

USING AFM TO IDENTIFY SINGLE MOLECULE CHARACTERIZATION

AN INTERVIEW WITH
FRANCESCO RUGGERI

p. 6

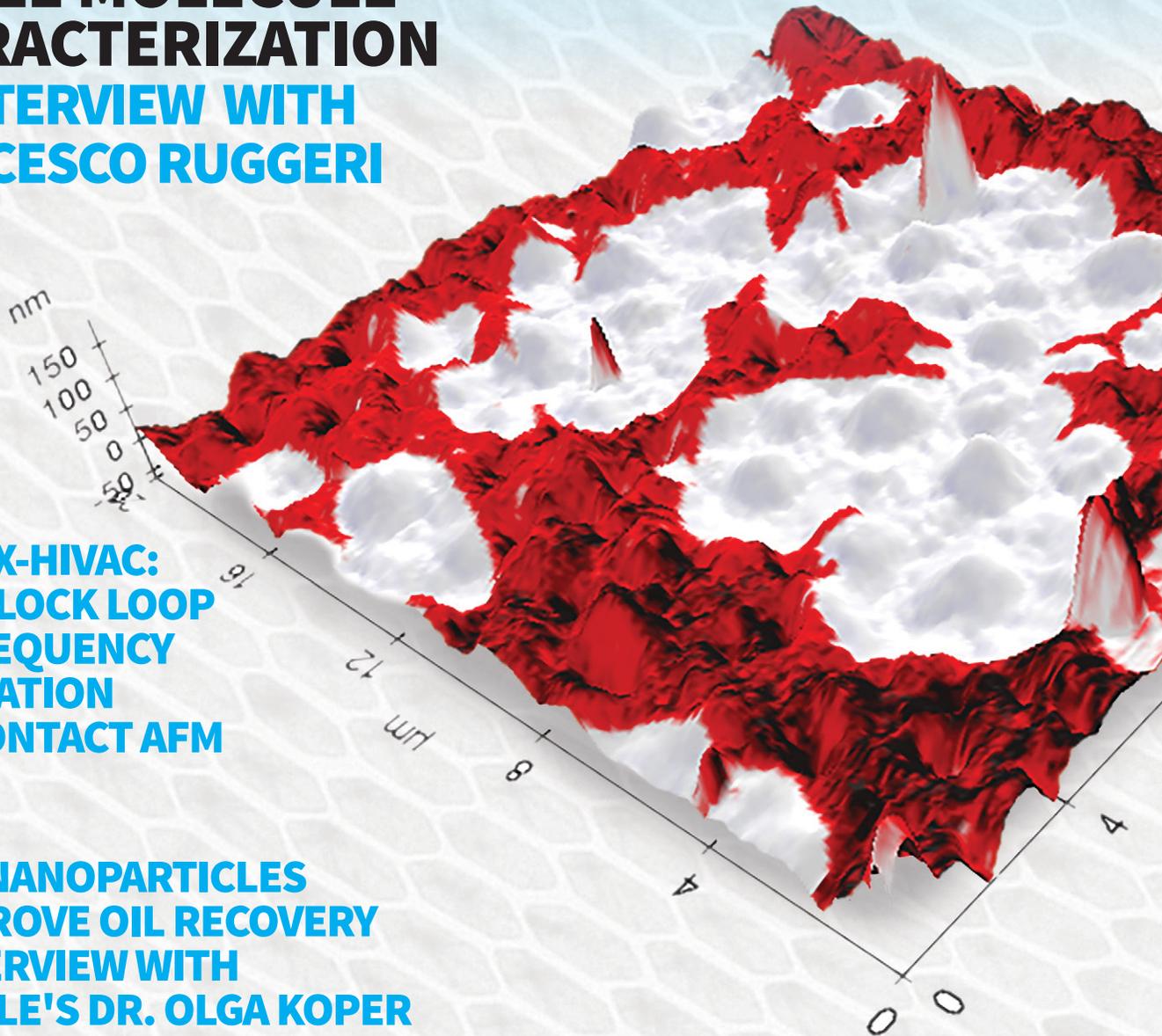
PARK NX-HIVAC:
PHASE-LOCK LOOP
FOR FREQUENCY
MODULATION
NON-CONTACT AFM

p. 12

USING NANOPARTICLES
TO IMPROVE OIL RECOVERY
AN INTERVIEW WITH
BATTELLE'S DR. OLGA KOPER

p. 16

ELECTROCHEMICAL ATOMIC
FORCE MICROSCOPY (EC-AFM):
IN SITU MONITORING OF
COPPER ELECTRODEPOSITION
ON GOLD SURFACE p. 19





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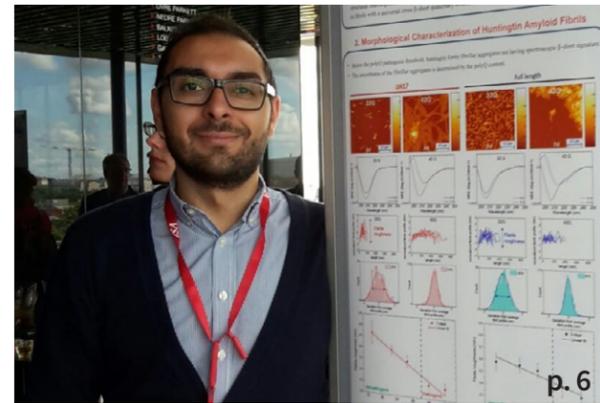


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TABLE OF CONTENTS

NanoScientific Vol 12 Winter 2018

Message from Editor	5
Feature Article: Using AFM to Identify Single Molecule Characterization, an interview with Francesco Ruggeri, Junior Research Fellow, Department of Chemistry & Centre for Misfolding Diseases, University of Cambridge	6
Abridged Article: Detecting nanoscale vibrations as signature of life Sandor Kasas, Francesco Simone Ruggeri, Carine Benadib, Caroline Maillard, Peter Stupar, Hélène Tourneau, Giovanni Dietler, and Giovanni Longo	8
In the News: Park Systems Opens Nanoscience Center at SUNY Polytechnic Institute	9
Park NX-Hivac: Phase-lock Loop for Frequency Modulation Non-Contact AFM Romain Stomp, James Wei, Hosung Seo, Dan Goo, and Gordon Jung	12
Feature Article: Using Nanoparticles to Improve Oil Recovery an interview with Battelle's Dr. Olga Koper	16
Electrochemical Atomic Force Microscopy (EC-AFM): In Situ Monitoring of Copper Electrodeposition on Gold Surface John Paul Pineda, Mario Leal, Gerald Pascual, Byong Kim, and Keibock Lee	19
Park AFM Scholarship Awards: Overwhelming Success Continues With Global Expansion Two new AFM Scholars Announced from Northwestern University.	24

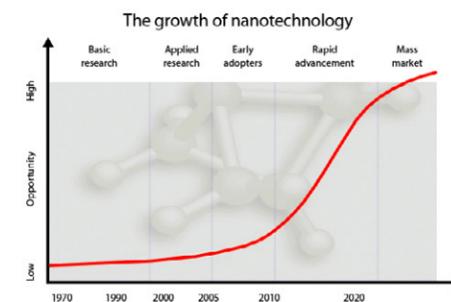


Keibock Lee, Editor-in-Chief

MESSAGE FROM EDITOR

Hello and Happy New Year

2017 was an amazing year for nanotechnology, where industries are beginning to see the results of astonishing research made possible through the explosion of nanoscience research world-wide. As new findings culminate into collective mass market products, (shown in graph below) nanotechnology is indeed living up the prediction of the National Science Foundation of becoming a trillion dollar industry and forever changing our world.



With significant improvements in virtually every industry, made possible through research and industry collaborations, nanoscience continues to expand our ability to heal ourselves and create sustainable solutions for a better world. In this issue, we highlight new discoveries and the application tools making them possible. In our first article we showcase the research being done at University of Cambridge by Francesco Ruggeri at the Center for Misfolding Diseases where identifying single molecule characterizations by using AFM is helping to find cures for neurological diseases like Alzheimers.

We also bring you exciting news about the

grand opening of the new Park Nanoscience Center at SUNY Polytechnic Institute, one of the world's most advanced high-tech education and research and development sites. Park received a tremendous welcome from SUNY Poly as demonstrated in this statement by Dr. Bahgat G. Sammakia, Interim President of the SUNY Polytechnic Institute, "SUNY Poly is thrilled that a worldwide leader in atomic force microscopy is selecting the campus for its newest location, and we warmly welcome Park Systems as we look forward to working closely to advance research capabilities in this important area."

And we showcase a technique developed with Zurich Instruments for combining Zurich Instruments HF2PLL with Lock-in Amplifier [HF2LI] with a Park Systems AFM [Park NX-Hivac]. This combination enables capabilities such as frequency modulation AFM, which allows researchers to observe the dynamic properties of an oscillating cantilever which can be used to quantify surface potential measurements.

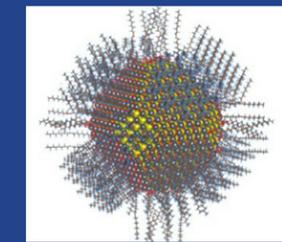
We also have a feature interview in this issue with Dr. Olga Koper highlighting a new very promising green chemistry method under development at Battelle using a soy-based surfactant. She also discusses other exciting research at Battelle using nanoparticles and nanofluids (solutions containing nanoparticles 1-100 nm in dimension).

We have a technical article presenting details on lateral force microscopy, a mode derived from atomic force microscopy developed for nanoscale frictional measurement, or nanotribology. This technique, as demonstrated in the article, is particularly powerful in identification and mapping of the relative difference in frictional characteristics with superior spatial resolution.

Lastly, we feature two additional recipients of the Park AFM Scholarship. So far, Park has given this recognition to ten outstanding researchers at some of our most distinguished institutions and this year, Park AFM Scholarships will expand globally to continue helping researchers advance nanoscale discoveries.

In each issue of NanoScientific, we provide informative articles about nanotechnology trends balanced with leading edge scientific research applications and concepts. As always, I encourage readers to submit your comments, story ideas, and user experiences. I hope you enjoy this issue and best wishes in the New Year.

Future timeline highlights discoveries that improve healthcare, energy and more as scientists world-wide continue to make astonishing breakthroughs using nanotechnology



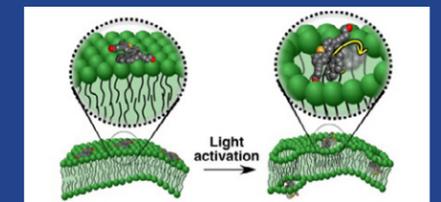
Researchers at the U.S. Department of Energy's (DOE) National Renewable Energy Laboratory (NREL) have set a new world efficiency record for quantum dot solar cells,

at 13.4%. Colloidal quantum dots are electronic materials and because of their astonishingly small size (typically 3-20 nanometers in diameter) they possess fascinating optical properties. Rapid improvements in quantum dot solar cells have pushed that number into double digits. Progress from the initial low efficiency came from better understanding of the connectivity between individual quantum dots, better overall device structures, and the prevention of defects in each dot.



Researchers at Texas A&M working with Los Alamos National Laboratory have found a way of protecting the materials in

fusion reactors from degradation caused by helium, using nanocomposite solids, which could make fusion energy viable. Nuclear fusion, is the process that powers main-sequence stars like the Sun. If harnessed, it would provide unlimited clean energy. However, constructing a fusion power plant has proven to be a daunting task, in part because no materials can adequately withstand the conditions found in a reactor core. Now, researchers at Texas A&M University have discovered a way to use materials that may be suitable by investigating how helium behaves in nanocomposite solids, materials stacked into thick metal layers. Rather than making bubbles, the helium in these materials formed long channels, resembling veins in living tissues. This discovery paves the way to helium-resistant materials needed to make fusion energy a reality.



Researchers at Durham University, North Carolina State University, and Rice University have demonstrated, in lab tests, how rotors in single-molecule nanomachines can be activated by ultraviolet light to spin at three million rotations per second and pierce the membranes of cancer cells to kill them within 60 seconds. The motor itself is a paddle-like chain of atoms that can be prompted to move in a single direction when supplied with energy and made to spin when activated by a light source. The motors, nanomachines so small you could put 50,000 of them across the diameter of a human hair, can target a cell's 8-10 nm lipid bilayer membrane and then either tunnel through to deliver drugs or other payloads, or disrupt it and kill the cell.

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For inquiries about submitting story ideas, please contact Deborah West, Content Editor at debbie@nano-scientific.org. For inquiries about advertising in NANOscientific, please contact Gerald Pascual at gerald@nano-scientific.org



INSET PHOTO ON COVER:

Image of a composite polymer sample (20 μm x 20 μm scan size). Lateral force microscopy (LFM) mode on a Park Systems atomic force microscope (AFM) was used to create this image. Here, data for both topography and surface friction characteristics of a composite polymer sample spun onto a glass substrate were acquired. The regions in white were observed to be less sticky than those in the red and dark regions.

USING AFM TO IDENTIFY SINGLE MOLECULE CHARACTERIZATION

AN INTERVIEW WITH FRANCESCO RUGGERI, JUNIOR RESEARCH FELLOW DEPARTMENT OF CHEMISTRY & CENTER FOR MISFOLDING DISEASES, UNIVERSITY OF CAMBRIDGE



Francesco Simone Ruggeri is a Junior Research fellow at the Darwin College and Research Fellow at the Department of Chemistry & Center for Misfolding Diseases at the University of Cambridge. Both at Cambridge and previously at École Polytechnique Fédérale de Lausanne (EPFL), his research is focused on the biophysical characterization of proteins at the single molecule scale by means of atomic force microscopy based techniques. This approach has brought new insights into the formation and structural characterization of misfolding proteins and their correlation with the onset of neurodegenerative disorders.

