni Damavandi Taher is currently a Postdoctoral Research Fellow in Prof. Wei Lu's group at the University of Michigan. He earned his Ph.D. from University of Michigan in 2018, M.S. from University of British Columbia in 2011 and B.S. from Iran University of Science and Technology in 2005, all in Mechanical Engineering. He is the recipient of the 2015 I.K. McIvor Award for excellence in research and scholarship in Applied Mechanics and the 2016 Rackham Predoctoral Fellowship Award for unusually creative, ambitious and impactful dissertation research and the nominee for the 2018 ProQuest Distinguished Dissertation Award. He has also co-authored over 50 publications with more than 1100 citations. His research focuses on micro/nanomechanics, micro/nanocharacterization and atomistic-to-continuum modeling of atomically thin layered materials, vdW hetero structures, biomaterials and smart materials as well as on the development of MEMS/NEMS devices incorporating such materials.

Please summarize the research you do and explain why it is significant?

My current research mainly focuses on developing novel micro/nanoscale measurement techniques combined with multiscale modeling at the atomistic and continuum levels to advance our fundamental understanding of mechanical and electrical behavior of atomically thin layered materials and newly emerged vdW hetero structures. Such powerful characterization techniques are then to be leveraged to improve synthesis, transfer, manipulation, integration and performance of opto-, bio- and nanoelectronic devices incorporating 2D layered materials and

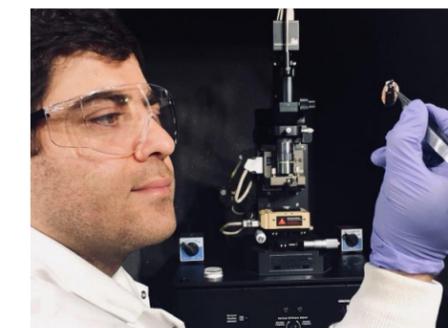
vdW heterostructures.

How might your research be used?

Atomically thin layered materials (ATLMs), such as graphene, hexagonal boron nitride and transition metal dichalcogenides (e.g., MoS₂ and WS₂), and vdW heterostructures have catapulted nanoscience and nanotechnology to unimaginable new heights in a relatively short period of time since graphene was isolated from bulk graphite in 2004. They exhibit a unique combination of high elasticity, extreme mechanical flexibility, visual transparency, and superior electronic performance that are not essentially observed in their bulk crystals, making them ideally suited to modern devices, such as flexible biosensors, photovoltaic devices, fast charging lithium ion batteries, transparent touchscreen displays, hydrogen evolution catalysis, flexible transistors, photo detectors, and memory devices. However, scalable, controllable and cost-effective production of ATLMs with high quality has still remained challenging mainly due to the poor understanding of the nature of weak interlayer interactions and the lack of precise experimental techniques to characterize such complex interlayer behavior of ATLMs. As a means to address such a challenging issue, we combined conductive atomic force microscopy and molecular dynamics simulations to unravel the relative contributions of electrostatic attraction/repulsion interactions and highly anisotropic vdW interactions to the exfoliation of the ATLMs from their bulk crystals. This unique nanoscale manipulation setup also allowed us not only to quantify, for the first time, the effect of layer number and electric field on the dielectric constant of ATLMs with few-layer down to monolayer thickness, but also to successfully produce The University of Michigan's thinnest possible logo by only mono-, bi- and trilayer graphene flakes, thanks to the ultrahigh force-displacement resolution of the conductive AFM-assisted electrostatic manipulation setup.

Why is the Park AFM important for your research?

The nature of our research requires a unique combination of high-resolution imaging, nanoscale mechanical manipulation and precise in-situ measurements of atomically thin layered materials (ATLMs) and newly emerged van der Waals (vdW) heterostructures. Fortunately, our Park XE-70 atomic force microscope offers such a unique nanoscale manipulation setup with ultra high force-displacement resolution, making nanoscale probing of weak interlayer interactions in ATLMs and vdW hetero structures possible.



What features of Park AFM are the most beneficial and why?

