NANDSCIENTIFIC SUMMER 2016

STUDYING POST-ETCHING SILICON CRYSTAL DEFECTS ON 300 MM WAFER

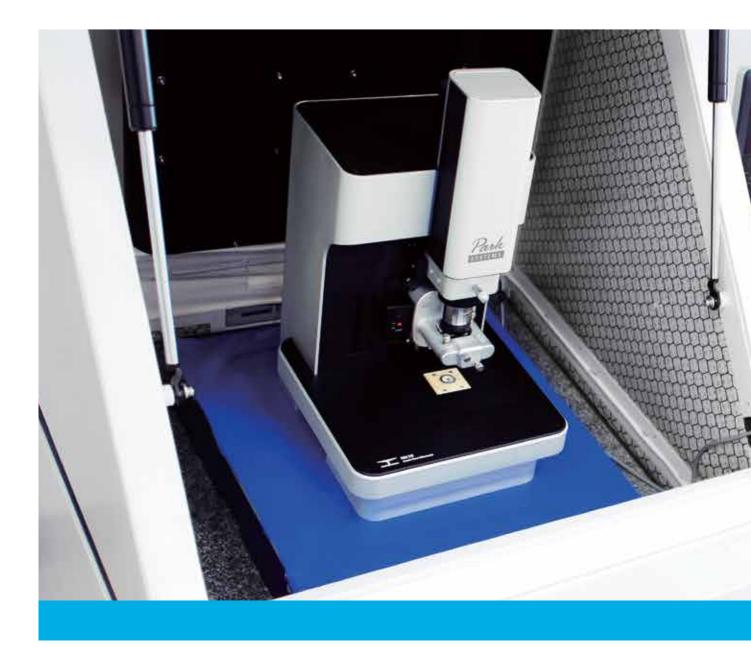
BY AUTOMATIC DEFECT REVIEW AFM

NANO ACADEMY A MICROSCOPIC VIEW FROM NANOSCIENTISTS

GROWTH AND SURFACE ENGINEERING OF NANOWIRES FOR OPTOELECTRONIC APPLICATIONS NANOWIRES

COMMERCIALIZING NEWEST NERVE REGENERATION PROTOCOL AND

PROTOCOL AND ADVANCEMENTS IN PEDIATRIC AND CARDIOVASCULAR TREATMENTS



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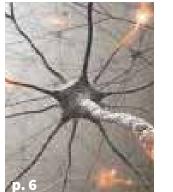
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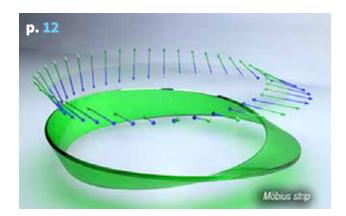
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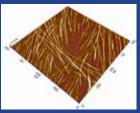
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Cover image:

Cellulose nanowhiskers are plants-borne biopolymer nanomaterials that are light, strong and easy to synthesize in volume, attracting much attention in recent years for their as novel nanocomposite materials. This image was taken by Park NX10 AFM system in 3D perspective of the Cellulose nanowhisker rods topography.

MESSAGE FROM EDITOR



Keibock Lee, Fditor-in-Chiel

OUR QUANTUM SURROUNDINGS: NOW VISIBLE USING ADVANCED MICROSCOPY

We can discover everyday quantum physics by investigating nature using today's advanced nanoscale microscopy tools. Examples in nature show us that biology does indeed function by using quantum physics and that something we call quantum coherence commonly occurs.

With the advent of microscopy methods that examine 3D cellular structures, scientists are learning more about the natural elements than ever before. The result is new products for advanced electronics, medical breakthroughs and ever advancing energy applications that are both sustainable and cost effective.

Coherent light harvesting in photosynthesis is a prime example of quantum biology in nature, where the energy from sunlight gets transferred through chlorophyll molecules to the photosynthetic reaction centers within picoseconds. With an efficiency rate of 100%, plants harvest light energy by converting the chlorophyll molecule into chemical energy, then virtually acting as nature's own solar cells. Labs all over the world are working on building chemical solar cell prototypes that are modeled upon this natural photosynthesis.

Another example of nature's unbelievable molecular genius is the ability of one protein molecule in a bird's eye to "see" the earth's magnetic grid to use for migration. Studied extensively by scientists, this is now considered a quantum mechanical process, whereby the protein molecule in the bird's eye sends signals to the brain that are sensitively dependent on the angle of change in magnetic field inclination, thereby allowing the bird to map routes.

We have found some wonderful examples of molecular and electrochemical reactions to highlight in this issue that detail the way quantum physics and quantum mechanics are advancing scientific research. And all this is possible thru the advent of nanoscale molecular microscopy.

In this issue, we explore light harvesting in an exciting interview with the Max Planck Institute's Science of Light scientist, Dr. Muhammad Y. Bashouti who reveals new discoveries in the field of photonics. The growth opportunities for optoelectronics for the fields of energy and medicine are exciting and discussed in depth in this feature interview.

We also investigate a new nerve regeneration protocol now being commercialized by Dr. Jayakumar Rajadas, Director of the Biomaterial and Advanced Drug Delivery Lab at Stanford School of Medicine that has many exciting implications for medical breakthroughs.

You will find a series of stories showcasing nanoscale AFM images, nanoscience webinars and pioneering technically advanced AFM methods that help scientists see with new accuracy the molecular cell reactions and apply it to advances beyond our imaginations. Imagination is limitless and continues to expand science as we move into a new era in nanotechnology, there is so much more yet to come.

We are excited to continue to bring you great stories and technical articles about our quantum world. Please share your story ideas and submit them to us. We are very interested in hearing from readers who know about new applications in NanoScientific research.



Quantum mechanics explains the 100% efficiency of the light harvesting method in nature called photosynthesis where plants create natural solar cells by converting light to chemical energy.



European Robins navigate using magetoreception. A new theory of quantum entanglement explains bird migration by a single molecule in their eye that sends signals to the brain that allows them to "see" the earth's magnetic grid.



"SCIENTIFIC DISCOVERY AND SCIENTIFIC Knowledge have been achieved only by Those who have gone in pursuit of it Without any practical purpose Whatsoever in view."

MAX PLANCK

FEATURE INTERVIEW

COMMERCIALIZING NEWEST NERVE REGENERATION PROTOCOL AND ADVANCEMENTS IN PEDIATRIC AND CARDIOVASCULAR TREATMENTS AT STANFORD UNIVERSITY SCHOOL OF MEDICINE



"AFM IS AN IMPORTANT TOOL FOR US TO USE IN NANO PATTERNING. WE USE PARK NX10 ATOMIC FORCE MICROSCOPE TO HELP US UNDERSTAND MOLECULE INTERACTIONS AND IDENTIFY THE NANO STRUCTURES OF PROTEINS."

DR. JAYAKUMAR RAJADAS, PHD, BIOMATERIALS AND ADVANCED DRUG DELIVERY RESEARCH

nano particles delivered in the blood and body fluids. My research is also involved in further the knowledge of genomic and proteomic changes due to the proteotoxic stress in immune cells, cardiomyocytes and neurons."

Dr. Jayakumar's extensive work with micro needles has contributed to a new drug delivery method, using micrometer-scale needles that dissolve in the skin that can eliminate the pain and dangers of hypodermic needle use and enhance delivery of the drug across the skin. Transdermal micro needles create micron sized pores in the skin, ideal

A Profile of Dr. Jayakumar Rajadas, PhD, Biomaterials and Advanced Drug Delivery Research

Dr. Rajadas is the founding director of the **Biomaterials and Advanced Drug Delivery** Laboratory at Stanford University. This center has been involved in transforming biophysical ideas into biomaterial and drug delivery technologies. These technologies include microencapsulation of drugs, vascular grafts, bio-implants, development of small molecule and protein-based drugs, regeneration of nerve and cardiovascular tissues, and wound healing applications. He is also a Lecturer in the Department of **Neurology and Neurological Sciences. His** research has involved transforming nano science ideas into biomaterial and drug delivery technologies. Before moving to Stanford, he served as the founding chair of the Bio-organic and Neurochemistry Division at one of India's national laboratories. He is a recipient of several awards including Young scientist award in chemistry for the year 1996 from the Government of India. He has also won the best scientist award from the Tamilnadu State Government India in the year 1999. He is also co-recipient of nine SPARK translational awards in Stanford University. He has published over 150 papers with numerous patent disclosures. He received his MS in Chemistry at the University of Madras and his Ph. D in **Biophysical Chemistry at the Indian Institute** of Technology.

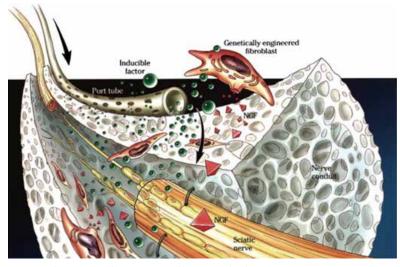
Before moving to Stanford, he served as the founding chair of the Bio-organic and Neurochemistry Division at one of India's national laboratories, Central Leather Research Institute Chennai, India, where he was responsible for both the organization and management of the division.