The Park AFM is fundamental for my research on amyloids because of some extremely important features: i) it has an extremely low electrical noise in the order of 25 pm, which enable high-resolution measurements at the nanoscale, ii) the decoupled XY and Z scanners, which enable easier post-processing (flattening) of the image at these small scales, together with a consistent exclusion of the molecules during the 3-D AFM map flattening, and iii) the versatility of the instrument interface and software make the tool extremely easy to use and allow high-throughput, high-resolution measurements.

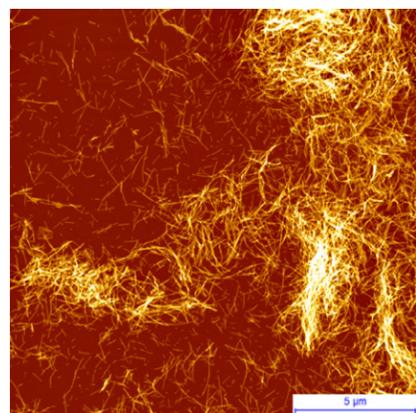


Figure 1. AFM image of amyloid fibrils of α -synuclein, the protein involved in the onset of Parkinson's disease.

The molecular origin and mechanistic link between amyloid formation and disease aetiology remain unclear and no disease modifying therapies are available for these disorders. Strong evidence links propensity of proteins to misfolding and aggregation to the pathological biology implicated in the onset

of these diseases. Despite its importance, unraveling amyloid properties and formation still represents a formidable experimental challenge, mainly because of their nanoscale dimensions and heterogeneous nature. Therefore, the investigation of the misfolding of monomers and aggregation into oligomers and fibrils is central to understand their stability, toxicity, and mechanism of clearance in the body and to design new therapeutic strategies to the amyloid diseases problem.

The objective of my research is to address the challenge of understanding the role amyloids serve in the onset of human diseases by studying aggregates one at the time to achieve a molecular level characterization of amyloid properties and formation. Indeed, the high complexity in the connection between protein aggregation and cellular toxicity may be reconciled by taking into account a structural diversity and heterogeneity of amyloid aggregates formed. Therefore, there is need for tools able to characterize the biophysical properties of heterogeneous amyloids at the nanoscale in vitro, in relation to the molecular mechanism of onset of the pathologies and to pharmacological treatments in vivo. My approach is to study amyloid heterogeneity from a biophysical point of view and to develop and apply novel methodologies to shed light on the physical determinants of protein aggregation.

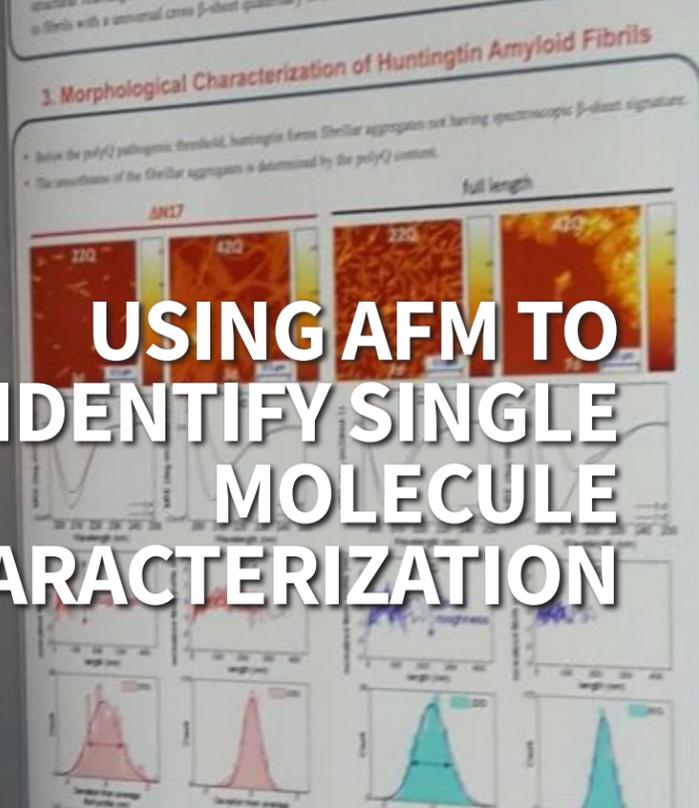


Figure 2. AFM imaging of A) α -synuclein, involved in Parkinson's and B) $A\beta_{42}$, involved in Alzheimer's fibrillation process after incubation of the proteins for 0 d, 7 d, 10 d.

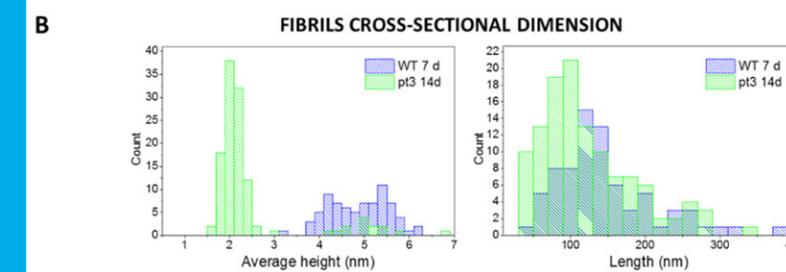
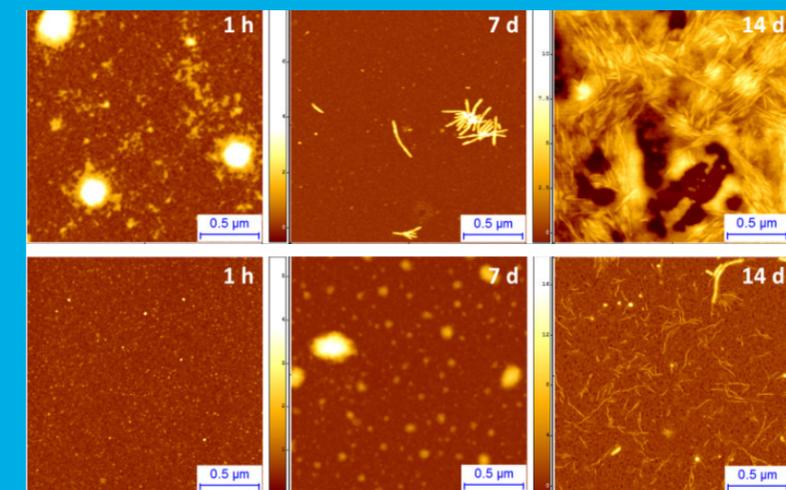


Figure 3. (A) Aggregation of wild type and mutated pT3 huntingtin followed by AFM. (B) Height and length distributions of mutated pT3 (green) a wild type oligomeric structures.

More specifically, I pursue this objective by bringing together atomic force microscopy, with cutting-edge biophysical techniques, such as microfluidics and biosensors to protein science and biophysics. As consequence, a major part of my work is the successful application of nanoscale imaging by means of atomic force microscopy (AFM) in order to investigate the biophysical properties and formation of amyloids at the single aggregate scale. AFM has emerged in the last decade as one of the most powerful and versatile single molecule techniques because of the possibility to acquire 3-dimensional morphology maps of specimens on a surface. This capability has been widely used in the field of protein aggregation and amyloid fibril formation. Indeed, a simple AFM map provides extremely valuable information at the nanometer scale on the structure of amyloid fibrils, such as height, width, periodicity, and the packing of single protofilaments inside mature fibrils (Ruggeri, Habchi et al. 2016).

A single molecule technique, such as AFM, allows one to gain information on the fibrillation process and on individual protein structural transition. The possibility to analyze the morphology at several time points, during the process of amyloid aggregation, enables one to shed light on the mechanisms of protein misfolding, on the pathway of aggregation and the hierarchical polymorphic process of assembly. Figure 2 displays the fibrillation process of α -synuclein and $A\beta_{42}$ by means of AFM imaging after deposition of the proteins on a positively functionalized APTES-mica substrate (Ruggeri, Adamcik et al. 2015).

In addition, AFM imaging can be also used to compare the kinetics of aggregation of different mutated forms of the same protein. Indeed, the study of the effects of mutation on the aggregation of amyloidogenic proteins is of fundamental importance to shed light on their role in both physiology and association with disease. For example, the aggregation of Huntingtin protein and its mutated forms is linked to the onset of Huntington's disease. Increasing evidence suggests that specific N-terminal post-translation modifications within the N-terminus of Huntingtin play a role in the pathogenesis of the illness. For this reason, we focused on the study of their fibrillation and on differences between wild type and a mutated form of this protein (Ansaloni, Wang et al. 2014, Ruggeri, Vieweg et al. 2016, Chiki, DeGuire et al. 2017). As an example, we show here how our AFM studies allowed us to quantitatively follow the kinetics of amyloid fibril formation, and to show that the N-terminus phosphorylated (pT3) mutated form of the protein significantly slows the fibrillation process by forming fibrils with smaller diameters and shorter lengths (Fig. 3).

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ABRIDGED ARTICLE:

DETECTING NANOSCALE VIBRATIONS AS SIGNATURE OF LIFE

Przybylski's Star in the Centaurus constellation.
Eckhard Slawik / Science Photo Library / Getty Images

Original article by Sandor Kasas^{ab}, Francesco Simone Ruggeri^a, Carine Benadiba^a, Caroline Maillarda, Petar Stupar^a, H el ene Tournu^c, Giovanni Dietler^a, and Giovanni Longo^a

^aLaboratoire de Physique de la Mati ere Vivante, Institut de Physique des Syst emes Biologiques, Facult e des Sciences des Base,  cole Polytechnique F ed erale de Lausanne, 1015 Lausanne, Switzerland; ^bD epartement des Neurosciences Fondamentales, Facult e de Biologie et de M edecine, Universit e de Lausanne, 1005 Lausanne, Switzerland; and ^cDepartment of Molecular Microbiology, Vlaams Instituut voor Biotechnologie (VIB), B-3001 Leuven, Belgium

Abridged by Park Technical Staff

Based on the principle that one of the common signatures of life is movement, because even small microorganisms vibrate in response to their metabolic activity, research done in 2014 with Francesco Simone Ruggeri and others explored the use of an innovative nanoscale motion sensor that could be used in life-searching experiments in Earth-bound and interplanetary missions. This nanomotion detector is used to study these fluctuations and associate them to the metabolic activity of the specimens. This technique does not measure the chemical response of life, which would require prior knowledge of the metabolic

pathways involved. Instead, it monitors the physical manifestation of any kind of metabolic activity the microorganisms might have. This research showed how this nanomotion detector can study any living system, paving the way to a complementary approach to the study of life in extreme environments.

The working principle of the technique may be summarized as follows. A microfabricated atomic force microscopy (AFM) cantilever is inserted into an analysis chamber, and specimens are attached to its surface. The cantilever transduces the movements of the samples with a sub-nanometer resolution. The dynamic deflections of this sensor are detected and recorded using a laser-based transduction system. The time resolution and sensitivity of this system make it ideal to study living specimens at the nanoscale. It can be operated in air or in a liquid environment; in this latter case, the living specimens can be exposed to triggering or inhibiting chemicals to characterize their response to the stimuli.

AFM proved to be a valuable technique to exploit the sensitivity of nanomechanical oscillators to transduce the small fluctuations that characterize living systems. By combining chemical and dynamical measurements, this method could help future missions seeking to explore the presence of life on satellites of the giant planets, such as Europa (Jupiter) or Titan and Enceladus (Saturn).



Europa, one of Jupiter's moons, has an icy shell, but scientists believe there is an ocean beneath.

Since Europa has water, future missions will search for signs of life. In February, NASA received a report for a possible landing mission to Europa, which has been in the works since June 2016. In the landing mission, a probe will search for evidence of life on Europa, assess its habitability, and characterize the surface and subsurface for future explorations to this moon and its ocean.

This ocean has at least twice as much water as Earth's oceans. While recent discoveries have shown that many bodies in the solar system either have subsurface oceans now, or may have in the past, Europa is one of only two places where the ocean is understood to be in contact with a rocky seafloor (the other being Saturn's moon Enceladus). This rare circumstance makes Europa one of the highest priority targets in the search for present-day life beyond Earth.



GRAND OPENING OF THE PARK NANOSCIENCE CENTER AT SUNY POLYTECHNIC INSTITUTE

RIBBON CUTTING CEREMONY AT THE GRAND OPENING OF THE PARK NANOSCIENCE CENTER AT SUNY POLYTECHNIC INSTITUTE ON NOV. 10, 2017

Pictured (left to right): Dr. Ryan Yoo, Vice President of Sales at Park Systems, Mr. Keibock Lee, President and General Manager at Park Systems, Dr. Sang-il Park Chairman & CEO from Park Systems, Prof. Alain Diebold, Interim Dean at the College of Nanoscale Science at SUNY Polytechnic Institute, Dr. Ardavan Zandiatashbar, Technical Accounts Manager and Sr. Applications Scientist at Park Systems

The Park Nanoscience Center in Albany, NY is a new branch of Park Systems and will showcase advanced atomic force microscopy (AFM) systems, demonstrate a wide variety of cutting-edge applications—ranging from materials science, to chemistry and biology, to semiconductor and data storage devices—and provide hands on experience, training and service, year-round.

The Center's grand opening at the State University of New York Polytechnic Institute (SUNY Poly), one of the world's most advanced high-tech education, research and development sites, was held on November 10, 2017 at 2 PM. The Center is located in the NanoFab East Building of SUNY Poly's Albany campus and is designed to become a hub for globally advanced metrology AFM research activities. It will be equipped with the latest

Park AFM systems, including the Park NX20, Park NX10, and Park NX-Hivac. Park Systems, a global AFM manufacturer, has offices in key cities worldwide, including Santa Clara, California; Tokyo, Japan; Singapore; Heidelberg, Germany; and Suwon and Seoul, South Korea.