The novel design of the Park XE-70 AFM coupled with highly user-friendly interface software XEP has made it possible to simultaneously image sample surfaces with atomic-scale resolution, precisely manipulate nano-objects down to single molecules and accurately measure the physical properties of interest through a comprehensive range of innovative modes supported by the Park AFM (e.g., imaging mode, electrical mode, nanomechanical mode, etc.). Also, data analysis software XEI, which is widely used in our lab for the physical and structural characterization of nano-features, plays a crucial role in extracting the essential information from our AFM measurements.

USING AFM PINPOINT™ NANOMECHANICAL MODE FOR QUANTIFICATION OF ELASTIC MODULUS IN MATERIALS TWO ORDERS OF MAGNITUDE FASTER THAN FORCE VOLUME SPECTROSCOPY

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

Introduction

Since the invention of atomic force microscopy, AFM has had a revolutionary impact in material sciences and device engineering by providing accurate, reliable, non-destructive imaging at the nanoscale. AFM is used across a wide range of nanotechnology applications such as biomedical implantable actuators to ultrathin cathode material for batteries to photo detectors and switches for memory storage and logic circuits. [1-3] As the dimensions of devices continuously shrink, measurement methods of local properties of materials have become more effective over bulk property in providing accurate nanoscale measurement. Local mechanical properties such as adhesion and elastic modulus are critical parameters in determining the reliability and performances of these devices. [4-6] There were existing AFM-based nanomechanical methods introduced to measure the mechanical properties, examples include force-volume spectroscopy and nanoindentation. However, some of these techniques are extremely time consuming and others are destructive which doesn't allow in situ and high-throughput monitoring in certain applications.

Figure 1 demonstrates the principles of AFM PinPoint™ nanomechanical mode developed by Park Systems. The patented PinPoint™ technique is at least two orders of magnitude faster than the conventional force-volume technique which allow users to acquire quantitative mechanical properties and high-resolution topography images of materials simultaneously in a short period of time. During operation, the tip moves in approach-retract manner, ensuring a frictionless operation which

eliminates the lateral force due to continuous tip-sample contact and preserves tip and sample condition, ideal in measuring both hard and soft samples such as hard disks and biological samples. At each point of the image, force-distance curve is acquired and used to calculate the mechanical characteristics of the sample being measured. During data acquisition, the XY scanner stops, and the contact time is controlled to give enough time for the scanner to acquire precise and accurate data.

In this experiment, 4 different materials with various ranges of modulus were successfully quantified. The results acquired for each test are close to the nominal modulus value of each material, proving the superior ability of the PinPoint™ mode in quantification of mechanical properties. In addition, high-resolution images were acquired simultaneously which revealed the surface features of the samples.

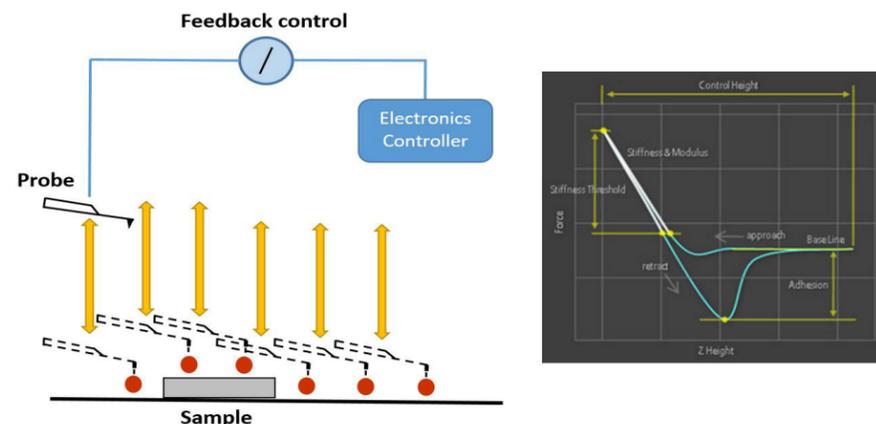


Figure 1. Principles of PinPoint™ nanomechanical mode by Park Systems. This diagram demonstrates the feedback-controlled approach and retraction of a probe at multiple sites along a sample's surface. Feedback control from the AFM systems' controller allows this technique to acquire both surface topography. The F/D curve demonstrate how PinPoint extracts mechanical property data.