His backgrounds in biochemical pharmacology and physical-chemistry have allowed him to effectively translate physical concepts into novel formulation technology.

"Pre-Designed nanosomes interact with biofluids such as blood and biological secretions to develop into structures of desired patterns. We have shown these soft particles can recognize endothelial linings of the blood capillaries that are proximal to the affected tissues in the heart, brain and liver. We have used these structures to stabilize and target the fragile peptides, proteins and water insoluble therapeutic molecules to different organs."

"My research foresees the application of various technologies in a research domain for developing novel formulation and therapeutics that would direct towards invention and targeted drug delivery systems. I have used biophysical and pharmacology approaches for the identification of the most optimal micro environments to implant the stem cells to repair injured skin, heart and brain. My expertise in stem cell survival and differentiation is currently utilized in developing technology for high yield survival of purified cardiovascular cells in the transplantation and imaging studies. I have also developed novel drug targeting approach using protein corona formation on the soft





Nerve conduits using techniques inspired by nanotechnology

for patients who can administer the drug by themselves . This method of drug delivery does not reach nerve fibers and blood vessels in the dermis and the drug gets directly into systemic circulation and is ideal for drugs with poor absorption rate.

Nerve Regeneration Protocol Under License

Nerve tissue engineering is a rapidly evolving and expanding field in the realm of biomedicine where newest methods can create, repair, replace cells, tissues and organs by using cell or combinations of cells with biomaterials and biologically active molecules which helps to produce materials which very much resembles to body's native tissue/tissues. New materials used for manufacturing nerve conduits offer such exciting results that the nerve regeneration company AVIVE is already onboard to license the new technology coming from Stanford School of Medicine.

Dr. Rajadas and the BioADD have recently licensed their formulation to deliver Bestatin for pulmonary hypertension to Eiger, a clinical –stage biopharmaceutical company. Developed in collaboration with Dr. Mark Nicolls, Professor of Medicine Pulmonary and Critical Care at Stanford, a lab that focuses primarily on the contribution of the immune response to lung disease, this product is in phase II clinical trials. At Dr. Nicolls lab, they are specifically examining the contribution of inflammation to the development of pulmonary hypertension. LaJolla Pharmaceutical has licensed a liver targeting nano formulation for the treatment of pediatric jaundice and adult liver fibrosis, developed in collaboration with Dr. David Stevenson, who was recently honored with the 2016 Joseph W. St. Geme Jr. Leadership Award by The Federation of Pediatric Organizations for his dedicated career to improving the lives of the smallest and sickest babies. Part of Dr. Stevenson's research is focused on the study of the ontogeny and control of heme catabolism and bilirubin production in the developing neonate.

Neonatal jaundice occurs when bilirubin, which is produced when the body disposes of excess red blood cells, builds up too quickly for disposal by the infant's immature liver and affects more than half the newborn babies in this country, according to the American Academy of Pediatrics. In most instances the condition resolves itself without treatment. But in rare cases the bilirubin levels are high enough to enter the brain and interfere with nerve cell signaling. If left untreated, the infant may die from prolonged exposure to the elevated levels.

At the Biomaterials and Advanced Drug Delivery laboratory at Stanford, they have developed a Nano-carrier combination platform, consisting of lipids and serum proteins, to develop novel structures that can recognize impaired heart and liver tissues. For the first time, they have used this technology to stabilize fragile peptides, proteins and water-



David K. Stevenson, MD, renowned neonatology leader at Lucile Packard Children's Hospital Stanford and Stanford Children's Health, is seen at work in our neonatal intensive care unit. He has been named recipient of the 2016 Joseph W. St. Geme Jr. Leadership Award by The Federation of Pediatric Organizations. Stevenson will be presented the award on April 30 at the Pediatric Academic Societies Meeting in Baltimore. (Photo: Business Wire) Stanford neonatology pioneer Dr. David Stevenson has been named recipient of the 2016 Joseph W. St. Geme Jr. Leadership Award by The Federation of Pediatric Organizations.

insoluble therapeutic molecules to target the delivery of these molecules to different organs.

Jayakumar Rajadas and his researchers have also discovered drugs with the potential to eliminate the Lyme disease-causing bacteria Borrelia burgdorferi at the onset of infection. He has developed novel therapeutic molecules for the treatment of Lyme disease and identified a upregulated stress protein as a bio marker in the Post treatment Lyme syndrome. He uses various biophysical approaches such as AFM, fluorescence, and NMR to understand the structural details of neurotoxic oligomeric forms of misfolded proteins involved in neurodegenerative diseases.

For the past eight years, his lab has also been involved in transforming biophysical ideas into biomaterial and drug delivery technologies. These technologies include microencapsulation of drugs, vascular grafts, bio-implants, development of small molecule and protein-based drugs, regeneration of nerve and cardiovascular tissues, and wound healing applications. He has published over 155 papers in peer-reviewed journal articles including publications in Nature Medicine, Science Translational Medicine, Molecular Cell, Journal of Clinical Investigation, and the Proceedings of the National Academy of Sciences (USA). Dr. Rajadas has numerous patent disclosures and seven products he has developed were licensed to commercial firms.

ATOMIC FORCE MICROSCOPE (AFM) TECHNICAL ARTICLE

By Ardavan Zandiatashbar, Patrick A. Taylor, Byong Kim, Young-kook Yoo, Keibock Lee, Ahjin Joc, Ju Suk Lee, Sang-Joon Cho, and Sang-il Park

ABSTRACT

Single crystal silicon wafers are the fundamental elements of semiconductor manufacturing industry. The wafers produced by Czochralski (CZ) process are very high quality single crystalline materials with known defects that are formed during the crystal growth or modified by further processing. While defects can be unfavorable for yield for some manufactured electrical devices, a group of defects like oxide precipitates can have both positive and negative impacts on the final device. The spatial distribution of these defects may be found by scattering techniques. However, due to limitations of scattering (i.e. light wavelength), many crystal defects are either poorly classified or not detected. Therefore a high throughput and accurate characterization of their shape and dimension is essential for reviewing the defects and proper classification. While scanning electron microscopy (SEM) can provide high resolution two-dimensional images, atomic force microscopy (AFM) is essential for obtaining three-dimensional information of the defects of interest (DOI) as it is known to provide the highest vertical resolution among all techniques [1]. However AFM's low throughput, limited tip life, and laborious efforts for locating the DOI have been the limitations of this technique for defect review for 300 mm wafers. To address these limitations of AFM, automatic defect review AFM has been introduced recently [2], and is utilized in this work for studying DOI on 300 mm silicon wafer. In this work, we carefully etched a 300

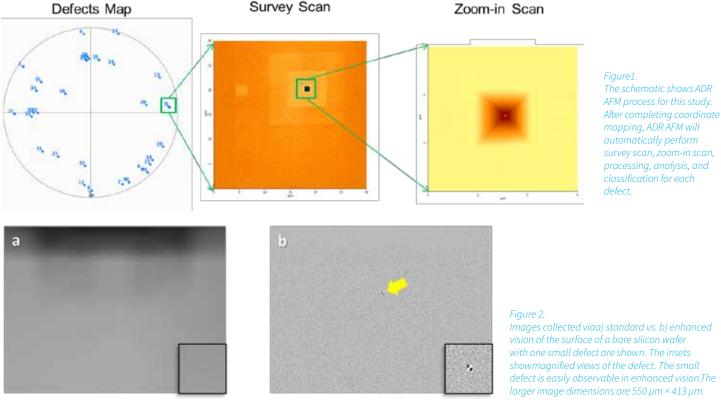
mm silicon wafer with a gaseous acid in a reducing atmosphere at a temperature and for a sufficient duration to decorate and grow the crystal defects to a size capable of being detected as light scattering defects[3]. The etched defects form a shallow structure and their distribution and relative size are inspected by laser light scattering (LLS). However, several groups of defects couldn't be properly sized by the LLS due to the very shallow depth and low light scattering. Likewise, SEM cannot be used effectively for post-inspection defect review and classification of these very shallow types of defects. To verify and obtain accurate shape and three-dimensional information of those defects, automatic defect review AFM (ADR AFM) is utilized for accurate locating and imaging of DOI. In ADR AFM, non-contact mode imaging is used for non-destructive characterization and preserving tip sharpness for data repeatability and reproducibility. Locating DOI and imaging are performed automatically with a throughput of many defects per hour. Topography images of DOI has been collected and compared with SEM images. The ADR AFM has been shown as a non-destructive metrology tool for defect review and obtaining three-dimensional topography information.

MOTIVATION

As integrated devices continue to shrink, incoming bare silicon wafer defectivity requirements become more and more stringent. The inspection of bare silicon wafers for surface defects is predominantly accomplished by measuring the difference in laser light scattering (LLS) between the clean surface and a surface defect, where the intensity of the scattered signal is compared to the LLS of a standard latex sphere. The actual surface defectivity can originate from added particles, topological defects, and crystal imperfections. To be able to reduce the number of defects one must know the source of the defect. LLS inspection can only give defectivity counts and a relative size. Therefore, one must rely on defect review techniques such as SEM and AFM to determine the nature and origin of the defects.