SUNY Poly's Albany NanoTech Complex is home to the College of Nanoscale Sciences and the College of Nanoscale Engineering and Technology Innovation and is a fully-integrated research, development, prototyping, and educational facility that provides strategic support through outreach, technology acceleration, business incubation, pilot prototyping, and test-based integration support for onsite corporate partners, including IBM, GlobalFoundries, Samsung, TSMC, Applied Materials, Tokyo

Electron, ASML, and Lam Research, as well as other next-generation nanotechnology-based research activities.

"As SUNY Polytechnic Institute provides cutting-edge educational and research and development opportunities, it is exciting that Park Systems will establish 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."



“SUNY POLY IS THRILLED THAT A WORLDWIDE LEADER IN ATOMIC FORCE MICROSCOPY IS SELECTING THE CAMPUS FOR ITS NEWEST LOCATION, AND WE WARMLY WELCOME PARK SYSTEMS AS WE LOOK FORWARD TO WORKING CLOSELY TO ADVANCE RESEARCH CAPABILITIES IN THIS IMPORTANT AREA,”

- DR. BAHGAT G. SAMMAKIA, INTERIM PRESIDENT OF THE SUNY POLYTECHNIC INSTITUTE, THE WORLD’S MOST ADVANCED, UNIVERSITY-DRIVEN RESEARCH ENTERPRISE AND HOME OF PARK SYSTEMS NEW NANOSCIENCE CENTER



Pictured (left to right): Dr. Tae-Gon Kim, Senior Researcher from imec, Mr. Phil Kaszuba, Senior Member of Technical Staff from Global Foundries, Dr. Sang-il Park, Chairman & CEO from Park Systems, Dr. John Allgair, 2.5D/3D Program Manager for BRIDG at the University of Central Florida, Dr. William Wilson, Executive Director of the Center for Nanoscale Systems at Harvard University, Prof. Gwo-Ching Wang, Travelstead Institute Chair Professor at Rensselaer Polytechnic Institute

PARK NANOSCIENCE CENTER GRAND OPENING EVENT

In his opening remarks at the ceremony Dr. Sang-il Park, Park Systems Chairman & CEO, highlighted Park Systems’ continued growth and success as the world leading atomic force microscope manufacturer for over two decades. This year marks the 20th anniversary for Park Systems and in 2015 the company went public, becoming the only successful public offering for an AFM business. Since the IPO, their stock has increased by 168% and many companywide growth initiatives have set the platform for continued future success.

Park’s recent accomplishments include expansion of their Korean facility and the creation of a new advanced cleanroom.

They also opened offices in Taiwan, Germany, and Singapore, and added key personnel for global expansion. The opening of the Park Nanoscience Center at SUNY Polytechnic Institute adds Park Systems to a prestigious list of material suppliers, equipment suppliers, and IC manufacturers at that location.

SUNY Polytechnic is a very semiconductor-centric site. “We have a national treasure here in terms of a robust ecosystem for semiconductor research,” commented 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. Dr. Diebold emphasized that the new Park AFM can solve challenges in measurement in material science as well.

“WE ALSO HAVE NANOBIOLOGISTS HERE AT SUNY WHO ARE EXCITED TO USE THE PARK AFM TO LOOK AT CELLS IN VARIOUS ENVIRONMENTS AND ALL THE VARIOUS BIOLOGICAL APPLICATIONS.” DR. DIEBOLD WAS ONE OF THE INVITED GUESTS WHO SPOKE AT THE PARK NANOSCIENCE GRAND OPENING CEREMONY.



“PARK SYSTEMS AND RPI HAVE A SPECIAL HUMAN CONNECTION BASED ON A COMMON PHILOSOPHY OF DEVELOPING A FUTURE GENERATION WORK FORCE AND A STRONG EMPHASIS ON TECHNOLOGY INNOVATION AND SCIENTIFIC DISCOVERY.”

Professor Gwo Ching Wang from Rensselaer Polytechnic Institute (RPI)

Prof. Gwo Ching Wang from Rensselaer Polytechnic Institute also spoke at the opening ceremony. She remembers RPI’s first connection with Park Systems and said in the early days their Park AFM was and still is a workhorse for their group. Not only do the PhD and master’s students use the AFM for research, but every one of their physics undergrads uses Park AFM in their experimental physics class.

Dr. John Allgair, 2.5D/3D Program Manager for BRIDG at the University of Central Florida said he is very excited that Park is opening a center at SUNY Poly and said they will use the Park AFM equipment in their development of a smart sensor fabrication system which will focus on images. The advanced and technically sophisticated smart sensor system will be built on a chip and will be useful for numerous applications such as autonomous cars.

“We will use Park AFM for surface analysis to support wafer bonding and die bonding and to integrate heterogeneous systems for surface activation bonding for just a couple of examples,” commented Dr. Allgair at the Park

Nanoscience Center Grand Opening. “We have a high need for compositional analysis using Park AFM and are very impressed with the versatility of the Park AFM system.”ed with the versatility of the Park AFM system.”

Dr. William Wilson, Executive Director of the Center for Nanoscale Systems (CNS) at Harvard University, welcomed Park Systems to SUNY Polytechnic Institute in his speech. Harvard CNS provides a collaborative multidisciplinary research environment to support world-class nanoscience and technical expertise. The northeast is a hub for everything nanoscience and collaborative research facilities serve an important and vital role in shared knowledge and research opportunities.



“THE PARK NX-3DM AFM RUNS 24 HOUR NON-STOP IN OUR (IMEC) WAFER FABRICATION OPERATION AND IS THE ONLY AFM THAT CAN DELIVER SIDEWALL VISUALIZATION WITH A STURDY ROBUST AND ACCURATE PLATFORM AND UNIQUE NON CONTACT TECHNOLOGY.”

imec Senior Researcher, Dr. Tae-Gon Kim

Dr. Tae-Gon Kim, who has spent years at imec working on developing integrations for Park AFM into various industrial processes, says this is just the beginning for the uses of Park Systems’ patented true non-contact mode single probe AFM.

Mr. Phil Kaszuba, Senior Member of Technical Staff from GlobalFoundries, said that Park AFM scanning probe microscopy technology has evolved into a mainstream analytical technique throughout the semiconductor industry over the past twenty years, steadily

progressing towards nanoscale microscopy methods currently analyzing images just 26 atoms across. “Park Systems has taken a giant step towards the further advancement of science with the opening of the Park Nanoscience Center at SUNY Poly,” states Mr. Kaszuba.

“Increasingly, AFM is being selected for nanotechnology research over other metrology techniques due to its non-destructive measurement and sub-nanometer accuracy,” states Dr. Sang-il Park, Park Systems Chairman and CEO. “The new Park Nanoscience Center at SUNY Polytechnic Institute provides researchers with greater access to Park Systems’ cutting-edge AFM nanoscopic tools, featuring reliable and repeatable high-resolution imaging of nanoscale cell structures in any environment without damage to the sample.”

Park Systems’ advanced AFM platform includes Park SmartScan, an innovative and pioneering AFM intelligence that produces high-quality imaging with a single click. SmartScan’s unique design opens up the power of AFM to everyone and drastically boosts the productivity of all users. “With Park AFM, throughput is significantly improved and time-to-solution and data reliability are exceptional compared to other metrology solutions,” adds Dr. Park.

The Park Nanoscience Center at SUNY Poly will be a unique source for researchers who are looking for the most advanced developments in scanning probe microscopy for materials research, analytical chemistry, life science research, and semiconductor metrology.

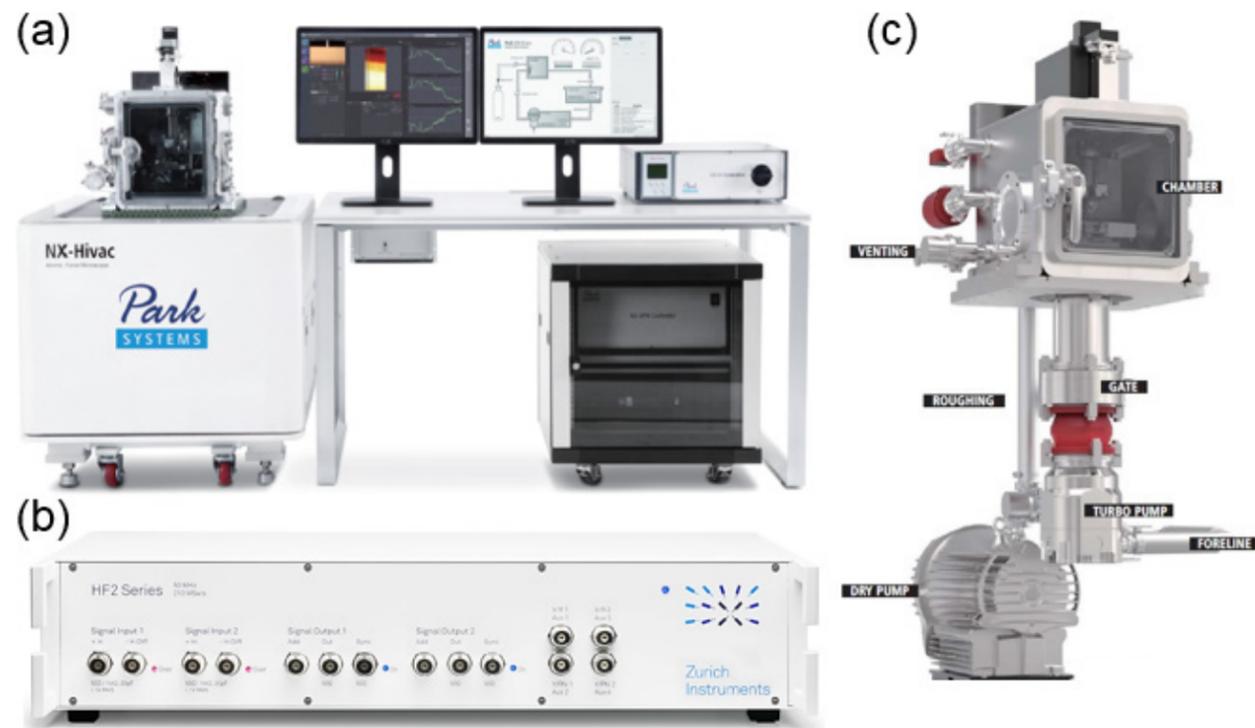


Figure 1 - (a) Park NX-Hivac and (b) HF2PLL system (c) The vacuum pump system

PARK NX-HIVAC: PHASE-LOCK LOOP FOR FREQUENCY MODULATION NON-CONTACT AFM

Romain Stomp^a, James Wei^a, Hosung Seo^b,
Dan Goo^b, and Gordon Jung^b
^aZurich Instruments; ^bPark Systems

Phase-locked looping is a powerful technique that allows synchronization of multiple measurements such that several signals can be acquired at precisely the same moment in time. Combining the phase-locked loop (PLL) with an Atomic Force Microscope (AFM) enables capabilities such as frequency modulation (FM) AFM, which allows researchers to observe the dynamic properties of an oscillating cantilever which can be used to quantify surface potential measurements. This document reports the successful operation of a phase-locked loop (Zurich Instruments HF2PLL with Lock-in Amplifier [HF2LI]) with a Park Systems AFM [Park NX-Hivac]. The HF2PLL is an open market instrument supporting higher frequencies, multi-mode measurements, and advanced imaging modes such as amplitude modulation (AM) and FM for surface potential measurements. Moreover, the HF2PLL increases the power of Park Systems AFMs by enabling the operation

of two cantilever modes simultaneously (performing measurements with 2PLLs) and demodulation of up to 6 arbitrary frequencies. To maximize the utility of a combined PLL-AFM, a high vacuum environment offers a high quality (Q) factor, which allows for increased adjustability of cantilever dynamics and Z height sensitivity. Taken together, the new Park NX-Hivac from Park Systems is an ideal system, offering an AFM environment with a base pressure well below 10⁻⁵ Torr with a Q factor three times greater than ambient situation. In this technical note, we will present the methodology for interfacing a Zurich Instruments PLL with a Park Systems controller using a Park NX-Hivac to control cantilever dynamics in Frequency Modulation Non-Contact AFM operation.

INTRODUCTION

Park Systems Park NX-Hivac allows failure analysis engineers to improve the sensitivity and resolution of their measurements in a high vacuum environment. High vacuum scanning offers improved accuracy, repeatability, and

less tip and sample damage than ambient or dry N₂ conditions. The Park NX-Hivac is not only the world's most accurate high performance AFM, but it is also one of the easiest and most convenient AFMs to use for failure analysis applications. With Park NX-Hivac, you can increase your productivity and trust that your results are sound. Combining the Park Systems NX-Hivac (Fig. 1a) with the Zurich Instruments PLL (Fig. 1b) enables modes such as amplitude modulation and frequency modulation imaging.

The Park NX-Hivac system features an evacuation chamber that houses an AFM. The chamber is connected to two vacuum pumps as shown in Fig. 1c. First the roughing valve opens to pump the system to 10⁻² Torr. Higher vacuum is then achieved by activation of a turbo pump, increasing the vacuum to 10⁻⁵ Torr. Standard AFM cantilevers made from silicon or silicon nitride exhibit very high Q values in vacuum. This slow response time is advantageous for FM mode, which works well for high-Q systems. In addition, by using a vacuum chamber, oxidation of the specimen can be prevented.

An Overview of NC-AFM

NC-AFM technique guarantees constant transfer function response by tracking any change in phase or amplitude at the cantilever resonance. This allows for quantitative measurements of conservative interaction (induced by frequency shift change) and dissipative interaction (energy put into the system to keep the amplitude constant). In contrast, intermittent-contact (IC) mode operates in an open loop with direct lock-in amplifier measurements at a fixed frequency (near-resonance). The NC-AFM mode operates in a closed loop to ensure both the phase and the amplitude measurements are taken exactly at resonance. Any tip-sample interaction will induce a change in the free resonance frequency f_0 either toward positive shift in frequency $f_0 + \Delta f$ due to repulsive interactions, or negative shift $f_0 - \Delta f$ due to attractive interactions.