Experimental

Sample and Tip

An Olympus micro cantilever (OMCL-AC160TS) with nominal spring constant $k = 26$ N/m and resonant frequency $f = 300$ kHz was used to measure the Highly Ordered Pyrolytic Graphite (HOPG), Polydimethylsiloxane (PDMS) and Polystyrene – low density polyolefin elastomer (PS-LDPE) in the experiment. The HOPG sample is a highly ordered form of highly pure Pyrolytic Graphite annealed under high temperatures and pressure to obtain a high degree of crystallographic orientation. The PDMS sample is a silicone polymer which is physically soft like rubber or resin mounted on a magnetic sample disk. The PS-LDPE sample is a copolymer composed of PS and PE that were spin-cast onto a silicon substrate creating a film with different modulus properties. PS serves as the matrix while PE is the low density doping component.

Pinpoint Imaging Conditions

The samples were investigated using Park NX20 AFM system under ambient air condition. Except for scan range, the scanned parameters used in measuring the samples were all the same.

Results and Discussion

Hertzian model

The quantification of mechanical properties of Pinpoint mode such as elastic modulus is based on the well-established Hertzian model. In this technique, it is assumed that no other interaction other than elastic deformation can be observed between tip and sample. The calculation is based on the equation E (Elastic modulus) = F (Loading force). This equation is greatly affected by the tip geometries. Assuming that a spherical tip with specific radius (R) was used to indent elastic halfspace to a displacement of d , the applied force can be derived as below:

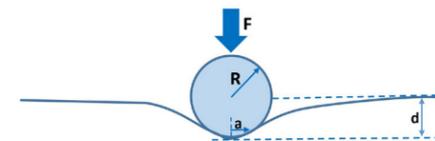


Figure 2. Illustration of spherical tip and sample interaction showing the displacement created in the sample surface as force is applied.

By measuring the corresponding F , R , and d , the E^* can be reversed-calculated. However, E^* is a function of two material and its equation can be expressed as below:

$$F = \frac{4}{3} E^* \sqrt{Rd^3}$$

E_{tip} and E_{sample} are the elastic modulus of the two materials and ν_{tip} and ν_{sample} are their corresponding Poisson's ratio. By knowing E^* , E_{tip} , ν_{tip} and ν_{sample} , E_{sample} can be reversed-calculated. [7]

Table 1 shows the topography images and the correlated maps of elastic modulus acquired during the tests. The images acquired in this experiment were all analyzed using XEI software developed by Park Systems which mapped the acquired signals to a color table. For topography images, the intensity of the shading correlates to the surface height variation with extremely bright and dark areas having the highest and lowest height regions. Same thing applies with the elastic modulus, wherein brighter and darker regions correspond to areas with highest and lowest elastic modulus. The topography image of HOPG revealed that surfaces of the sample are made of atomic terraces with step edges.

These terraces demonstrate a homogenous diagonal path and they were measured to have approximately 1.5 nm step height. On the other hand, the elastic modulus map shows only few variations on the mechanical property of the sample surfaces. The diagonal dark lines visible on the elastic modulus image represent the surfaces with low modulus. The topography image of the PDMS revealed the rough surface of the sample. It can be also observed on both topography and modulus image that there are only few variations on height and modulus of the surfaces. The topography image of PS-LDPE confirms that the sample is composed of two different materials. The dark circular dots features are the LDPE (low density polyolefin) materials, while the brighter regions in this image are the PS (polystyrene) materials. As expected, the elastic modulus map of this sample has two domains, wherein LDPE materials represented by the dark circular dots are the domains with lower modulus, while PS represented by the areas with bright color are the domains with higher modulus.