SEM provides two-dimensional aerial images of the defects which lacks the information about depth or height of the defects. On the other hand, AFM can provide threedimensional topography images of the defects with the highest vertical resolution among all techniques[1]. The shortcomings of conventional AFM systems were low throughput, limited tip life, and arduous efforts for locating the DOI on the 300 mm wafers. To address the limitations of conventional of AFM systems for defect review, ADR AFM has been introduced for 300 mm wafers recently[2]. We used ADR AFM in this study for studying the defects found by LLS inspection tool.

In this study we focus on very small crystal imperfections which are not easily observed by LLS without some means to make them larger. We have used a decorative etching technique to highlight crystal imperfections to be studied



by LLS, SEM, and AFM. The defect analysis can only be accomplished with accurate and reproducible defect coordinate transfer between analysis tools. Here we show how we have successfully and reliably found and characterized the decorated defects by ADR AFM.

ADR AFM PROCEDURE

The process in ADR AFM is depicted in figure 1. During this process, the defects of interest are located accurately and imaged nondestructively. Two factors are essential in order to achieve these objectives. First proper linkage between ADR AFM and LLS inspection tool is required to minimize the positioning errors and locate the defects accurately. The linkage for blank wafers is achieved by sample coordinate alignment. Generally there are no alignment markers or fiducials available on blank wafers to be used for alignment. Therefore ADR AFM uses specialized vision to perform the sample alignment properly. Another important factor in AFM defect review is non-contact mode imaging which is required for non-destructive imaging of the samples while preserving AFM tip life such that the tip can last throughout the process for multipledefects.

Coordinate alignment

Sample coordinate alignment is needed for proper linkage between the stage coordinates of ADR AFM and LLS inspection tool. In the case of blank wafers, no fiducial or alignment

marker exists on the sample to be used for sample alignment. To overcome this challenge, a coarse alignment followed by a fine alignment is performed. In the coarse alignment, three randomly selected peripheral and the notch or an angular reference are selected to correct for translational and rotational errors. This is followed by a fine alignment to eliminate positioning errors due to non-affinity between the stage coordinates of ADR AFM and LLS inspection tool. A few large defects with known inspection coordinates are used for performing fine alignment. Since the defects are hardly visible in a standard AFM optical image, an enhanced vision is used to locate the defects in the optics of the ADR AFM and utilize the defects as aligner markers. Upon the sample alignment, ADR AFM is able to locateadditional defects accurately. More details on coordinate alignment can be found in ref [2].

Enhanced vision

Enhanced vision is utilized during fine coordinate alignment to locate the defects in the optical vision of ADR AFM. The technique is developed based on well-known differential frame averaging of the optical frames collected from the sample surface at two accurately separated locations. The sample can be moved accurately since ADR AFM uses a separated Z and XY scanners configuration. This architecture was initially developed to eliminate the crosstalk between the XY and 7

Images collected viaa) standard vs. b) enhanced vision of the surface of a bare silicon wafer with one small defect are shown. The insets showmagnified views of the defect. The small defect is easily observable in enhanced vision. The larger image dimensions are 550 μm × 413 μm.

scanners (which has been a common artifact in tube scanner based AFM systems)[2]. In this setup, sample is moved by XY scanner while tip is following the sample topography by Z scanner. In enhanced vision, the optical frames of the sample are collected at two precisely separated locations, and then the final frame is generated from the difference between the collected frames. The resulting frame possesses an enhanced contrast of surface details which are not easily observable in the standard vision of ADR AFM. A comparison between the frames collected by standard vision versus enhanced vision is depicted in figure 2.

Non-contact mode imaging

Non-contact mode is the standard imaging mode in ADR AFM. It is essential to maintain tip sharpness during the defect review process from the first to the last defect that is located and imaged. In addition to keeping tip costs low, well-maintained tip sharpness ensures consistent image quality and accuracy between the images of all defects during the process. It therefore enables the automated system to uninterruptedly locate and image the defects with a high throughput. In order to perform non-contact mode imaging, the AFM cantilever is oscillated at its resonance frequency. The oscillating cantilever is brought close enough to the sample that the oscillation amplitude reduces to a pre-defined setpoint due to the van der Waals tip sample interaction. ADR AFM maintains the oscillation amplitude

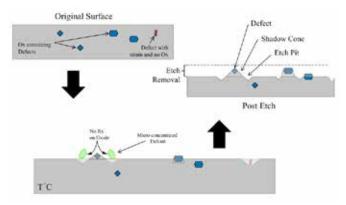


Figure 3. Schematic of the process used to decorate crystal imperfections for defect inspection.

to avoid tip contacting the sample. As the tip scans the sample surface, the oscillation amplitude is maintained bymoving the cantilever up and down with the Z scanner to maintain its tip sample interaction in attractive regime. More details on noncontact mode imaging can be found in reference[4]. Although ADR AFM's functionality is based on non-contact mode imaging, it is capable of performing in other dynamic or contact imaging modes if needed.

Automatic defect search and imaging

The significant improvements in throughput of defect review are obtained by ADR AFM due to its fullyautomated process. Once defect coordinates from LLS inspection tool are entered into ADR AFM, coordinate alignment is performed, the defect is locatedand imaging starts for the list of selected defects. The process of locating and imaging the defects is fully automated. The automation includes locating the defect, tip-sample engagement, non-contact mode parameter optimization, survey scan, optimizing the scan size, final scan, processing, and defect classification. Defects can be classified into two groups of bumps and pits. Defects are typically located within $\pm 10 \ \mu m$ of their LLS coordinates.

SAMPLE PREPARATION (ETCHING PROCESS)

Bare 300mm diameter CZ silicon wafers were treated with a gaseous acid in a reducing atmosphere at a temperature and for a sufficient duration to grow the crystal imperfections [3]. The size and shape of the decorated defects depends on the nature of the original defect as shown in figure 3. Once decorated, thedefect size is capable of being detected as LLS event. TheLLS inspection tool locatesand sizes the LLS events, providing the coordinates to be used by the SEM and AFM.

RESULTS

A wafer containing surface decorated defects was inspected by a LLS tool and 34 defects were selected to be reviewed by ADR AFM. The coordinates of the defects were entered to ADR AFM, coordinate alignment performed, and the defects were located and imaged by ADR AFM. The first 21 defects had been imaged by SEM before being studied by ADR AFM. However, SEM images only provide aerial two-dimensional view of defects without sufficient information on the defects depth and out of plane dimensions. The remaining 13 defects were not found by SEM despite the signal collected by the LLS tool. The summarized results of decorated defect study with ADR AFM and comparison with SEM results aredemonstrated in figure 4. ADR AFM was able to find all the 34 defects including those that had not been found by SEM.

| • | SEM | ATM. | | SEM | AFM | | SEM | AFM | • | SEM | AFM | | -SEM | AFM |
|---|-----|------|----|-------|-----|----|-----|-----|----|-----|---------------|----|------|-----|
| 1 | - | 0 | 8 | | ۵ | 15 | | 0 | 22 | N/A | 0 | 29 | N/A | |
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| 3 | | 0 | 10 | - | | 17 | | 0 | 24 | N/A | | 31 | N/A | |
| 4 | | 0 | 11 | | C | 18 | E | 10 | 25 | N/A | | 32 | N/A | 10 |
| 3 | 0 | 0 | 12 | Z | | 19 | | 0 | 26 | N/A | 0 | 33 | N/A | a. |
| | | 0 | 11 | E | 6 | 20 | | 0 | 27 | N/A | in the second | 34 | N/A | 0 |
| 7 | | | 14 | | D | 21 | 12 | 131 | 28 | N/A | 0 | | | |

Figure 4. The results of defect review with ADR AFM and comparison with SEM are shown. ADR AFM was able to locate and image all the 34 defects. Defects 22 to 34 were not found in SEM. Wafer notch is up in AFM and down in SEM. AFM and SEM images are 180 degree rotated with respect to each other.

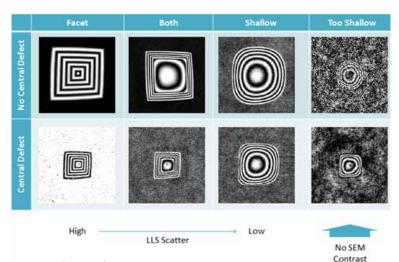


Figure 5. Defect classification based on the LLS, SEM and AFMdata.