Amplitude modulation

Amplitude modulation (AM) was one of the original modes of operation introduced by Binnig and Quate in their seminal 1986 AFM paper. (1) In this mode, the sensor is excited at a frequency that is slightly offset from the resonance frequency. By exciting the sensor just above its resonant frequency, it is possible to detect forces which change the resonant frequency by monitoring the amplitude of oscillation. An attractive force on the probe causes a decrease in the sensors resonant frequency, thus the driving frequency is further from resonance and the amplitude decreases. The opposite is true for a repulsive force. The AFM microscope's control electronics can then use amplitude as a reference channel, either in feedback mode, or it can be recorded directly in constant height mode. AM can fail if the non-conservative forces change during the experiment, as this changes the amplitude of the resonance peak itself, which will be interpreted as a change in resonant frequency. Another potential problem with AM is that a sudden change in sample-time interactions (increased repulsive forces) can shift the cantilever resonant frequency past the drive frequency. This will ultimately cause an instability that would typically lead to an image artifact in constant height mode, but in feedback mode the feedback will read this as a stronger attractive force, causing positive feedback until the feedback saturates. An advantage of AM is that there is only one topography feedback compared to three in frequency modulation (the phase/frequency loop, the amplitude loop, and the topography loop), making both operation and implementation much easier. AM, however, is rarely used in vacuum as the Q of the sensor is usually so high that the sensor oscillates many times before the amplitude settles to its new value, thus slowing operation.

Frequency modulation

Frequency modulation (FM), introduced by Albrecht, Grütter, Horne and Rugar in 1991, is a mode of NC-AFM where the change in resonant frequency of the cantilever is tracked directly, by continually exciting the cantilever at resonance. (2) The energy exchange is maximized if the driving signal has a 90° phase difference with respect to the oscillation of the cantilever. Therefore, to perform FM-AFM practically, the electronics must maintain this shift by either driving the sensor with the deflection signal phase shifted by 90°, or by using an advanced phase-locked loop which can lock to a specific phase. (3) The microscope can then use a reference channel to monitor the change in resonant frequency (Δf) operating in either feedback mode or constant height mode. While recording frequency-modulated images, an additional feedback loop is typically used to keep the amplitude of resonance constant, by adjusting the drive amplitude. By recording the drive amplitude during the scan (usually referred to as the damping channel as the need for a higher drive amplitude corresponds to more damping in the system), a complementary image is recorded showing only non-conservative forces. This allows conservative and non-conservative forces in the experiment to be separated. This new resonance frequency f_0 corresponds to the PLL output and to the new reference frequency for the lock-in measurements, illustrated in the upper feedback loop of the Fig. 2. This induced 'FM' on the cantilever resonance is also referred to as the FM-NC-AFM technique. Once the phase is locked, the same lock-in measurements provide the amplitude at the resonance and further establish a closed-loop operation to maintain this set-point by adjusting the drive excitation, shown in the lower feedback loop of the Fig. 2.

To benefit from steep slope sensitivity (i.e. high Q factor in vacuum), it is common practice to use a PLL based on the phase measurements to capture the resonance frequency shift resulting from the tip-sample interaction. In this manner, the resonator settling time is no longer determined by the natural bandwidth of the resonator, namely $f_0/2Q$, but rather by the user-defined PLL bandwidth in a closed-loop operation. This is sometimes also referred to as the constant amplitude mode.

Optimization of NC-AFM parameters

The Lennard-Jones potential results from the contribution of short and long range forces, which is both non-linear and non-monotonic. The choice of frequency shift set-point (directly related to the interacting force) is therefore crucial and the best way to determine a set-point and Z-controller slope to perform a force (i.e. frequency shift)-vs-distance curve.

Depending on the depth of the interaction well, three conditions, as shown in Fig. 3, can be set for repulsive, attractive, or minimum of the well (using differential measurements with height modulation). It is useful to note that in the repulsive regime, the dissipation channel can also be used as feedback for height. Dissipation has the advantage of exhibiting a monotonic decay with increasing tip-sample distance. (4) In addition, since electrostatic contribution is ubiquitous on most surfaces (from trapped charges, to different material composition and stray fields) it is also recommended that the experimenter evaluate if these forces are contributing to their signal by vary the tip-sample bias voltage once the PLL (on the phase) and PID (on the amplitude) are locked. Dissipation-vs-bias and frequency shift-vs-bias plots can be generated through a simple

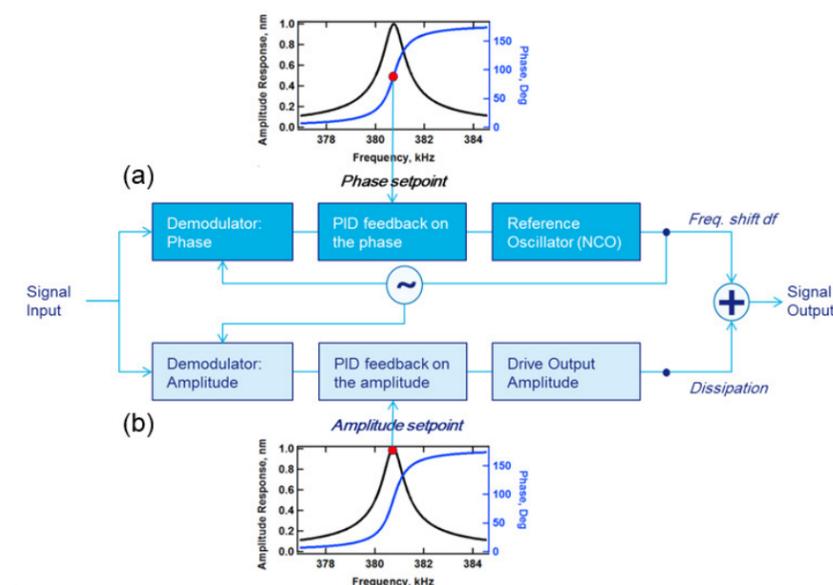


Figure 2 - Feedback loop where (a) FM and (b) AM techniques occur at the same time using HF2PLL system.

sweep, at constant height above the surface (i.e. Z-controller feedback off). The parabolic behavior of the electrostatic force can be displayed and its contact potential difference (CPD) determined from the maximum of the parabola. Electrostatic interactions can also be useful to ensure a smooth landing by generating an 'electrostatic cushion': a DC offset (away from CPD) generates a large electrostatic force thus producing long range interaction that the tip can probe while still being far away from the surface.

In this technical note, we will show an example of how to generate the Δf -distance curve and the dissipation-bias curve, allowing for imaging.

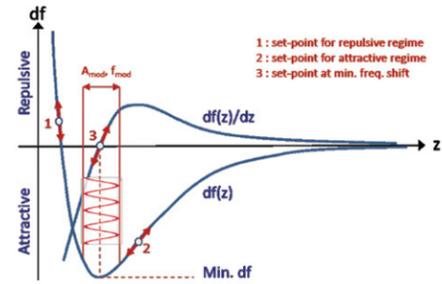


Figure 3 - A representative frequency shift-distance curve. The forces reflect the Lennard-Jones potential for sample-tip interactions.

EXPERIMENTAL

Measurement (Park NX-Hivac) Setup

Example measurement

The Park NX-Hivac AFM was used with a cantilever that was 125 μm long and 4 μm thick with a nominal resonance frequency of 330 kHz (free space) and a quality factor of 600. Typically, a 14 mV (peak-peak) drive amplitude led to the position sensitive photo detector (PSPD) signal of 450 mV in vacuum & free space (spring constant of 42 nN/nm). The cantilever dynamics was detected by laser reflection onto a 4-quadrant photodiode (see Fig. 4).

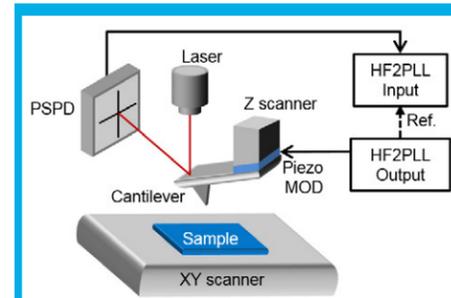


Figure 4 - AFM diagram for NC-AFM. A cantilever is brought into frequency/amplitude feedback and modulated by a piezo modulation (MOD). The vertical and lateral cantilever movement is detected by a PSPD using laser reflection on the cantilever.

Requirements

To connect the HF2PLL with the Park NX-Hivac AFM, a Signal Access Module (SAM provided by Park Systems) and 5 BNC cables are required. Moreover, the reader should be familiar with the functionality of both the HF2PLL and the AFM. Both instruments are controlled with their original operation software. The HF2PLL can be connected to an independent computer that runs the control software ziServer and ziControl on either a Windows 7, Vista or Linux platform. The Park NX-Hivac AFM is operated from a Windows 7 based computer via the software SmartScan version RTM 8. The vacuum system is controlled by Hivac Manger, where pumping and venting processes are logically and visually controlled using one click. Each process is visually monitored by color and schematic changes, streamlining the vacuum operation. Faster and easier vacuum control software brings you ease of use AFM operation and better productivity. AFM data can be displayed and analyzed with Park system's standalone analysis software package XEI.

FM-NC-AFM

Initially a direct comparison of one of the lock-in amplifiers integrated in the NX-Hivac controller and the external HF2PLL can be achieved by connecting the following items:

- **The vertical deflection signal** (connector labeled Monitoring A-B on the SAM) to the Signal Input 1 of the HF2PLL
 - **The HF2PLL Signal Output 1** to the drive piezo modulation input (connector Driving Piezo MOD on the SAM)
 - **The HF2PLL Auxiliary Outputs 1 (Δf)** to the feedback set-point input of the Z scanner (connector Driving A-B on the SAM)
 - **The HF2PLL Auxiliary Outputs 2 and 3 (Aux 2/3)** to the NX-Hivac Controller back panel Aux 2 and 3
- See Fig. 5 for a schematic of the connections between the PLL and AFM. The switches on the

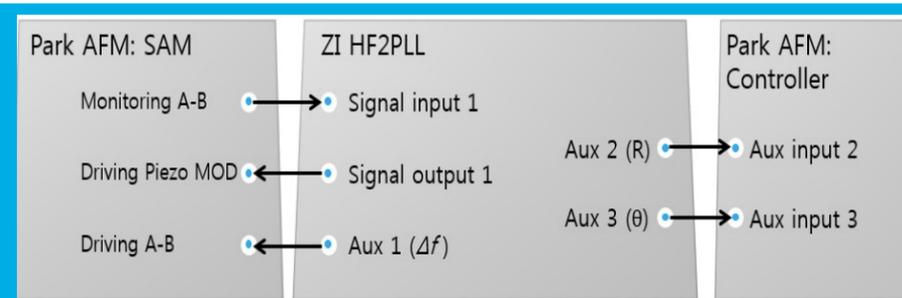


Figure 5 - Setup BNC connections: Park NX-Hivac with the four inputs for recording and controlling of image data and the one output for input the PSPD signal to HF2PLL, HF2PLL with Signal Input 1, Signal Output 1, and the Auxiliary Outputs 1 to 3, SAM with the Vertical Output (PSPD A-B signal), the input for the piezo modulation and the change in frequency due to the interaction between the sample and tip.

SAM can be set to External \rightarrow Controller to input an external signal into the system and breaking the internal signal line from the NX-Hivac controller.

Definitions of Recorded and Displayed Quantities of the HF2PLL

- **Vertical cantilever amplitude R:** The amplitude reflects the information related to cantilever vibration.
- **Phase Theta θ :** The difference in phase between the cantilever drive frequency and actual cantilever oscillation. This parameter is to tip-sample-interactions (damping).
- **Delta frequency Δf of PLL:** By tracking the phase of the resonant frequency, Δf reflects the variation from the set frequency in air.

In addition, the horizontal cantilever deflection can be detected in all modes of operation discussed here. By changing the switch position on the SAM to External \rightarrow Controller the cantilever driving piezo modulation is controlled by the HF2PLL and the Vertical PSPD Output (A-B) is applied to the Signal Input 1 for demodulation. The change in frequency due to the interaction between the sample and tip to the two signals connect to the set-point signal of the AFM feedback loop. The demodulated signals R and θ are routed via Auxiliary Outputs 2 and 3 to the analog inputs of the Park NX-Hivac controller for recording and imaging.

RESULTS AND DISCUSSION

Sample Characterization

Δf -distance curve measurement (tip-sample distance)

The samples measured here are highly oriented pyrolytic graphite (HOPG) and silicon. First, care must be taken in the approach process in FM-NC-AFM due to sample-tip interactions. In vacuum, air

damping is negligible, which makes it challenging to approach the sample surface. To evaluate damping, a Δf -distance curve is performed (see Fig. 6). The air damping value depends on the tip condition and increases when the tip is blunted or damaged. Also, in air in the imageable frequency, 43.7 Hz in air and 8.6 Hz in vacuum changes every 1 nm as the distance between tip and specimen closes.

Δf - bias curve measurement

For FM-NC-AFM, the approach process requires minimal noise due to the operation of the motor. To approach the sample in a gentle manner, air damping is used regardless of whether the system is in air or vacuum. In air, damping is significant however, in vacuum the air damping is minimal; therefore, forced air damping is employed by applying a tip bias. As shown in Fig. 7, we can confirm that air damping increases by applying a tip bias. When the Z scanner height is -0.5 μm and the tip bias is varied from 0 V, 2 V to 4 V, the Δf value are 0.81 Hz, 1.01 Hz and 1.72 Hz, respectively. After stopping the Z stage using the value of the forced air damping interval, the approach can be completed by entering the area of interest for imaging with the Z scanner and low noise. The tip bias is used because the sample bias is not affected by the tip when using an insulating sample.