Sample	Topography	Elastic Modulus
HOPG		
PDMS		
PS-LDPE		

Table 1: Topography image and correlated maps of elastic modulus acquired for each material.

The quantitative results of elastic modulus are shown in Table 2 and corresponding graph shown in Figure 3. The results were plotted to better analyze the data. To evaluate the performance of Pinpoint mode, the results acquired in this experiment were compared to the nominal values of each sample. And it was found out that the measured modulus values of all the samples are close to their nominal values. For HOPG, the measured modulus is 22.09 GPa, close to its nominal value of 18 GPa. For PDMS, the measured modulus is 0.0018 GPa, close to its nominal value of .003 GPa. While for PS-LDPE, the measured values are 1.955 GPa and 0.132 GPa, which are also comparable to their nominal values of 2 GPa and 0.1 GPa respectively. The standard deviation of the modulus results were also

calculated and based on the results, PDMS sample has the lowest standard deviation with $Std = 4.03E-04$ GPa, while HOPG sample has the highest standard deviation with $Std = 2.224$ GPa.

Sample	Modulus (GPa)	Std (GPa)
PDMS	0.0018	4.03E-04
LDPE	0.132	0.048
PS	1.955	0.187
HOPG	22.093	2.224

Table 2. Measured mean and standard deviation (Std) of elastic modulus.

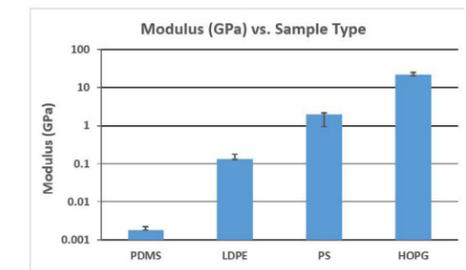


Figure 3. Plotted value of measured mean and standard deviation of elastic modulus.

Conclusion

The quantitative and qualitative data of the four materials were successfully acquired by Park NX20 AFM using PinPoint™ nanomechanical mode. The surface features of the samples such as the atomic terraces surface of the HOPG, the rough surface of the PDMS, and the circular dots matrix of the PS-LDPE were clearly observed on the topography images. The measured elastic modulus of each sample was close to their nominal modulus value, confirming the capability of PinPoint™ nanomechanical mode in quantifying a wide range of mechanical properties of various materials. The new PinPoint™ nanomechanical mode as demonstrated in this experiment provides researchers accurate material surface morphology and nanomechanical property information critical in determining the reliability and performances of certain materials and devices.

REFERENCES

- [1] Dr. R. Cook, *Nanomechanical Measurements and Tools*, Written by AZoNanoJan 17 2010.
- [2] F. Wu, et al., *Ultrathin Spinel Membrane-Encapsulated Layered Lithium-Rich Cathode Material for Advanced Li-Ion Batteries*, *Nano Lett.*, 2014, 14 (6), pp 3550–3555.
- [3] H. Kind, et al., *Nanowire Ultraviolet Photodetectors and Optical Switches*, *Advanced Materials*.
- [4] Z. Drira, et al., *Nanomechanical measurements of polyethylene glycol hydrogels using atomic force microscopy*, *Volume 18, February 2013, Pages 20-28*.
- [5] J. Pineda, et al., *Optimum Current Distribution Measurement of Zinc Oxide Nanorods via PinPoint™ Conductive AFM*.
- [6] C. Clifford, et al., *Quantification issues in the identification of nanoscale regions of homopolymers using modulus measurement via AFM nanoindentation*, *Applied Surface Science*, *Volume 252, Issue 5, 15 December 2005, Pages 1915-1933*.
- [7] M. Hong, et al., *Collagen Fibrils Imaging in Air and in Liquid Using Atomic Force Microscope-Based Fast Nanomechanical Mode*.

Park NX12

The most versatile
atomic force microscope
for analytical and electrochemistry

- *Built on proven Park AFM performance*
- *Equipped with inverted optical microscope*

Proven Performance

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

Built for Versatility

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

Competitive Pricing

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



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

www.parkAFM.com