The defects selected to be reviewed by ADR AFM belong to eight types according to their LLS signal. The tentative classification by the LLS tool is based on the defect's light scattering which is dependent on morphology, depth, and presence of a central defect. As the decorative etching process proceeds, crystal imperfections are exposed and etch at a different rate than the perfect crystal surface. Defects exposed at the initial stages of the etch are deeper and more developed than defects exposed late in the etching proccess. Defects with an inverted pyramid shape are generally deeper and posses higher LLS signal. They are classified as "Facet". Defects with curved shape formed during the late stages of etching are shallower. These defects are classified as "Shallow". Some defects are

exposed at an intermediate point in the decorative etch and have some degree of faceted walls with curved bottom. This category is classfied as "Both". Defects which have only started to be decorated have a very weak LLS signal and are classified as "Too shallow". The defects are also categorized whether or not they have the center defect, hence, a total of eight defect types were identified. The defect classification is tabulated in figure 5. As we go from left to right side of the table in figure 5, the LLS signal become weaker. This was

attributed to the depth of defects and the sharpness of the defect's edges. AFM images confirmed the depth difference between different classes of defects. Since the AFM images contain Z heights, we were able to use a banded color scale to depict the surface topography of the defects more accurately in 2D view.

DISCUSSION

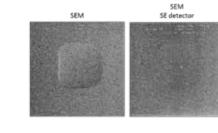
Figure 6 depicts a comparison between the data collected with SEM vs. AFM for the same defect. Primary SEM image provides an aerial 2D view of the defect. However, the shallow depth of the defect reaches the limitations of SEM, hence, poor contrast in the image. As indicated in figure 5, shallower defects were not found by SEM. A secondary electron image helps identify the center defect. Identification of center defect by secondary electron is possible only if the defect was found in primary SEM image.

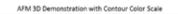
On the other hand, AFM image not only provides an aerial view of the defect, it also contains the height/ depth values for each pixel. Therefore, more information can be obtained about the true topology of the defect by using a 3D representation of the AFM image or using a contoured color scale. Contoured color scales can also help understanding the topology of the defect in aerial view as shown in figure 5. As indicated before, AFM has the highest vertical resolution among all imaging techniques [1], hence, better contrast of AFM images in aerial view.

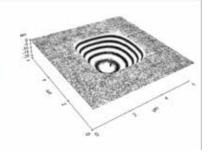
All of the 34 defects were found by ADR AFM including the 13 defects that were not found by SEM. Figure 7 depicts the AFM images a defect that was not found by SEM. The defect depth is below 4 nm and contains a center defect. This example indicates once again the limitation of SEM resolution in out of plane direction. It was indicated above that ADR AFM is a non-destructive imaging technique. It utilizes noncontact mode imaging for survey scan and final imaging scan. However, SEM beam can still modify the sample surface. Figure 8 indicates the sample contamination as a result of electron beam "burning" the surface during SEM imaging. These SEM burn-mark sizes are related to the SEM magnification. Figure 8 shows that several SEM magnifications were used in analyzing this defect.

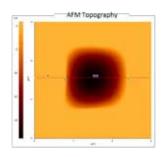
SUMMARY

We have demonstrated the power of









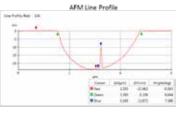
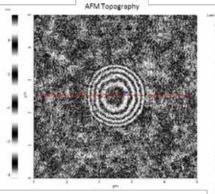


Figure 6. Comparison between the data collected with SEM versus AFM. SEM image provides an aerial 2D view of the defect. Secondary electron image indicates the presence of center defect. AFM image, in addition to aerial 2D view, includes the 3D data. Therefore a line profile, 3D demonstration, and contoured color scale can be utilized to obtain



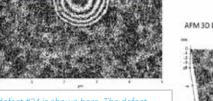


Figure 7. AFM data for defect #24 is shown here. The defect was not found by SEM due to its shallow depth.

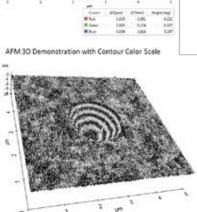
the ADR AFM to provide quality 3D information for defect review on bare silicon wafers. Crystal defects on surface of a 300 mm wafer are highlighted using a decorative etching technique. The surface defects are located by LLS inspection. Select defects of various classes are studies by SEM and ADR AFM. While shallow defects are not found by SEM, ADR AFM successfully found all the defects and provided high resolution three-dimensional topographical information of the defects. With the automated ADR AFM this type of analysis is simple and yet powerful.

REFERENCES

[1] G. T. Smith, Industrial Metrology: Surfaces and Roundness.: Springer, 2002.

[2] Ardavan Zandiatashbar et al., "High-throughput automatic defect review for 300mm blank wafers with atomic force microscope," in Proc. SPIE 9424, Metrology, Inspection, and Process Control for Microlithography XXIX, 2015, p. 94241X. [3] J. Libert and L. Fei, Method to Delineate Crystal Related Defects.: PCT Publication, WO2013055368(A1).

[4] Ardavan Zandiatashbar, "Sub-angstrom roughness repeatability with tip-to-tip correlation," NanoScientific, no. Winter, pp. 14-16, 2014.



AFM Line Profile



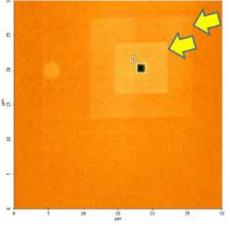


Figure 8. AFM image of a Facet defect with several SEM burn-marks is shown. The SEM burn-marks are marked by arrows.





GROWTH AND SURFACE ENGINEERING OF NANOWIRES FOR OPTOELECTRONIC APPLICATIONS NANOWIRES (NWS)

ARE A PROMISING CANDIDATE FOR THE REALIZATION OF HIGHLY INTEGRATED ELECTRONIC, PHOTONIC AND OPTOELECTRONIC DEVICES AS WELL AS FOR FUNDAMENTAL STUDIES IN NATURAL SCIENCES.

Interview with Dr. Muhammad Y. Bashouti, Physics Department, Max -Planck Institute for the Science of Light Photonic Nanostructures Group

What is the science of light and what can we learn about it to apply to new technologies?

The science of light at our group at Max Planck at Erlangen focuses on research fields that are particularly innovative or especially demanding in terms of funding or time requirements. In particular; our interest is somewhere between basic research and technology development with a focus on the photonic nanostructures for various applications in the areas of opto-, nano- and large area electronics, optical and electrical sensing as well as nano-photonics. This implies the synthesis, building and structuring of novel nano- and nanocomposite materials together with their characterization and modeling. This work covers a wealth of different scientific disciplines ranging from condensed matter physics, semiconductor and theoretical physics over physical and organic chemistry to engineering disciplines like electrical and mechanical engineering as well as material science. It is important to mention that that group is leaded by Prof. Dr. Silke Christiansen who is the Director of the Institute of Nano-architectures for Energy Conversion at the Helmholtz-Center for Materials and Energy at Berlin as well.

What is photonic nanomaterials and how is it useful in society?

Photonics nanomaterial can be defined by the combination of "photonic" and "nanomaterial" which is the interaction of light (photon) generation, detection, and manipulation (through emission, transmission) with nanomaterials. Thus, many classes of materials such metamaterials, photonic crystals, plasmonics, diffractive structures can be included. Photonic nanomaterials with efficient optical (or electrical) transfer while keeping standards, belong to the biggest challenges of mankind in the 21 century. Our proposed sciences and technology will have an important impact on future technology and ultimately on our daily life. By answering the scientific and technological questions

that we have addressed to ourselves, new innovation and developing devices will be revealed and will be done. Fast data transfer, high capacity storage at the level of a few nanometers, and saving energy are examples for the impact of the research between light and matter.

Why Semiconducting Nanowires?

Given the demand for ever more compressed and fast systems, there is growing interest in the development of nano-devices which enable new functionality and enhance performance. Over the last 50 years, the scaling of silicon-based electronics shifted to ever smaller dimensions and the focus of device design mechanisms changed from bulk to surface and interface properties which became significantly more important. With the progress in nanomaterial fabrication, Silicon nanowire (Si NW), in particular, a wealth of novel nano-device concepts were developed. A legitimate question is: what makes a silicon nanowire different from bulk systems? The answer is mainly to the credible platform on which to build the next generation of optoelectronic devices. They are always semiconducting, because of their aspect ratio they can be simultaneously used as the active region of

Dr. Muhammad Y. Bashouti,

the device, e.g. the channel of a field-effect transistor, or to connect different devices, the ability to manipulate their properties through controlling their surfaces (by molecules for example), and – most importantly – they can be naturally integrated within the existing Si-based nanotechnology.

What are solar cell based silicon nanowires and why are they important?