FM-NC-AFM image in vacuum condition

Fig. 8a-b show the topography and error signal for HOPG, respectively. It has a 10 μm x 10 μm image size and several layers are visible. Fig. 8c and Fig. 8d shows the topography and error signal for silicon, respectively. The image sizes are measured to be 0.2 μm and confirmed to have high resolution with a roughness RMS of 0.382 nm. This image is measured at a scan rate of 1 Hz and can be measured at a faster rate.

Setting parameter value

- Resonance frequency: 306.516 kHz
- Z-feedback set point: 10.83 Hz
- Amplitude swing: 4.5 nm
- Δf full range: +/- 20.83 Hz
- Cantilever stiffness: 35 N/m

Conclusions

FM-NC-AFM operation allows for quantitative tip-sample interaction measurements with high sensitivity from high Q factor in vacuum. It has been demonstrated to work seamlessly with the new Park NX-Hivac system from Park Systems, providing experimentalists with additional AFM modes to probe both mechanical and electrostatic interactions simultaneously, in a well-controlled environment.

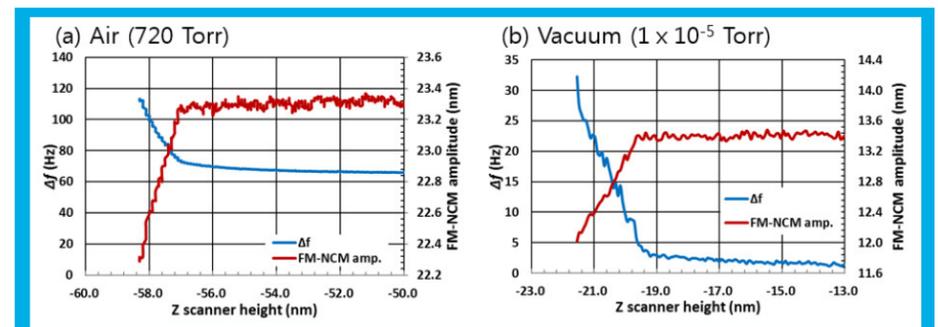


Figure 6 - Δf -distance curve in a narrow Z scanner height range (10nm) about (a) air and (b) vacuum condition.

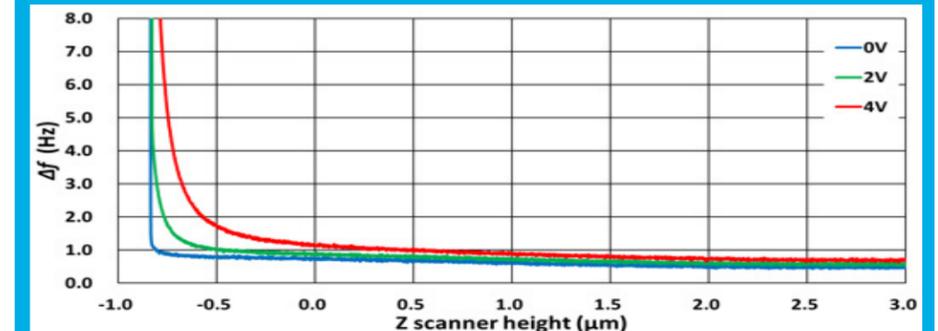


Figure 7 - Δf -tip bias curve in vacuum. The Δf value begins at 0 Hz (graph is read right to left) and when the slope of the FM-NC-AFM amplitude increases sharply, the tip has approached the surface. As the tip bias is increased from 0 to 4 V, the approach is increasingly dampened and the sharp increase in Δf occurs further from the sample surface with increased voltage.

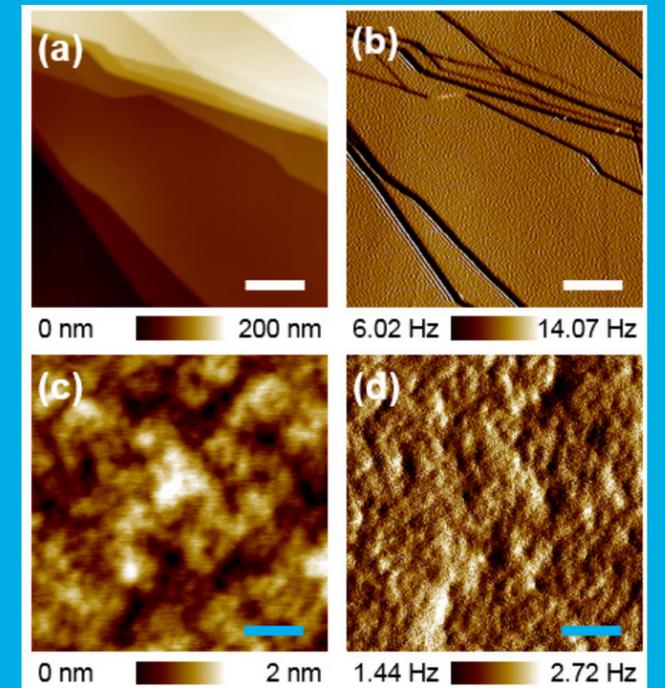


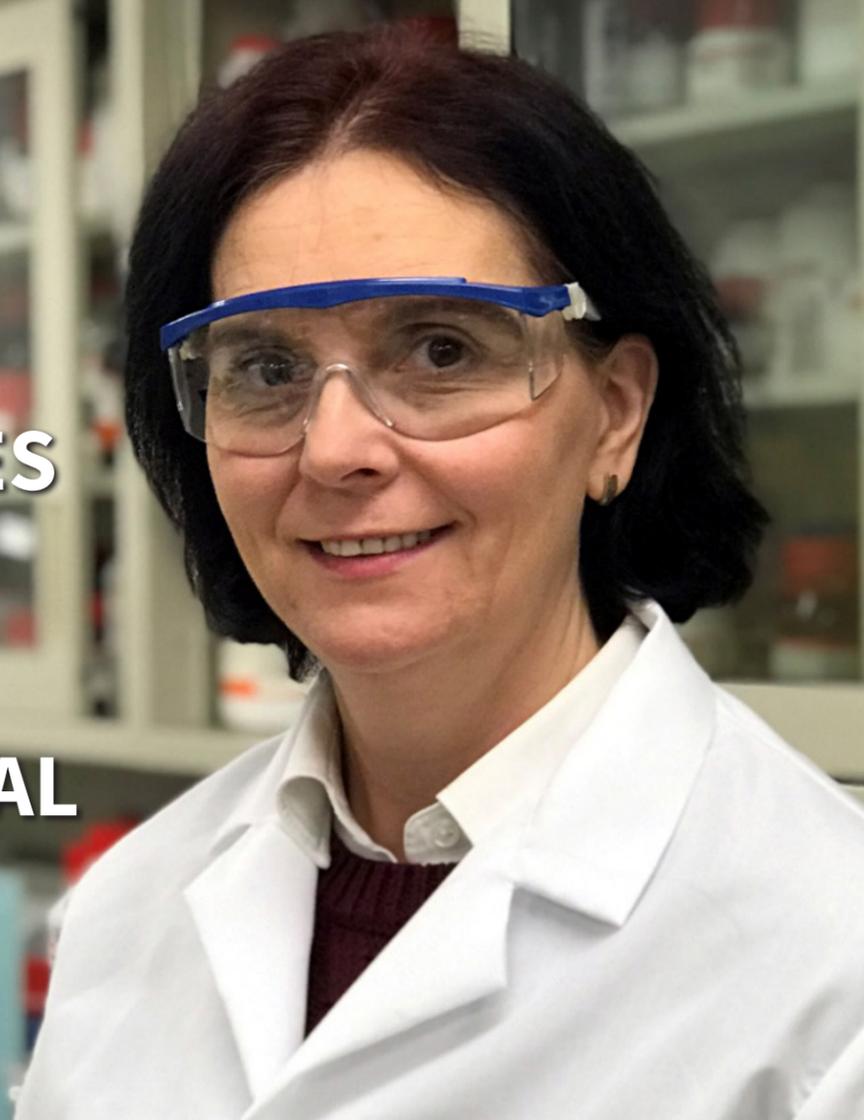
Figure 8 - FM-NC-AFM image in vacuum condition. HOPG (a) topography and (b) error signal (Δf). White bar is 2 μm . Silicon (c) topography and (d) error signal (Δf). Blue bar is 40 nm.

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USING NANOPARTICLES TO IMPROVE OIL RECOVERY AND REDUCE ENVIRONMENTAL IMPACT

AN INTERVIEW WITH
BATTELLE'S DR. OLGA KOPER



BREAKTHROUGH GREEN CHEMISTRY SOYBEAN-BASED SURFACTANT UNDER DEVELOPMENT AT BATTELLE

Dr. Koper has a Master of Sciences in General Chemistry from Silesian University, Poland and a Doctor of Philosophy in Inorganic Chemistry from Kansas State University. In 2013, she was awarded The White House Champions of Change Award for Immigrant Innovators and was chosen by Entrepreneur Magazine as one of seven selections for 2013's Entrepreneurial Women to Watch. She is proposal reviewer for the Department of Defense, National Cancer Institute, and European Commission.

Dr. Olga Koper is responsible for the development and implementation of Battelle's technologies related to advanced materials and technologies for the oil and gas exploration and production industry. She has designed nanomaterials for sweetening of natural gas, water and air filtration, biomass conversion, decontamination of toxic chemicals and biological species, as well as cancer detection and treatment. These

activities resulted in over 30 issued U.S. and international patents and 34 peer-reviewed publications.

Some examples of Dr. Koper's research activities include development of self-healing cement for enhancing wellbore integrity, development of a platform membrane technology for water treatment applications (forward osmosis, reverse osmosis and nanofiltration) for treatment of flowback and produced water from the shale gas industry, utilization of nanoscale metal oxides and carbon nanotubes for supercapacitor electrodes, development of reactive nanoparticles as destructive adsorbents for biological and chemical decontamination. This work resulted in commercialization of a hazard mitigation product (FAST-ACT) as well as several patent and peer-reviewed publications. Development of catalytic topical skin cream protectants based on nanoparticles,

as well as nanoparticles and polyoxometalate combinations: This work was carried out in collaboration with Emory University, leading to two patents jointly owned with the Department of Defense.

Her research has been done in several diverse fields of application. She has designed nanomaterials that can be incorporated into fabric, such as for soldier uniforms, to protect and decontaminate toxic materials. She has worked on nanomaterials for batteries, supercapacitors, air filtration applications, chemical and biological decontamination, natural gas and oil recovery, water purification, and catalysis. Further, Koper has applied nanomaterials to cancer diagnosis and treatment, using functionalized core/shell magnetic nanomaterials to detect over-expressed proteases. The resulting product is injectable, and during surgery can be used to fluoresce and define malignant cells, thus allowing surgeons to fully clear the margins of a cancerous mass.

"AS OIL AND GAS MARKET LEADER AT BATTELLE, I CONNECT ENERGY COMPANIES WITH INNOVATIVE TECHNOLOGIES AND SERVICES THAT SOLVE CRITICAL EXPLORATION AND PRODUCTION CHALLENGES. DRAWING ON MY SCIENTIFIC RESEARCH BACKGROUND, I LEAD INTERDISCIPLINARY TEAMS OF BATTELLE EXPERTS TO DEVELOP GAME-CHANGING SOLUTIONS THAT HELP OIL AND GAS COMPANIES THRIVE IN THE CURRENT LOW PRICE ENVIRONMENT."



Photo Caption: Battelle won two of the recently announced R&D 100 Awards for its PipeAssess PITM software and coal-to-liquids process. In addition, Battelle was on a team with the Ohio Soybean Council, Kentucky State University and Redwood Innovations that won Special Recognition for a technology called EnzoMeal. Center: Dr. Olga Koper.

Since R&D Magazine started the awards 55 years ago, Battelle and its affiliated national labs have won a total of 380 R&D 100 Awards. Known as the "Oscars of Innovation" in the science and technology world, the annual awards recognize the most significant technologies the nation's scientists and engineers create.

BATTELLE MEMORIAL INSTITUTE

As the world's largest, independent research and development organization, Battelle provides innovative solutions to the world's most pressing needs through its four global businesses: Laboratory Management; National Security; Health and Life Sciences; and Energy, Environment and Material Sciences. It advances scientific discovery and application by conducting \$6.5 billion in global R&D annually through contract research, laboratory management and technology commercialization. Headquartered in Columbus, Ohio, Battelle oversees 22,000 employees in more than 130 locations worldwide, including seven national laboratories which Battelle manages or co-manages for the U.S. Department of Energy and the U.S. Department of Homeland Security and a nuclear energy lab in the United Kingdom.

Battelle also is one of the nation's leading charitable trusts focusing on societal and economic impact and actively supporting and promoting science, technology, engineering and mathematics (STEM) education.



Every day, the people of Battelle apply science and technology to solving what matters most. At major technology centers and national laboratories around the world, Battelle conducts research and development, designs and manufactures products, and delivers critical services for government and commercial customers. Headquartered in Columbus, Ohio since its founding in 1929, Battelle serves the national security, health and life sciences, and energy and environmental industries. For more information, visit www.battelle.org.



AN INTERVIEW WITH DR. OLGA KOPER

Business Development/Sales Leader Energy, Battelle

Can you elaborate on the soy based surfactant that is looking very promising for a new green chemistry method?

Battelle is working on developing sustainable oilfield chemicals to reduce production costs as well as environmental impact while improving chemical performance. One such product is a cationic surfactant from soybean oil. The material has been formulated and it being tested for multiple oilfield applications.

How is this new surfactant developed?

Soybean oil, which is used as feedstock, is processed to yield a cationic surfactant. This surfactant is then blended with a co-solvent to optimize its solubility in water as most hydraulic fracturing fluids are water-based.



What are the initial results and how is this a better or "greener" method?

The surfactant is designed based on the principles of green chemistry. Our initial test results suggest the soy-based cationic surfactant shows no signs of estrogenic or anti-estrogenic activity based on BG1

Luc estrogen receptor transactivation test. Hence, our product is showing signs of promise of a greener technology as opposed to conventional cationic products.