Solar cells based aligned Si NW arrays can bare the potential for efficiencies >15%. They can be fabricated on single or multi -crystalline silicon (mc-Si) layers on Si wafer or glass respectively. They can be realized through top down approach such as reactive ion etching (RIE) with lithographic large area nano patterning using densely packed polystyrene (PS) spheres. Geometrical parameters such as diameter, length, density and shape of SiNWs can be tuned for highest absorptions (close to 90%). Various SiNW solar cell concepts are presented: (i) a hybrid organic/inorganic cell with SiNW -based absorber and a hole conducting polymer (PEDOT:PSS encapsulation procedures for long term stability suggested); (ii) a semiconductor -insulator -semiconductor (SIS) cell with SiNW absorber, oxide (few angstroms Al2O3 by atomic



MAX PLANCK INSTITUTE

for the science of light

"THE SCIENCE OF LIGHT AT OUR GROUP AT MAX PLANCK AT ERLANGEN FOCUSES ON RESEARCH FIELDS THAT ARE PARTICULARLY INNOVATIVE OR ESPECIALLY DEMANDING IN TERMS OF FUNDING OR TIME REQUIREMENTS. IN PARTICULAR; OUR INTEREST IS SOMEWHERE BETWEEN BASIC RESEARCH AND TECHNOLOGY DEVELOPMENT WITH A FOCUS ON THE PHOTONIC NANOSTRUCTURES FOR VARIOUS APPLICATIONS IN THE AREAS OF OPTO-, NANO- AND LARGE AREA ELECTRONICS, OPTICAL AND ELECTRICAL SENSING AS WELL AS NANO-PHOTONICS."

DR. MUHAMMAD Y. BASHOUTI, PHYSICS DEPARTMENT, MAX -PLANCK INSTITUTE FOR THE SCIENCE OF LIGHT PHOTONIC NANOSTRUCTURES GROUP

layer deposition -ALD)) tunneling barriers for charge carrier separation and a transparent conductive oxide (TCO – here: Al:ZnO, by ALD). Initial thin film solar cell prototypes reached open -circuit voltages of > 630 mV, short -circuit current densities of even ~ 30 mA/cm 2 and efficiencies >13%.

What is optoelectronic characterization and how is it done?

Correlated microscopies / spectroscopies are used to improve materials / cells: (i) electron beam induced current (EBIC) – to study charge carrier distributions in nano -architectures; (ii) electron backscatter diffraction (EBSD) – to study structural quality of the multi -crystalline Si layer before and after patterning; (iii) integrating sphere measurements, external quantum efficiency, photo - and cathode -luminescence – to study optical properties and (iv) 4 -point nano -probing of individual NWs – to study electrical properties. Novel electrodes (e.g. graphene, silver nanowire webs) to further improve the cells are evaluated.

Why is it important to understand surface properties and which equipment is used to measure surface properties?

The large surface area per unit volume of nano-materials makes them highly sensitive to surface characteristics such as surface morphology, topography and physical/chemicalbonds with otheratoms and molecules. Indeed, the terminationof surface dangling bonds with chemical and/ or biochemicalmoieties is expected to have a significant impact on the final physical and chemical properties of the nanomaterials (such as nanowires). Therefore, Si NW based composites can be scaled down to the molecular level by applying surface functionalization, which cover the NW surfaces with molecules bonded to individual surface atoms.The resulting Si NWs are known as "hybrid-Si NWs". For example, a large body of chemistry has been developed for linking moieties chemically to oxidized Si NW surfaces, generally through -OH chemistry, and to oxide free silicon through Si-C bonds. It is therefore essential to understand the surface properties and charge exchange between the NW surfaces and their bulk on a microscopic level. The main analytical tool adopted in surface science is photoelectron spectroscopy and kelvin probe. Band diagrams will be extracted based on this analysis and correlated with electrical and material properties of the NWs.

With so much discussion about light, can you tell us what Dark Matter andDark Energy are and what does that tell us about the universe?

The interaction between light and matter gives a lot of information about our surrounding. Since the first pioneer discovery of Hasan Ibn al-Haytham(Alhazen)regards real matters at the 10th century, the field of optics get a great light spot from the researchers and a new hypothetical matters were suggested such as Dark Matter. Generally speaking, dark matter is a term used in astronomy and cosmology to describe matter that is undetectable by



Dr. Muhammad Y. Bashouti, Physics Department, Max -Planck Institute for the Science of Light Photonic Nanostructures Group, Germany

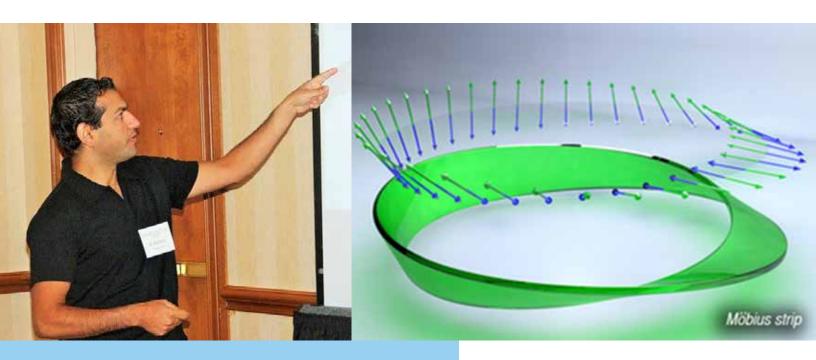
Since 2012, Dr. Muhammad Y. Bashouti has been principle investigator at the research group 'Photonic Nanostructures' headed by Prof. Silke Christiansen in the Division of Prof. Dr. Gerd Leuchs at the Max-Planck Institute for the Science of Light, (MPL).

Bashouti received his bachelor degree in chemistry at the Hebrew University/Israel and completed his Ph.D. in physical chemistry at Technion-Israel Institute of Technology (IIT). He has published more than 35 papers in peer-reviewed publications, is an editorial board member of several scientific and received numerous awards.

The Max Planck Institute for the Science of Light performs basic research in optical metrology, optical communication, new optical materials, plasmonics and nanophotonics and optical applications in biology and medicine. The Institute founded in 2009 is based on the Max Planck Research Group "Optics, Information and Photonics".

The Max Planck Society consists of 83 research institutes with over 13,300 employees who are doctoral students, postdoctoral students, research fellows and visiting scientists. The Max Planck Research Institutes are divided into three categories: Biology & Medicine, Chemistry, Physics and Technology and Human Science. The Max Planck Society was voted best employer in the natural sciences for three consecutive years and is part of a diverse international partnership with affiliates in Canada, USA, Spain, Israel, India, Korea, Japan, France and Switzerland.

The Max Planck Institute for the Science of Light (MPL) was founded in January 2009, making it one of the youngest Max Planck Institutes. It consists of three divisions, several independent research groups and three technical development and service units (TDSU).



its emitted radiation, but whose presence can be inferred from gravitational effects on visible matter and composed of baryons, i.e. protons and neutrons. Interestingly enough, at 1998,the NASA scientists' observed with the help of Hubble Space Telescope (HST)that the Universe was actually expanding more slowly than it is today. So the expansion of the Universe has not been slowing due to gravity, as everyone thought, it has been accelerating. Theorists still don't know what the correct explanation is, but they have given the solution a name and called it "dark energy".

What is white light and how can it be used in our society in the future?

Saving energy is crucial for the mankind nowadays. Bright and white light sources are considered as an energy-saving and are necessary for humanity in many disciplines such as housing, roads, etc. Thus, the great work of Shuji Nakamura, one of the three recipients of the 2014 Nobel Prize for Physics was regarded as the inventor of the blue LED, a major breakthrough in lighting technology. Together with Isamu Akasaki and Hiroshi Amano, they own the prize for the invention of efficient blue light-emitting diodes, which has enabled bright and energy-saving white light sources. This is an example for the importance of the science contribution for society and impact of light related material physics understanding.

How is Mobius strip and polarization of light helping scientist study light and particles?

Möbius strips are simply a three dimensional

structure that has only one side. It's very simple to create, for example, twisting a piece of paper gives you the Möbius strip shape. The Möbius strip created from the polarization of lightand opens up new possibilities for material processing and for and nanotechnology and confirming a light's electromagnetic fieldtheoretical prediction.

What are the most interesting new findings at the Max Plank Institute that is currently expanding our understanding of our world?

I think the best answer to deliver is to continue discussion the pervious question. The Möbius strip generated from laser light by scientists from the Max Planck Institute for the Science of Light (our director Prof. Dr. GerdLeuchs) and Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU), among others, could be the appropriate optical tool to produce the corresponding nano-scopic structures from a material. I suggest to contact him for further information.

"INTERESTINGLY ENOUGH, IN 1998, NASA SCIENTISTS OBSERVED WITH THE HELP OF THE HUBBLE SPACE TELESCOPE (HST) THAT THE UNIVERSE WAS ACTUALLY EXPANDING MORE SLOWLY THAN IT IS TODAY. SO THE EXPANSION OF THE UNIVERSE HAS NOT BEEN SLOWING DUE TO GRAVITY, AS EVERYONE THOUGHT, IT HAS BEEN ACCELERATING. THEORISTS STILL DON'T KNOW WHAT THE CORRECT EXPLANATION IS, BUT THEY HAVE GIVEN THE SOLUTION A NAME AND CALLED IT DARK ENERGY."

"I HAVE USED ATOMIC FORCE MICROSCOPE (AFM) IN MY RESEARCH BECAUSE IT IS VERY USEFUL IN MAPPING THE SURFACE, ROUGHNESS AND TOPOGRAPHY AND I ALSO PLAN ON USING AFM ON MY NEXT PROJECT"

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AFM APPLICATION NOTES

CELLULOSE NANOFIBRILS TOPOGRAPHY & YOUNG'S MODULUS IMAGING USING PARK ATOMIC FORCE MICROSCOPE

By Prof. Javier Carvajal, Pontificia Universidad Católica in collaboration with Park Apps Engineering

INTRODUCTION

Cellulose Nanofibers (CNFs)are biopolymer nanomaterials [1]that look and act in many ways like carbon nanotubes. But, unlike carbon nanotubes, which are hard to synthesize in volume, the CNFs are produced from trees and other plants, so that the supply of the CNFs is endless and the mass production of it is cost effective. Driven by such technical, economic and environmental advantage, people have looked into using CNFs, instead of carbon nanotubes, to create novel nanocomposite materials [2] that are lighter and stronger. Mechanical property of nanocomposite materials can be heavily dependent on exact shape, size, and morphology of CNF elements that go in there to reinforcing it. So, novel metrology technique suitable for accurately characterizing CNF's shape, size and morphology is needed [3, 4].

EXPERIMENTAL

Three samples of cellulose nanofibrils suspension were prepared and provided by Prof. Javier Carvajal Barriga at Pontificia Universidad Católica del Ecuador. The CNF suspension samples were labeled by Park Systems as Sample 1, Sample 2, and Sample 3. A droplet of each sample further diluted in DI water was placed onto freshly cleaved Mica substrate. Liquid droplet residue was blown off using air puff and the sample was left in ambient air to dry. The sample was then mounted onto the Park NX20 Atomic Force Microscope (AFM) stage for AFM imaging. The sample was imaged in air in the Noncontact mode or PinPoint mode AFM using Si-based cantilever AFM probes.

RESULTS AND DISCUSSIONS

All of the three samples of CNFs showed similar shapes and sizes in AFM topographies. The topography of Sample 1 in Fig. 1a shows the CNFs rods that are relatively long and straight. The CNFs are approximately 100 nm to 1000 nm long and 1 nm to 3nm wide, as measured from the AFM height profile in Fig. 1b. It is assumed that the CNF rods are circular cross section and the width is determined by measuring the height of the CNFs. It is interesting to note that the CNFs are well distributed and appear to be oriented along diagonal direction. The air puff that was applied to drive off the residual droplet from the Mica surface was aimed such that the droplet was forced to flow from one side to the opposite side. This rapid droplet flow may have induced the force gradient suited for the CNFs to flow and orient in the direction of the liquid flow.

Although not as preferentially oriented, as shown in Figs 2a and 3a, the CNFs of similar shapes can be observed from the AFM topographies of the other two samples, Sample 2 and Sample 3, respectively. They also appear relatively straight and well dispersed but not as densely distributed–bending or entanglement of CNFs were minimal. The width of the CNFs from the Sample 2 and 3 ranging from 1 to 3nm can be seen from the AFM height profiles of Fig. 2b and 3b, respectively. It is noted that more nanofibrils are seen from the Sample 1 topography than those of the Sample 2 and Sample 3 because the sample 1 was not diluted with DI water as highly as the other two samples.

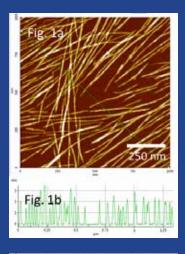
Image in Fig. 4a is topography of CNFs dispersed on Mica which is overlaid with color scale of Young's modulus measured in the PinPoint mode AFM. In Pinpoint mode, the force-distance (f-d) curve is acquired from each pixel in the areas where topography is imaged, and from each f-d curve, elastic modulus is calculated and mapped out in real-time in unison with corresponding topography image. The darker color scale in Fig 4 arefersa lower modulus value. The CNFs are seen darker than surrounding Mica which indicates that The modulus line profile in Fig 4b shows the modulus value for CNFs is ~180GPa while that for Mica is ~ 210GPa. The measrned value of the CNF's Young's modulus is not too far off of the value of 150 GPa that is predicted therocally for crystalline nanocellulose fibrils [5].

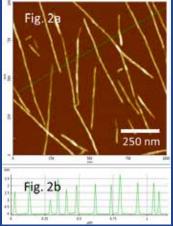
SUMMARY

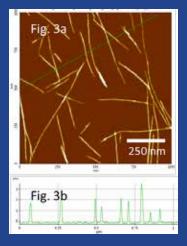
The CNFs were successfully imaged in high resolution and high quality using Park NX20 AFM. The CNFs samples that were well dispersed on Mica without much entanglement allowed to easily measure the width, length as well as Young's modulus of individual CNFs. The results demonstrate that AFM can be used for dimensional nanometrology as well as quantitative nanomechanical property measurements for CNFs characterization.

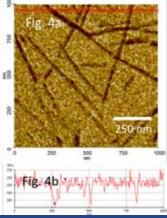
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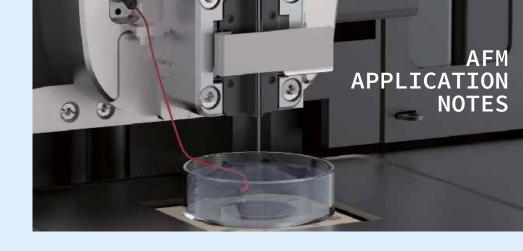
[1] Yanxia Zhang, TiinaNypelo, Carlos Salas, Julio Arboleda, Ingrid C. Hoeger, Orlando J. Rojas, J. Renew. Mater., Vol. 1, No. 3, July 2013. [2] XuezhuXu,HaoranWang,Long Jiang, Xinnan Wang, Scott A. Payne, J. Y. Zhu, and Ruipeng Li ,Macromolecules 2014, 47, 3409–3416 [3] Michael T Postek, Andr´as Vlad´ar1, John Dagata1, Natalia Farkas, Bin Ming1, Ryan Wagner, Arvind Raman, Robert J Moon, Ronald Sabo, Theodore H Wegnerand James Beecher, Meas. Sci. Technol. 22 (2011) 024005. [4] Ingrid Hoeger, a Orlando J. Rojas, Kirill Efimenko, cOrlin D. Velevc and Steve S. Kelleya, Soft Matter, 2011, 7, 1957. [5] Xiaodong Cao, Youssef Habibi1, Washington LuizEstevesMagalhães, Orlando J. Rojas, and Lucian A. Lucia, CURRENT SCIENCE, VOL. 100, NO. 8, 25 APRIL 2011 (and references within).











IN ELECTROLYTE **SOLUTION TOPOGRAPHY IMAGING OF VARIOUS ORGANIC SAMPLES USING PARK NX10 SCANNING ION CONDUCTANCE** MICROSCOPY

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

INTRODUCTION

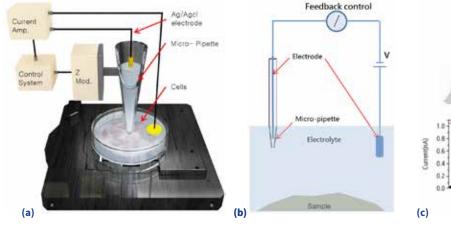
In order to identify and understand the root cause of a problem, and develop an appropriate countermeasure, it is often necessary to characterize a sample in situ. This is needed in several cutting edge applications such as: 1) the study of failure mechanisms in battery electrodes 2) the study of membrane transport of ions between cells and 3) the study of static and dynamic biomechanics at the cellular level. Common to all these applications is the need to perform in situ measurements of a sample experiencing a phenomenon while in an electrolyte solution. Techniques such as Atomic Force Microscopy (AFM) have been used for in-liquid imaging of samples in the past, but AFM may not be considered an ideal solution. Organic samples, in particular have a tendency to soften and swell when immersed in liquid, increasing their likelihood of being damaged by an AFM tip. Furthermore, even the slightest motion of an AFM probe can cause a wet sample to sway and change position during scanning. Since the challenges of a wet sample are not easily met by a technique requiring direct probe-sample contact, an alternative to AFM is desirable. Fortunately, this need has

been filled with the development of Scanning Ion Conductance Microscopy (SICM) and a new instrument: the Park NX10 SICM system. In SICM, sample topography is acquired by regulating the ionic current flowing through the opening of a glass pipette as it moves across a sample surface, all while in an electrolyte solution. This mechanism is completely noninvasive as it applies no pressing force onto a sample. Doing so overcomes AFM's limitations with soft and delicate wet samples and avoids the possibility of accidentally swaying a sample in an in-liquid setup.