Which companies are working on this project and how soon might it be used commercially? What benefits will it have over the current products being used?

Currently, Battelle is in talks with a variety of oilfield chemical companies and operators to further develop and optimize this technology. The technology is still in early stages of development. Time to market will depend on a host of factors, such as the technical progress during the next stage of development, commitment of the commercial client and the specific application of the product, i.e., enhanced oil recovery. The benefits of using this product include:

- Lower production costs
- Reduced environmental impact
- Multiple wellbore applications
- Flexibility in surfactant design



What new technologies is Battelle working on related to nanotechnology? Not just in oil and gas but in other areas as well.

Nanotechnology is part of many applications, from design of catalysts to new sensors. One specific example is anti-icing and deicing technology for in-flight aircrafts based on a resistive heating using carbon nanotubes. This innovative coating solution is small in size, weight and power envelope and has unmatched performance in the industry. This coating can be used on drones when the weight constrains are of paramount importance.

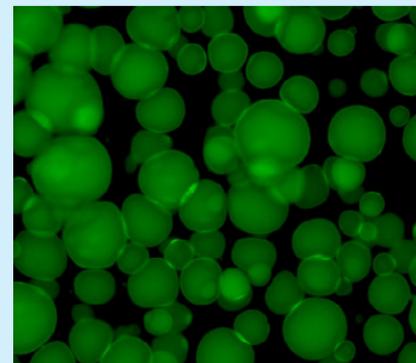
And what do you see as the emerging trends in nanoscience that will revolutionize the world of engineering and have a big impact on society?

The largest impact is related to medicine, and particularly to drug delivery and early detection, particularly non-invasive

using biomarkers. For example, designer nanoparticles can target specific cancer cells increasing treatment effectiveness and decreasing the side effects. Other areas are energy related, such as in energy storage (battery interfaces in Li-ion batteries) and harvesting (solar cells).

What are nanofluids and how are they used to enhance oil recovery? Why are they called smart fluids?

Nanofluids are solutions of nanoparticles (1-100 nm in dimension) and can be used in a variety of oilfield applications such as drilling, stimulation and improving oil recovery. Silica nanoparticles can alter the rock wettability of oil-wet reservoir rocks to water-wet thereby reducing the tendency of the rock to stay in contact with oil. This will eventually lead to higher oil recovery. Nanofluids also help control the mobility of injection fluids which in turn improves the sweep efficiency of injectates and therefore the resulting oil/gas recovery. However, a definition of smart fluids can be much broader and include materials that upon a stimuli will perform specific actions. This could include a controlled release from nano or micro capsules upon exposure to specific temperature, pressure, presence of ions, or other chemicals.



Pictured: Smart nanobeads

Why does the size of the nanoparticles matter, and how is it measured? Do you use atomic force microscope?

For specific applications the size of the particles is crucial. For example, with carbon nanotubes in the deicing coating, the small size will provide lower weight of the coating, which is particularly important for drone applications, to extend the battery life during flights. In drug delivery applications not only size, but also shape and surface chemistry have a large impact on cellular uptake and overall treatment efficiency. Such correlations are studies using many methodologies including AFM.

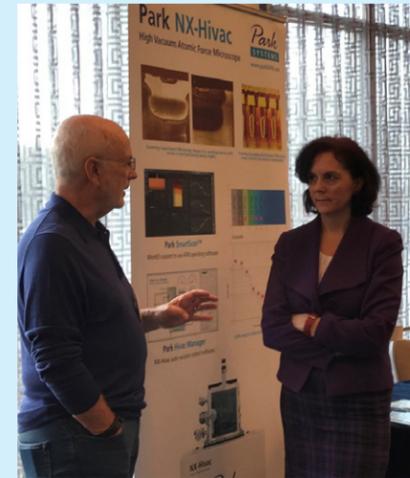


Photo Caption: Dr. Olga Koper, a speaker at the Oil & Gas Chemistry and Polymers 2017 conference, Nov 5-6 in Houston, TX, is shown pictured at the Park Systems booth at the event.

How does the oil and gas industry use controlled release methods or encapsulation for oil production and why? Can you give examples?

An example of a controlled release can be delivery of the anti-scaling agents at the desired place and time. The controlled release can use different trigger mechanisms including temperature, pressure, presence of specific ions, etc. The type of the trigger is usually chosen based on the desired parameters, i.e., if you want the active ingredient to be released uniformly over a two-year period of time, or if you want to release it abruptly when the material reaches downhole conditions (specific temperature and pressure). The encapsulation approach eliminates using excess quantities of materials (cost savings and environmental benefits) as well as decreases labor associated with reapplication.



Photo Caption: Pictured above left to right, Ram Lalgudi and Manoj Valluri from Battelle contributed to this article.

ELECTROCHEMICAL ATOMIC FORCE MICROSCOPY (EC-AFM): IN SITU MONITORING OF COPPER ELECTRODEPOSITION ON GOLD SURFACE



JOHN PAUL PINEDA, MARIO LEAL, GERALD PASCUAL, BYONG KIM, AND KEIBOCK LEE, PARK SYSTEMS

INTRODUCTION

In recent years, understanding electrochemical process such as electrodeposition (also known as electroplating) has become evident in various technologies and sciences including microelectronics, nanobiosystems, solar energy conversion, chemistry, among others, due to its wide range of applications [1,2]. Electrodeposition is a conventional process that utilizes electrical current through a solution called electrolyte to modify surface properties, either chemical or physical to make the material suitable in certain applications. Based on the principle of electrolysis, it is a process that applies direct electrical current into an electrolyte solution to reduce the cations of a desired material and deposit particles onto the conductive substrate surface of the material [3]. This technique is commonly done to enhance electrical conductivity, improve corrosion resistance and heat tolerance, and to make products more aesthetically appealing. A good deposition mainly depends on the substrate surface morphology [4]. Thus, a technique that can measure this characteristic and monitor electrodeposition process in nanoscale is greatly needed. There are several methods that were employed for this surface characterization. Examples include scanning electron microscopy (SEM) and scanning tunneling microscopy (STM). These techniques allow measurement of structures on the nanoscale, however, some of them are ex situ, some typically requires high vacuum, and others are not applicable in monitoring continually changing process because

of its time consuming image acquisition [2,5]. To overcome these shortcomings, Electrochemistry combined with Atomic Force Microscopy (commonly known as EC-AFM) was introduced. This technique allows users to perform in situ imaging and visualization of changes in surface morphology of sample being studied under certain electrochemical environment in nanoscale [6].

In this study, the deposition and dissolution of copper particles into the gold surface was successfully demonstrated. The morphological changes of the copper particles was clearly observed in situ using Park NX10 AFM and a current-voltage (CV) curve was simultaneously acquired using the potentiostat during the experiment.

AFM HEAD AND TIP

A liquid probehead shielded with glass was used instead of conventional probehead to allow measurement in liquid. A NANOSENSORS PointProbe® Plus-Contact (PPP-CONTSCPt) cantilever (nominal spring constant $k = 0.2 \text{ N/m}$ and resonant frequency $f = 25 \text{ kHz}$) coated with platinum and mounted on Teflon chip carrier was used in the experiment. Platinum coated cantilever was selected to maintain beam intensity when the tip is immersed in the solution. Moreover, the tip was mounted on Teflon chip carrier to protect the EC Cell from other unwanted electric signals that could affect the conditions of the electrolyte solution.

EC CELL SETUP

A small EC Cell manufactured by Park Systems was used in the experiment. The cell is made of polychlorotrifluoroethylene (PCTFE) to ensure chemical stability. A sample is mounted with a top cover and sealed with a thin silicon O-ring to securely prevent leaking on the backside of the sample. The working electrode (WE) that was used is gold (Au) in the 111 orientation evaporated on a mica surface while reference electrode (RE) and counter electrode (CE) are silver chloride (AgCl) and platinum-iridium (Pt-Ir). The 3 electrodes were connected on a Solartron Modulab XM potentiostat. The aqueous solution contained 0.1mM CuSO_4 in 50ml of 0.01 mM H_2SO_4 . Sulfuric acid was added in the solution to stabilize and prevent copper to precipitate.

EC-AFM EXPERIMENT CONDITIONS

The deposition and dissolution of the copper particles in gold thin films with 111 orientation was monitored using a Park NX10 system. A reference image of the gold surface was acquired in ambient air and in liquid condition without introducing chemical reaction to serve as a point of comparison of the sample surface before and after the experiment. The images were acquired using non-contact mode, to get high quality image. After obtaining the reference image in-liquid, the 3 electrode was connected into the potentiostat. The CE and RE were bent before immersing in the liquid solution to make a good contact and prevent saturation of the signal. AgCl electrode was utilized as RE since a chloride solution was not used. Furthermore, Pt-Ir electrode was selected as CE since this material is chemically stable, hence it will not contaminate the solution.

In EC-AFM, the WE is the sample surface where the electrodeposition process takes place, the CE is where the electric current is expected to flow. EC cell is like an electronic circuit and the main function of CE is to close the current loop in the circuit, the RE is used as a fixed reference point and serves as a feedback to maintain a stable voltage in the solution. An electric field is supplied in the working electrode to transmit electrons to the ions in the solution so that oxidation or reduction will take place. The type of chemical reaction depends on the amount of voltage supplied (either positive or negative voltage) in the EC cell. In this study, a cyclic voltammetry was applied in the EC cell to know the oxidation and

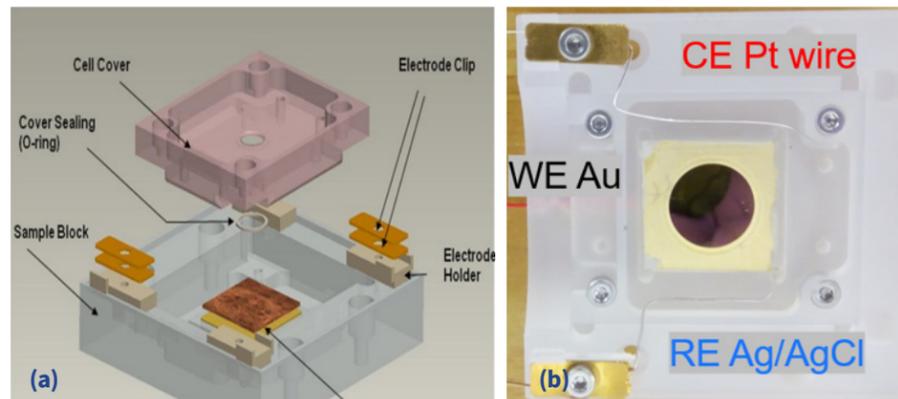


Figure 1. (a) Overall structure of the NX EC Cell (b) Actual setup of EC cell

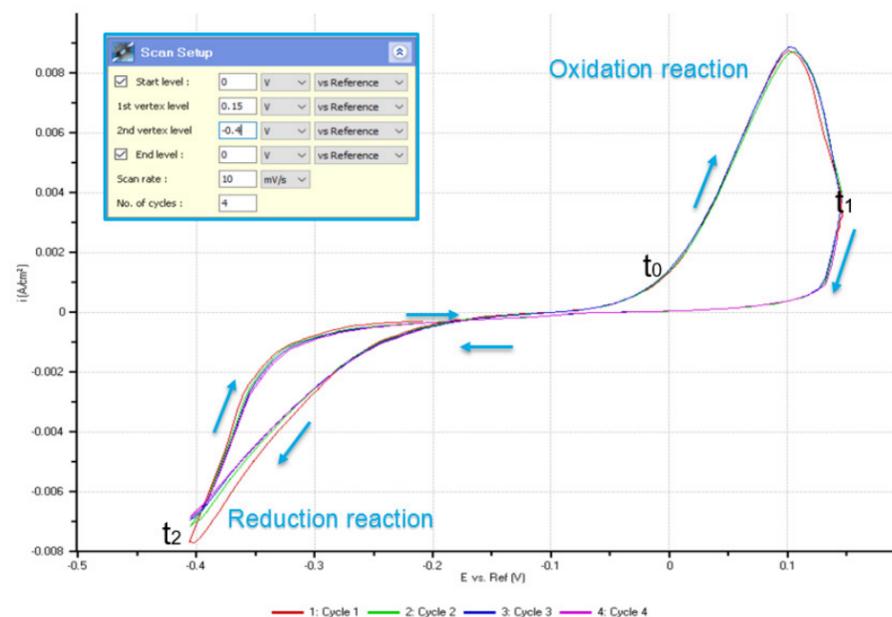


Figure 2. Cyclic voltammograms. The negative peaks demonstrate the reduction reaction state where copper is deposited in the sample. While the positive peaks demonstrate the oxidation reaction state where copper is dissolved in the sample.

reduction peaks of the solution. After determining the threshold voltage where the oxidation and reduction occurs, linear sweep voltammetry was applied to deposit and dissolve copper particles on the gold surface. Two scans were performed with -0.2 V to -0.4 V to cover the entire surface of the gold with copper particles. On the other hand 4 scans were performed with -0.2 V to 0 V to completely dissolve the copper in the solution.

RESULTS AND DISCUSSION

Figure 2 shows the CV curve obtained during cyclic voltammetry. 4 complete cycles of oxidation and reduction process (redox process) was selected in obtaining the curve.

The result implies that these processes are reversible depending on the amount of potential applied on the solution. The deposition of copper begins when -0.2 V was applied on the cell and the highest reduction state takes place when -0.4 V was applied. The result can be interpreted that more negative potentials applied on the cell will increase the magnitude of the copper deposited in gold surface. On the other hand, the dissolution of copper begins when the voltage is 0V and the highest oxidation state takes place in application of 0.1 V. The magnitude of the copper dissolved in the gold surface will increase as more positive potentials applied on the cell. The CV curve also shows that in -0.1 V, the solution is in neutral state wherein no chemical reaction

1ST DEPOSITION TEST

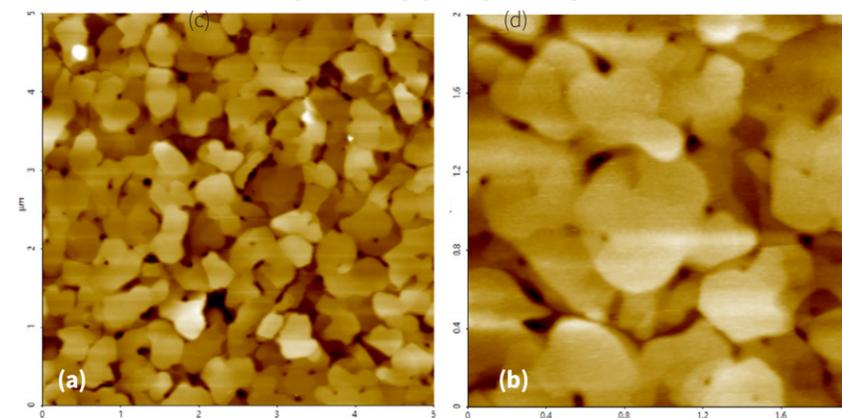


Figure 3 - (a) AFM image acquired in air, (b) AFM image acquired in liquid

Figure 3 (a) and (b) revealed that the 5um by 5um scanned region in ambient air and 1um by 1um scanned region in liquid condition of gold surface is made of individual grains which are believed to be crystallized. The high quality images show that no foreign particles are present on the surface prior introducing electrochemical reaction in the solution.

2ND DEPOSITION TEST

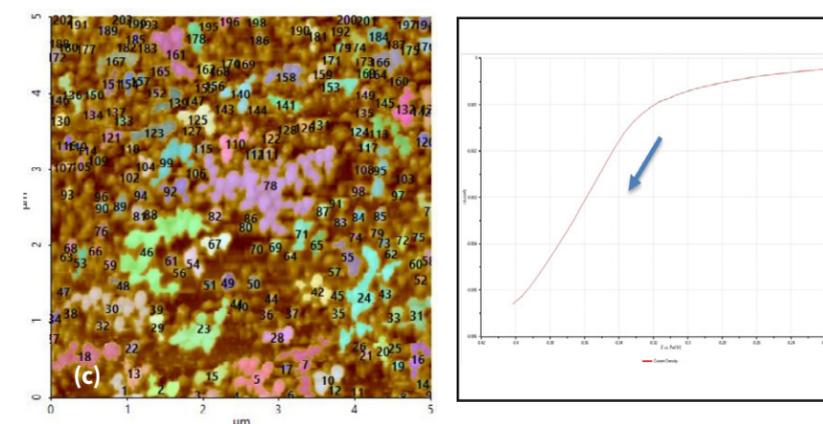
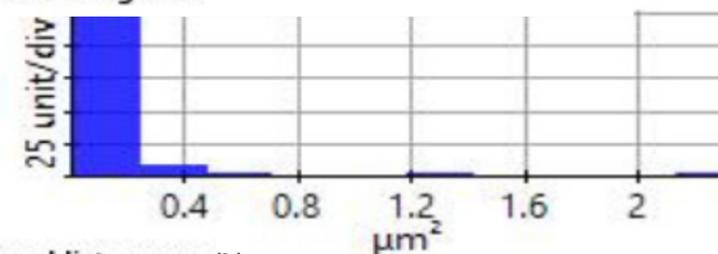


Figure 4 - (a) and (c) AFM images acquired on 1st and 2nd deposition test, (b) and (d) CV curves acquired using linear sweep voltammetry with voltages from -0.2V to -0.4V on 1st and 2nd deposition test.

Area Histogram (a)



Area Histogram (b)

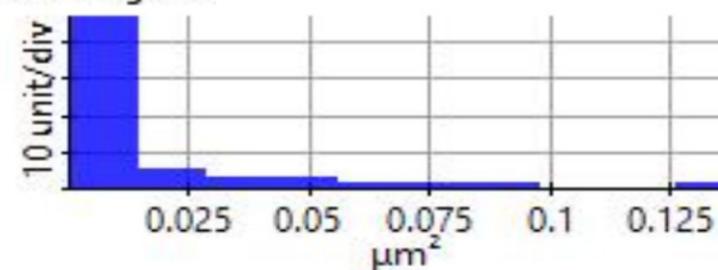


Figure 5 - Histogram plots of particles area distribution on 1st (left) and 2nd (right) deposition test.

The redox process was confirmed using AFM and voltages were applied using linear sweep voltammetry. Figure 4(b) and 4(d) shows the CV curves acquired by applying voltages from -0.2V to -0.4V. The current density decreases as more negative voltages were applied. This phenomenon demonstrates that reduction reaction is occurring. Figure 4(a) and 4(c) confirms this process wherein the images clearly show that copper particles were successfully deposited on the gold surface. Copper particles were quantified using XEI software developed by Park Systems which marked each detected particles with different colors and numbers. The detection method used in XEI is Upper Threshold Grain Detection. In this method a threshold value is set and particles whose heights are smaller than the threshold value are not detected.

In this experiment 5.5 nm was used as a threshold value to quantify the copper particles. Since the gold surface is composed of individual grains with various heights, most of the copper particles deposited on lower surface with height value smaller than the threshold value were not detected. Figure 4a shows the image acquired during the first deposition test. The number of particles detected on this test was approximately 7 with mean area value of 2 nm. In addition, there are particles deposited on lower regions with smaller height value that were not detected by XEI. Furthermore, 199 particles were detected on the 2nd test with mean area value of 36 nm as shown in Figure 4b. The data shows that the number of particles deposited on the 2nd test is 28 times more than the number on the 1st test with larger area which covered almost the entire region of gold surface.

The CV curves acquired by applying reverse voltage from -0.2 V to 0 V during dissolution process show that current density increases as more positive voltages were applied. This phenomenon demonstrates that oxidation reaction is occurring. The AFM images acquired in this process confirm that such phenomenon happened since the number of copper particles deposited on the gold surface decreases when dissolution tests were performed. Figure 6 (a) shows the image acquired in the 1st dissolution test. The number of particles detected on this test was 180 with a mean area value of 37 nm². The number of particles detected on this test was slightly fewer with a smaller mean area value compared to particles detected on the 2nd deposition test. Almost similar results were observed in 2nd dissolution test wherein the number of particles detected was 181 but with a smaller mean area value of 24 nm². On the other hand, the results on the 3rd dissolution test are far from the first two test. The detected number of particles was only 19 with a mean area value of 7 nm². Lastly, the 4th and final dissolution test shows that almost the remaining copper particles on the gold surface including those deposited on the lower region were completely dissolved on the solution.

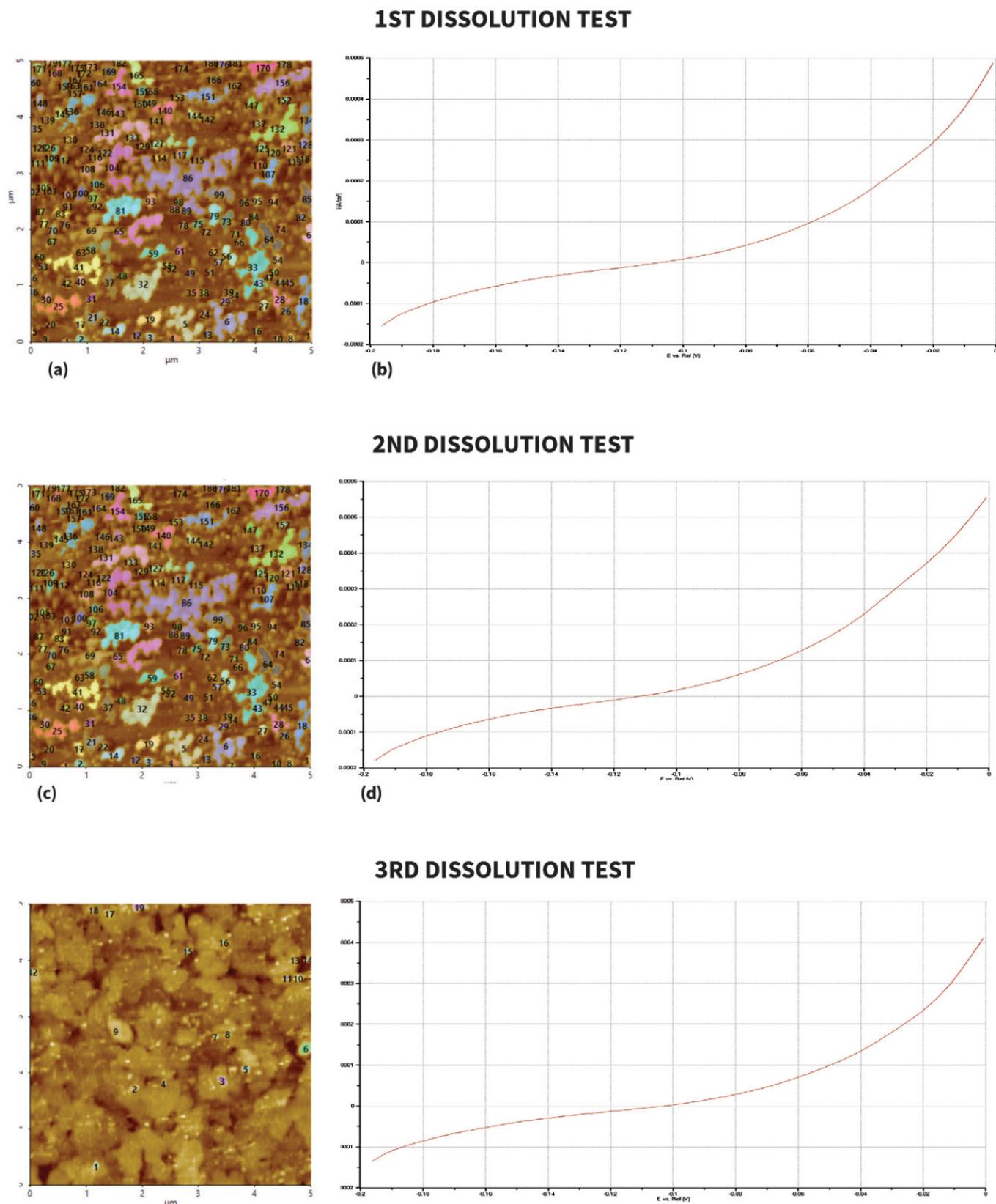


Figure 6 - (a), (c), (f), and (h) AFM images acquired on 1st to 4th dissolution test, (b), (d), (g) and (i) CV curves acquired using linear sweep voltammetry with voltages from -0.2 V to 0 V on 1st to 4th dissolution test.

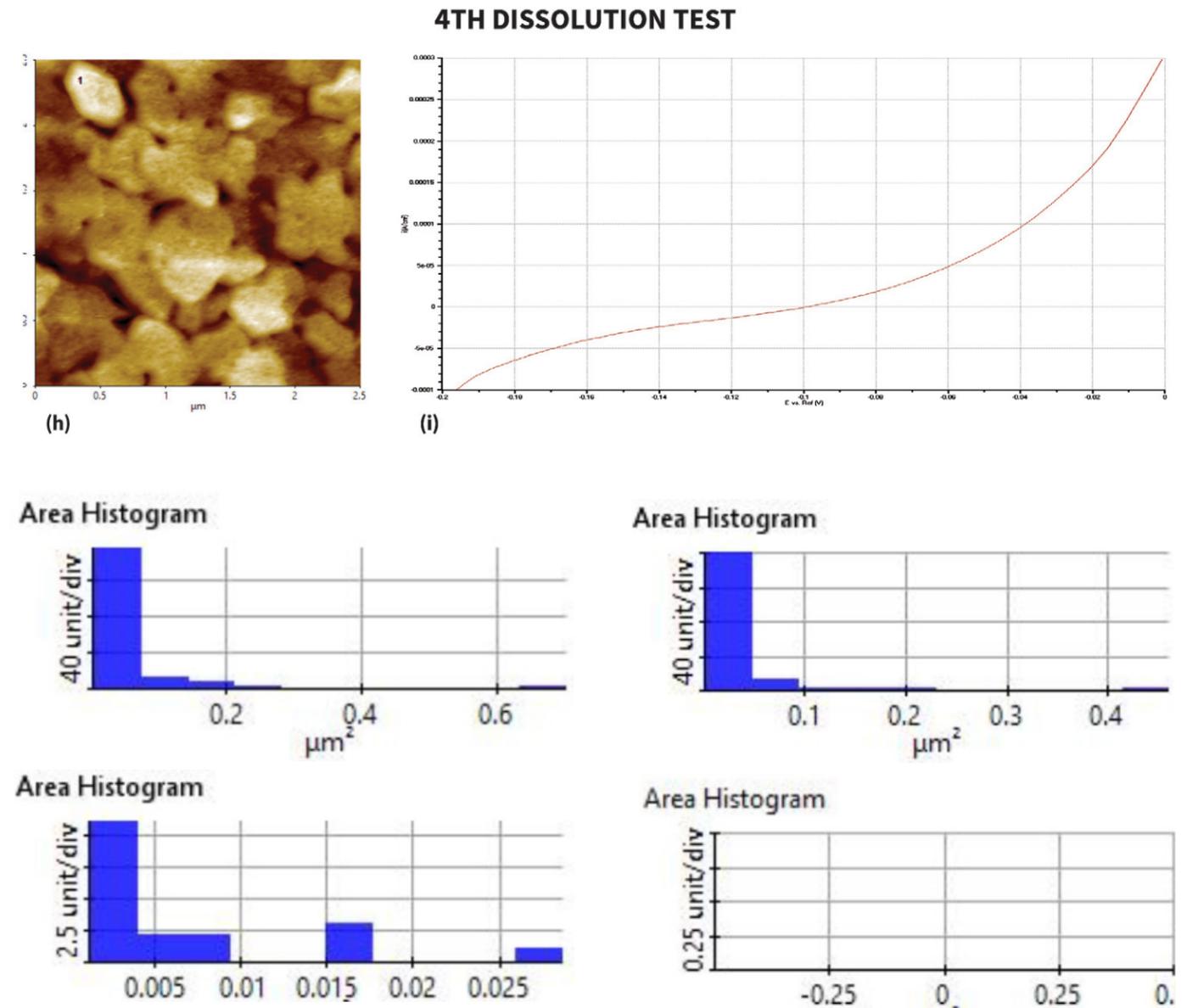


Figure 7. Histogram plots of particles area distribution on 1st (upper-left), 2nd (upper-right), 3rd (bottom left), and 4th (bottom-right) dissolution test.