EXPERIMENTAL

The Park NX10 SICM is based on the Park NX10 AFM platform. The hardware is virtually the same with the exception of an SICM head replacing the standard AFM head on the system. Instead of using an AFM tip to sense the interaction force between the tip and the sample, the SICM head uses instead a glass pipette with an inner diameter ranging from 80-100 nm or a pipette made of quartz with an inner diameter of 30-50 nm. The pipette is filled with electrolyte solution and connected with an Ag/AgCl electrode while another electrode

AFM APPLICATION NOTES



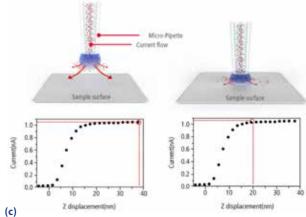


Figure 1. The snapshots showing the (a) SICM hardware setup, (b) circuit mechanism and (c) current-distance relationship between the pipette end and the sample surface.

is connected with the sample in liquid (Figure 1a). A closed circuit is formed with an applied bias between the two electrodes, and the ionic current flowing from the pipette to the sample (Figure 1b). When the pipette gets closer to the sample, the current decreases and the currentdisplacement relationship (Figure 1c) is tracked by the system's fast and accurate feedback loop. If the pipette touches the sample surface, the current drops to zero. The degree of current drop can be used to calculate the surface topography. The pipette scans the surface at a given current set-point (normally 99%), keeping the pipette a few hundred nm away from the surface without actually touching the bottom. Quite similar to Scanning Tunneling Microscopy (STM), which takes advantage of the tunneling current to characterize the materials surface, the SICM keeps track of the current change to provide no-contact and no-force imaging. Furthermore, the SICM no longer requires cantilever tuning prevalent in AFM technique that adds complexity to non-contact in-liquid AFM imaging. The SICM technique does not only provide remarkably stable imaging and quantitative data, but it also makes possible the observation of extra soft or sensitive biological materials, such as live cells.

There are two types of modes in the SICM imaging. The first is called approach-retract scan (ARS) mode. This mode is widely applied to samples with features higher than 1 μ m. Due to the high variation in height, the pipette may easily break if used doing a continuous scan on such samples. Instead, in ARS mode, it approaches the sample until reaching the set-point and fully retracts to a pipette-safe position

before moving on to scan for the next pixel in the image. The approach and retract process is repeated at each pixel of the image to generate the whole topography until it completes the scan. The second mode is called direct current (DC) mode. This mode is mainly for samples with features that are within a few hundred nm in height. The pipette continuously scans the surface at a given current set-point, meaning that it scans with fixed distance from pipette to the sample surface.

RESULTS & DISCUSSIONS

In order to show the ease of use and accuracy of SICM, we picked three representative samples, polydimethylsiloxane (PDMS), a polycarbonate membrane with 400nm pores, and collagen fibrils. The DC or the ARS mode was chosen according to each of the materials' properties.

Polydimethylsiloxane (PDMS):PDMS was chosen primarily for the imaging demonstration, as it is a material used to create standards for SICM system calibrations. That said, it should be noted that PDMS is also used in contact lenses. In situ investigations of PDMS can give us insight into how it behaves in targeted application environments. For the contact lens, its environment would be the surface of the human eye which is regularly moistened by basal tears, a biological lubricant containing electrolytes.

Two PDMS standard samples with different geometries provided by Park Systems were imaged. One is the XY square shaped grid, pitch size 10 µm and the other one is the 117.5 nm high bar shaped grid. Phosphate-buffered saline(PBS) standard solution from Thermo Fisher Scientific was used as the electrolyte solution.

Figure 2 and 3 show the topography images acquired by DC mode of the XY and Z standards respectively. These images successfully exhibit unambiguous and high contrast surface features. The XEI image processing software from Park Systems was used to do all image post-processing including quantification analysis. For the XY standard sample, the 10µm pitch-to-pitch distance and ~160nm grids height are accurately revealed (Figure 2). For the Z standard sample, the height information recovered from DC mode is quite close to the desired value of 117.5 nm.

Poretics polycarbonate membrane: Nanopores refer to holes with the size of tens to thousands of nanometers that exist in thin membranes. Due to the penetration of molecules or proteins through the pores, the membranes can be powerful sensors of molecules and ions, and this property is applied in a variety of fields including engineering, chemistry, biology, medicine, and so forth. Recent advances in nanotechnology can precisely control the morphology as well as physical and chemical properties of the pores to make them increasingly attractive for regulating and sensing transport at the molecular level[1]. In addition, with certain chemical modification, nanopore membranes can be used for highthroughput nanoparticle separation or filtration [2, 3]. Extensive research was also carried out in the fabrication, characterization, and modeling of nanopore membranes. Traditional AFM typically relies on the interaction forces between a probe tip and the sample surface.

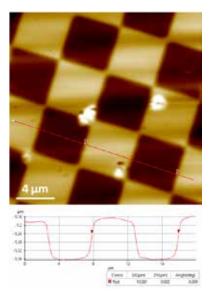


Figure 2. Topography image of the XY standard sampleacquired by DC mode. Scan size 20µm×20µm. Image size215 px× 215 px.

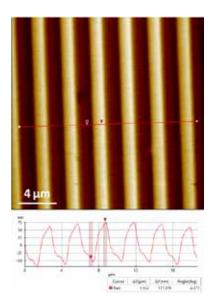


Figure 3. Topography image of the XY standard sampleacquired by(DC mode. Scan size 20µm×20µm. Image size 256 px × 256 px.

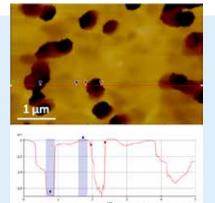


Figure 4.Topography images of 400nm diameter pore polycarbonate membrane recorded in PBS solution with SICM ARS mode. The average pore depth is ~ 600nm.

> many biological samples, these fibrils soften and swell once rehydrated and they can sway if disturbed with an AFM probe.

> This third sample, provided by our collaborators at Niigata University in Japan, allowed us to demonstrate SICM's capability to image a sample whose nanoscale topography would otherwise be difficult to acquire with other microscopy techniques.

The collagen fibril was cut and spin-cast on a petri dish and imaged with a Park NX10 system in PBS solution with SICM. Due to the large height variation of collagen fibrils, we applied ARS mode only. The Park XEP software was used to run all the tests. As shown in Figure 5, the protein bundles as well as each individual fibrils can be identified clearly in the 10µm sized images at a 256 x 256 pixel resolution.

The thinnest fibril that SICM distinguished (indicated by the black arrow) was about 90 nm only in width. Given the fact that there is no real contact or force between the end of the pipette and the sample surface, such resolution is quite impressive.

SUMMARY

The topography images of all three samples

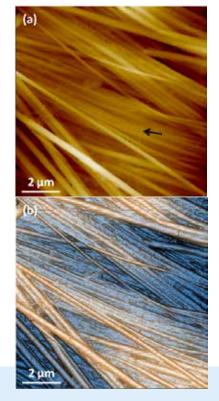


Figure 5. (a) Topography and (b) contrast enhanced topography images of collagen sample imaged in PBS solution with SICM ARS mode. Individual fibril can be clearly identified and the thinnest one observed is about 90nm in width, as pointed by the black arrow. Scan size 10µm×10µm. Image size 256 px× 256 px.

were acquired efficiently and accurately in buffered solution using the Park NX10 SICM from Park Systems. This innovative technique effectively overcomes the problems associated with traditional in-liquid AFM investigations, and it provides a no-force solution ideal for all materials, especially the ones that are very soft or sensitive. With the ease of use and high performance, the SICM effectually provides researchers with high quality in-liquid imaging to help them better research and study their samples in various solutions at nanoscale.

REFERENCES

1.Adiga, S.P., et al., Wiley Interdiscip Rev Nanomed Nanobiotechnol, 2009. 1(5): p. 568-581. 2.Anmiv, S.P., et al., J. Phys. Condens. Matter, 2010. 22(45): p. 454107. 3.Tokarev, I. and S. Minko, Adv. Mater, 2010. 22(31): p. 3446-3462.

4. Chen, C., Derylo, M.A. and Baker, L.A., Anal. Chem., 2009. 81(12): p.4742-4751 5. Zhou, Y., Chen, C. and Baker, L.A., Anal. Chem., 2012. 84: p.3003-3009

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We would like to thank Ms. Wenqing Shi and Prof. Baker Lane at University of Indiana for providing the nanopore polycarbonate membranesamples and fruitful discussions of SICM.

However, it is not sufficient enough to test the sharp geometry of a sample membrane surface or the ionic transportation through its pores. SICM has proven to be a powerful technique in accomplishing both [4, 5].

We tested the poretics polycarbonate membrane with 400nm pores supplied by GE Water & Process Technologies in PBS solution with SICM. The membrane was put on PDMS substrate for imaging. In order to prevent the pipette from being damaged by the deep pores, we only ran ARS mode on this sample. Figure 4 exhibits the pore sizes acquired by SICM. The data reveals the pores have an average diameter of about 416 nm and an average depth of about 600 nm. Indeed, more advanced work with this type of sample has been done such as identifying the transport activity of individual pores[4, 5].