CONCLUSIONS

Here we demonstrate the use of electrochemical atomic force microscopy in situ monitoring of morphological changes of samples that are undergoing electrochemical process. The deposition and dissolution of the copper particles on the gold surface were successfully performed by applying the suitable voltages suggested by the CV curve acquired during cyclic voltammetry. The data on the deposition process demonstrate that the magnitude of copper deposited on the surface increases tremendously on the 2nd deposition test. On the other hand, the

dissolution data shows that greatest number of copper nanoparticles was dissolved on the 3rd dissolution test. Overall, the technique described in this study will successfully provide researchers nanoscale information that is significant in monitoring an electrochemical process.

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PARK AFM SCHOLARSHIP AWARD OVERWHELMING SUCCESS CONTINUES WITH GLOBAL EXPANSION

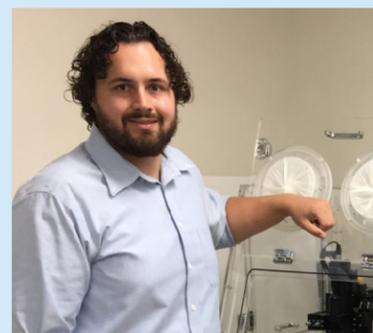
PARK AFM SCHOLARSHIPS HAVE BEEN AWARDED TO TEN OUTSTANDING RESEARCHERS FROM LEADING INSTITUTIONS AND THIS YEAR THE PROGRAM WILL EXPAND GLOBALLY

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 up to 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.

“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 up to 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.

TWO NEW PARK AFM SCHOLARSHIP WINNERS ANNOUNCED



JAMES HEDRICK

GRADUATE STUDENT, MIRKIN LAB,
DEPARTMENT OF CHEMICAL
AND BIOLOGICAL ENGINEERING
NORTHWESTERN UNIVERSITY

Faculty Mentor: Professor Chad Mirkin

James Hedrick received a S.B., in chemical engineering with a minor in biology from the Massachusetts Institute of Technology. His research work is currently focused on the area of nanolithography. He is developing a method for synthesizing nanomaterial libraries over macro length scales on a single chip. After forming these nanocombinatorial libraries, they can be screened for a multitude of properties for a range of potential applications. Concurrently, he has been researching and building a scanning probe-based 3D printer to print multifunctional materials with nanoscale resolution. He is the owner and founder of his company Pintastic Pins since 2007.

Abstract: Hard Transparent Arrays for Polymer Pen Lithography

Patterning nanoscale features across macroscopic areas is challenging due to the vast range of length scales that must be addressed. With polymer pen lithography, arrays of thousands of elastomeric pyramidal pens can be used to write features across centimeter-scales, but deformation of the soft pens limits resolution and minimum feature pitch, especially with polymeric inks. Here, we show that by coating polymer pen arrays with a ~175 nm silica layer, the resulting hard transparent arrays exhibit a force-independent contact area that improves their patterning capability by

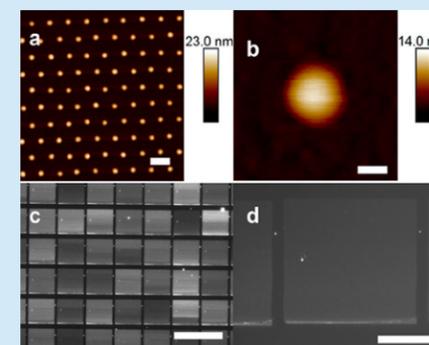
reducing the minimum feature size (~40 nm), minimum feature pitch (<200 nm for polymers), and pen to pen variation. With these new arrays, patterns with as many as 5.9 billion features in a 14.5 cm² area were written using a four hundred thousand pyramid pen array. Furthermore, a new method is demonstrated for patterning macroscopic feature size gradients that vary in feature diameter by a factor of 4. Ultimately, this form of polymer pen lithography allows for patterning with the resolution of dip-pen nanolithography across centimeter scales using simple and inexpensive pen arrays. The high resolution and density afforded by this technique position it as a broad-based discovery tool for the field of nanocombinatorics.

“A CRITICAL PART OF MY RESEARCH IS BEING ABLE TO PATTERN HARD AND SOFT MATERIALS WITH NANO RESOLUTION USING POLYMER PEN LITHOGRAPHY AND DIP-PEN NANOLITHOGRAPHY. BOTH TECHNIQUES ARE POSSIBLE USING THE PARK XE-150. ON A DAILY BASIS, I RELY ON THE PARK XE-150 TO CREATE HIGH-RESOLUTION, LARGE-AREA PATTERNED ARRAYS THAT I CAN THEN USE TO SYNTHESIZE AND SCREEN HIGHLY COMPLEX NANOPARTICLES.”

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

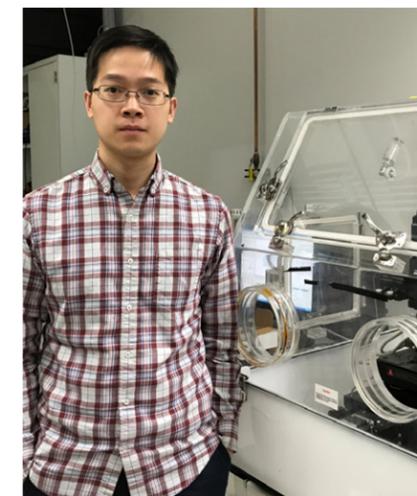
As materials go from the macro-to-micro and then to the nanoscale, their properties will vastly change. At the nanoscale, small changes in size and composition of material have dramatic effects on the chemical activity, energy capture, and mechanical properties.

Polymer pen lithography (PPL) utilizes an array with millions of pyramidal pens to deposit single attoliter features on a surface. When combined with scanning probe block copolymer lithography, which utilizes PEO-b-P2VP and a metallic precursor to direct the on-surface and spatially confined synthesis of metal nanoparticles, combinatorial libraries of billions of nanoparticles can be synthesized in unison with variations in both size and composition. This innovative, high-throughput screening platform enables rapid discovery of next generation catalysts that have the potential of drastically impacting multiple industries.



(a) AFM of a dot array with a 190 nm pitch in a hexagonal pattern array written using hard transparent array, 200 nm scale bar; (b) single polymer feature with a circle of small droplets from original meniscus, 20 nm scale bar; (c) dark-field optical microscopy of array with 500 nm pitch written using hard transparent array with PPL with 14 641 features written per pen, 100 μm scale bar; and (d) zoomed in image pattern made from a single pyramidal pen, 25 μm scale bar.

Published in: James L. Hedrick; Keith A. Brown; Edward J. Kluender; Maria D. Cabezas; Peng-Cheng Chen; Chad A. Mirkin; *ACS Nano* 2016, 10, 3144-3148. DOI: 10.1021/acs.nano.6b00528
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PENGCHENG CHEN

PHD STUDENT AT NORTHWESTERN UNIVERSITY

“Park XE-150 AFM is a very useful and convenient tool to perform scanning probe lithography experiments. The lithography mode in XEP software offers the highest degree of freedom to design specific lithography patterns. The software allows for controlling the movement of XYZ piezos in a precise and programmed way, which is crucial in our study for printing polymer nanoreactors on target substrates.”

Faculty Mentor: Professor Chad Mirkin

Pengcheng Chen received his B.S. and M.S. in Polymer Science from Zhejiang University (ZJU), China. His research work is currently focused on developing combinatorial library of nanoparticles for catalyst screening. This research involves synthesizing nanostructures with scanning probe lithographic method, and is important for use in future applications, like pharmaceutical synthesis and energy conversion.

Abstract: Polyelemental nanoparticle libraries

Multimetallic nanoparticles are useful in many fields, yet there are no effective strategies for synthesizing libraries of such structures, in which architectures can be explored in a systematic and site-specific manner. The absence of these capabilities precludes the possibility of comprehensively exploring such systems. We present systematic studies of individual polyelemental particle systems, in which composition and size can be independently controlled and structure formation (alloy versus phase-separated state) can be understood. We made libraries consisting of every combination of five metallic elements (Au, Ag, Co, Cu, and Ni) through polymer nanoreactor-mediated

synthesis. Important insight into the factors that lead to alloy formation and phase segregation at the nanoscale were obtained, and routes to libraries of nanostructures that cannot be made by conventional methods were developed.

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

“Park XE-150 AFM is a very useful and convenient tool to perform scanning probe lithography experiments. The lithography mode in XEP software offers the highest degree of freedom to design specific lithography patterns. The software allows for controlling the movement of XYZ piezos in a precise and programmed way, which is crucial in our study for printing polymer nanoreactors on target substrates.”

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

Metal nanoparticles are commonly used as catalysts in scientific research and industrial manufacturing processes because they provide chemical transformations with superior reactivity and selectivity. Multimetallic nanoparticle catalysts, however, remain

PARK AFM SCHOLARSHIP PROGRAM ACCEPTING SUBMISSIONS FOR 2018

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>

due to the difficulty in synthesizing them in pure forms while encompasses the enormous compositional and structural parameter space. My research mainly focuses on developing methods for combinatorially synthesizing complex multicomponent nanoparticle systems and exploring its application in high-throughput catalyst screening. This research relies on scanning probe lithography to generate arrays of spatially separated polymer nanoreactors to synthesize individual nanoparticles. It frees the experimentalists from making numerous batches of uniform nanoparticles as each nanoparticle is locally defined by unique synthetic conditions in the polymer nanoreactors. Through parallel screening of the vast number of nanoparticles, novel catalytic nanomaterials can be discovered in a systematic way, avoiding the inefficient process of serially making and characterizing nanoparticles. Overall, my research work will offer the opportunity for rapid identification of next generation multimetallic catalysts, which will increase the sustainability and efficiency of chemical processes that leads to benefits for both academic and industrial communities.

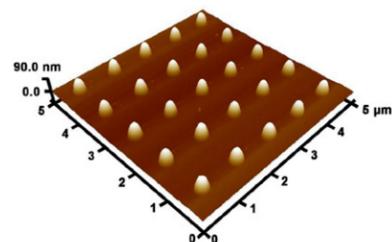


Photo Caption: AFM topographical image of a polymer dot array printed by dip-pen nanolithography

**PARK AFM SCHOLARSHIP WINNERS
RESEARCH AT THE MIRKIN
RESEARCH GROUP UNDER THE
LEADERSHIP OF DR. CHAD MIRKIN
AT NORTHWESTERN UNIVERSITY**

Dr. Chad Mirkin leads the Mirkin Research Group and is the Director of the International Institute for Nanotechnology (IIN), the first institute of its kind in the country established in 2000 and representing \$1 billion in nanotechnology research.

The Mirkin Research Group focuses on developing methods for controlling the architecture of molecules and materials on the 1 – 100 nm length scale, understanding their fundamental properties, and utilizing such structures to develop novel tools that can be applied in the areas of chemical and biological sensing, gene regulation, immunomodulation, lithography, catalysis, optics, and energy generation, storage, and conversion. The Mirkin Research Group has pioneered the use of nanoparticle-biomolecule conjugates as synthons in materials science and the development of many nanoparticle-based extra- and intracellular biodiagnostic and therapeutic tools. Since its inception, more than 1,800 products and systems have been commercialized worldwide. Twenty start-up companies have been launched based upon IIN research, and they have attracted over \$700 million in venture capital funding.



Caption: Dr. Chad Mirkin who leads the Mirkin Research Group at Northwestern University is the Faculty Mentor for Park Systems AFM Scholars James Hedrick and Pengcheng Chen and the Director of the International Institute for Nanotechnology (IIN) at Northwestern University.

**“LIKE THE SCIENTIFIC AND
ENGINEERING BREAKTHROUGHS
OF THE EARLY 20TH CENTURY,
NANOTECHNOLOGY WILL
BRING DRAMATIC AND
POSITIVE IMPROVEMENTS
TO THE EVERYDAY LIVES OF
PEOPLE THROUGHOUT THE
WORLD AND HOLDS THE
PROMISE TO SOLVE SOME OF
THE WORLD’S MOST PRESSING
PROBLEMS IN AREAS AS DIVERSE
AS MEDICINE, INFORMATION
TECHNOLOGY, ENERGY, AND
HOMELAND SECURITY.”**

**RYAN FELLOWS AT NORTHWESTERN
UNIVERSITY GIVE BACK BY
INTRODUCING NANOTECHNOLOGY
TOPICS TO FUTURE SCIENTISTS**

Park AFM Scholars James Hedrick and Pengcheng Chen are both Ryan Fellows at Northwestern University, where educational outreach is an important part of the fellowship experience. Ryan Fellows engage in a variety of volunteer initiatives in the community including STEM & Sports Day supported by the National Informal STEM Education Network giving "What is Nano?" presentations to local school children and "Zoom into Nano" events at the Museum of Science & Industry, Chicago (pictured left).



2018 DPG Spring Meeting

Please visit Park Systems at booth #5 at the DPG Spring Meeting 2018, organized by Deutsche Physikalische Gesellschaft (DPG) – the world’s largest physical society.

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Berlin, 11-16 March 2018**



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