Collagen fibrils: Collagen fibrils are a widely accepted standard sample for measurements of biological and soft material properties. Like

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Park Systems provides the best quality AFM for Semiconductor microscopy for failure analysis and defect review, an integral part of the process of advancing semiconductor research and manufacturing. Park Smart ADR is the most advanced defect review solution available, featuring automatic target positioning without the need for labor intensive reference marks that often damage the sample. The Smart ADR process improves productivity by up to 1,000% compared to traditional defect review methods and offers up to 20x longer tip life thanks to Park's groundbreaking True Non-Contact[™] Mode AFM technology. Since 2007, Park has gained a reputation as the technology leader of nanoscale measurement and systems in both research and industry for the semiconductor and other industries and their impressive client list includes Harvard, Stanford, NASA, NIST, Micron, Imec, Seagate, Western Digital and IBM.

Park NX-Wafer makes accurate, repeatable, and reproducible sub-Angstrom roughness measurements for the flattest substrates and wafers with minimized tip-to-tip variation. Park NX-Wafer delivers the industry's lowest noise floor of less than 0.5 Å throughout the wafer area, combined with True Non-Contact Mode™ to achieve reliable measurements even for the long-range waviness measurement of scan sizes up to 100m x 100m.



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Electrochemistry which deals with the links between chemical reactions and electricity given by Dr. Lane Baker of Indiana University

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"Electrochemical imaging techniques utilize the measurement of electrons or ions to record the electrical, chemical, biological and physical properties of a sample. Park SICM provides high resolution, noncontact imaging necessary for advancing scientific analysis *in this critical area of nanoscience* research," explains Dr. Baker, who leads the Baker Group at Indiana University, a group of analytical and materials chemists whose research covers a broad area of topics in electrochemistry, bioanalytical chemistry, new mass spectrometry methods, materials for electrode fabrication and instrumentation development."

High Performance Polymers



Webinar on the design, synthesis and characterization of new polymers and nanoscale materials with applications that spread across a wide range

of industries including energy production, aerospace, consumer electronics, medicine, and food production is given by **Dr.** Rigoberto Advincula Professor of Macromolecular Science & Engineering, Case Western Reserve University.

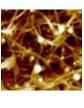
"As we scale up these industries to tackle more complex challenges and *extreme demands, the development* of high-performance polymers plays a critical role in sustaining and advancing our current capabilities as a society. Park Systems' Atomic Force Microscopes are important as they produce highly accurate 2D and 3D microscopic images that identify, at the nanoscale level, all of the various reactions polymers have to external stimuli."



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Growthcone Network In addition to electrochemical applications, SICM is also versatile enough to image live cells in aqueous conditions. Imaged here is a mass of rodent neurons.



Nanopatterned lines on a precursor polymer thin film Nanoscale smart coating features such as these patterned lines can be imaged using a conductive AFM setup.

Layer-by-Layer Nanostructured Coatings

This webinar on nanostructured smart coatings discusses the enhanced performance in materials acquired by layered coatings that improve



performance traits such as conductivity, tensile strength, and corrosion resistance.



of patented features for multimode investigations including superior z-axis or vertical profiling resolution," states Dr. Advincula, Professor at Case Western University and the webinar presenter. "Park AFM is used when applying multi-layered smart coatings to accurately characterize the morphology and changing properties of each layer deposited at the most stringent nanoscale requirements, only available using Park AFM with the world's only patented noncontact mode design."

Metrology Challenges and Opportunities for Semiconductor Market

ights for Electronics Ma

This webinar from Solid State Technology, addresses advanced materials used in semiconductor silicon wafer manufacturing and new device structures and designs under various stages of development, presented by industry expert and SPIE Fellow, Dr. Alain Diebold, whose career includes cutting edge research on advanced metrology methods to improve nanoelectronics fabrication as Director of the Center for Nanoscale Metrology at CNSE. Many of the new design challenges and opportunities will be presented in the webinar, showcasing the enhanced concepts under research and being commercialized. A critical role in the development and ongoing application of new device structures and materials is the advanced microscopy provided by world-leading Park Systems Atomic Force Microscopes, designed to meet the industry's strict requirements for nanoscale accuracy.

Alain Diebold is Interim Dean College of Nanoscale Sciences. He is also the Director of the SRC NRI



INDEX Center. He is a fellow of the American Vacuum Society and SPIE as well as a senior member of the IEEE. He is an associate editor for IEEE's

Transactions on Semiconductor Manufacturing. Before moving to Albany, Alain was a Senior Fellow at SEMATECH. Prior to moving to Austin, He was a senior chemist at Allied Signal in Morristown, NJ. Alain received his PhD from Purdue University in 1979.

"AFM enables the determination of surface and sidewall roughness and feature line shape and is often used in conjunction with TEM, CD-SEM, and Scatterometry in Hybrid Metrology. The goal of Hybrid Metrology is to use the measurement information from multiple methods to improve 3D determination of feature shape and dimensions," explains Dr. Alain Diebold, Interim Dean at the College of Nanoscale Sciences and Director of the SRC NRI INDEX Center.

Much emphasis is being placed on new designs for more complex device structures and exploration of highly advanced new materials. This webinar will focus on some of the new device structures such as FinFETs and 3D stacking, new materials that are emerging and

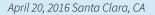
how they are proceeding towards future manufacturing. The industry continues to search for materials for transistors and interconnects. For example, a high dielectric constant "high K" material that is compatible with Ge channels are key to enabling the use of Ge channels. Thinner barrier layers for copper interconnects are another topic of research as well as the often mentioned replacement for copper itself.

"Lithography continues to drive a significant research effort. Quadruple patterning will replace double patterning. Double patterning (Self Aligned Double Patterning) refers to the use of oxide spacers on the side of lithographically patterned lines to double to line pattern density," explains Dr. Diebold. "The space process can be applied multiple times. When the spacer process is applied twice, the pattern density *is quadrupled. There are many* variations of the use of multi-pattern methods."

"Over the past several years, the industry has also investigated Directed Self Assembly of Block Co-Polymers as a means of increasing pattern density beyond that possible with traditional lithography. Research into EUV lithography also continues to be a critical topic, adds Diebold.

INDUSTRY NEWS

PARK SYSTEMS GLOBAL EXPANSION IN AFM MARKET INCLUDES APPOINTMENT OF NEW EXECUTIVES



Park Systems, world-leader in atomic force microscopy (AFM) announced today the appointments of Charlie Park as Senior Vice President of Global Sales, and Jong-Pil Park as Vice President of Production. The new Vice Presidents will develop strategic global sales initiatives and further Park's best-in-class position as the world's leading AFM manufacturer by enhancing production capabilities to meet increased product demands and customer technology requirements.

"The addition of these highly talented executives is a continuation of Park's strategic business focus on global expansion," states Dr. Sang-il Park, Park Systems Founder and CEO. "The new appointments establish the groundwork for an integrated world-wide targeted sales operation and will jointly aggressively increase our production capabilities to meet anticipated product demands."

Charlie Park's role as Senior Vice President of Global Sales will focus on further establishing Park Systems trademarked global Atomic Force Microscope (AFM) brand. He brings over three decades of global sales and marketing experience at leading companies including Samsung where during his tenure as Senior VP he expanded global operations, leading the sales & marketing divisions in both the Korean and European Headquarters. He has had numerous global assignments in the UK, Germany and the Netherlands and will use his successful global sales experience to implement Park's long-term strategy for growth and innovation leadership in Atomic Force Microscopes.

Jong-Pil Park, PhD-ME, newly appointed Vice President of the Production Division will expand the highly successful production capabilities of Park AFM with quality-driven state-of-the-art systems and leading-edge performance capabilities. His successful 30 year career as an engineering-based expert in production and quality management include operations vice president at Motorola Korea, Production VP at Doosan Infracore Co and at Huneed Technologies Company, and a senior engineer at Defense Technology & Quality. His leadership skills combined with technical knowledge of automated atomic force microscope equipment will expand Park Systems world-renowned production systems to meet the AFM needs for an expanding world market.



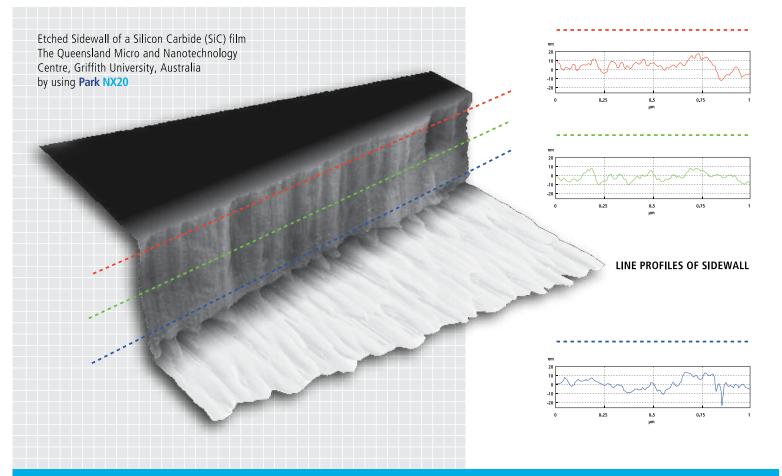
Charlie Park, Senior Vice President of Global Sales



Jong-Pil Park, Vice president of Production

About Park Systems

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



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Park NX10

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