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MESSAGE FROM EDITOR

Welcome to the 21st edition of NanoScientific. As we look back at the beginning of a new year, NanoScientific has a few surprises coming out. First, we will introduce NanoScientific TV, compelling interviews and videos of the leading-edge nano researchers, a sneak peek into nanotechnology of tomorrow. We will also introduce NanoScientific App, which will give you easier access to submit abstracts, read articles online and network with others using the new NS Community Forum.

We have published articles in this issue from four of our young presenters at NanoScientific Symposium for a Changing World, including Winner of “The Most Promising Future Application Award” at the Poster Exhibition, Estephany Santiago, with her article Magnetic Nanosavengers: The New Trend To Improve Water Quality and a short Q&A from her below. Congratulations for all of our winners at NSCW, Best Poster – Lynne Rogers, Committee Choice – Cody Leasor, Most Promising Future Application – Estephany Santiago, Committee Choice – Hang Ran, Microscopy Award – Myunghoon Choi, Best Presentation – Louis Santiago, Most Promising Future Application – Priti Kidambi and to all the presenters who gave inspiring talks about future research that will improve our world. In this issue, you will find four of these presenters’ articles with research leading to new approaches in understanding nanoscience which were presented at the Virtual NanoScientific Symposium. Next year, in 2023, we are planning many more virtual NanoScientific Symposia with an in person option, if possible.

Stay tuned for dates of these events. I am also proud to announce a new member to the NanoScientific Board, Marine Le Bourer, CEO of NanoTechnology World Association. Marine has been a huge collaborator with NanoScientific for several years and we are glad to have her as an official member of the board.

We pledge to continue to bring you exciting articles and symposiums that highlight the best of the most creative scientific minds of our time, who collectively are elevating nanotechnology to great new heights. We applaud the intensity and dedication that nano researchers put into the effort to unravel the mysteries of science one atom at a time. It is our honor to report on their achievements. Stay safe during the New Year and may you have a very prosperous and rewarding year to come.

Keibock Lee
Editor-in-Chief

INSET PHOTO ON COVER:
Park AFM’s nanolithography is an advanced AFM technique used to pattern nanoscale shapes on the sample surface. A bias voltage is applied to the tip to generate an oxide pattern on a metallic or semiconductor substrate in the bias-assisted or anodic oxidation method. The 3D image of oxide patterns formed through Park AFM nanolithography shows the types of nanotextured structures that can be fabricated with Angstrom-level imaging precision using SmartScan3D®. Nanolithography of Park NX10 AFM system.

Q&A with Estephany Santiago, winner “Most Promising Future Application” Poster at NanoScientific Symposium for a Changing World

What was it like to receive the NanoScientific Most Promising Future Application Award?
It was an honor and a pleasant surprise that my research can be considered for a promising application gives me the motivation to keep working towards new applications.

Did you enjoy the NanoScientific Symposium and would you recommend it to others?
The NanoScientific symposium is a great symposium, the best online experience that I had until now, very well organized with a great interactive website and with great presenters/posters. I will recommend all my colleagues and friends to join the upcoming symposiums.

What is your hope for your research to expand the field? To help prevent water pollution?
My hope is that governments and private companies are encouraged to use new environmentally-friendly alternatives and invest in projects like this to take advantage of water and its reuse. It is urgent and mandatory that we focus on treating and reusing the water we throw away, to reduce our environmental impact and help the most needed communities.
MAGNETIC NANOSCAVENGERS: THE NEW TREND TO IMPROVE WATER QUALITY

Estephany Santiago, Georgina Pina-Luisa, Marisela Martínez-Quiroz, Oscar Perez-Landeros, Navor Rosas-González, Benjamin Valdez-Salas, Mercedes T. Oropeza-Guzmán

Introduction

Recycling water is mandatory in regions where water scarcity is due to climatic conditions, but also it offers the opportunity to mitigate water pollution by reducing the discharge of wastewater to natural surface waters. The challenge is to develop sustainable processes to recover clean water from wastewater avoiding the use of non-degradable materials (synthetic polymers and resins), keeping low energy consumption and diminishing the investment cost to operate the technology. Water conditioning technologies such as inverse osmosis, chemical precipitation, ion exchange, and electrochemical removal, are generally used to improve water quality and aesthetics (such as alkalinity and hardness) after primary and secondary processes. However, all of them are onerous and require meticulous maintenance to keep efficient operations and improve water quality [3,5]. In this pathway, we report the conception of magnetic nanoparticle scavengers with high potential to remove hardness and alkalinity from reclaimed water (RW). With this innovation we look to increase the water potential reuse in a simple magnetic contact stage to trap salts.

In this paper three types of eco-friendly magnetic nanoparticles were prepared using chitosan, nanodiamond powder, and grafted chitosan. All of them were coupled with magnetite nanoparticles to obtain magnetic chitosan (MCH), magnetic nanodiamond (MND), and magnetic carbamoyl chitosan (MCCH). All the materials were characterized previously their use, by FT-IR, STEM, Zeta Potential, DLS, and TGA studies, proving the binding between magnetite and the modifiers.

Experimental

Low molecular weight chitosan and nanodiamond powder were used as a model to evaluate their alkalinity and hardness removal capacity. We found out that each magnetic nanoscavenger proved to be effective for trapping and removing alkaline carbonates from reclaimed water, and we demonstrated it by different techniques. At the same time, we have the possibility of recovering them for further use, reducing the operation cost.

Results and discussion

RW original characteristics are shown in Table 1.

Table 1. Composition of reclaimed water obtained from the wastewater plant treatment “La Manta” in Tijuana, Baja California, México.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Reclaimed Water</th>
<th>Cations in Reclaimed Water</th>
<th>Units (ppm)</th>
<th>Anions in Reclaimed Water</th>
<th>Units (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Turbidity (NTU)</td>
<td>23</td>
<td>L+</td>
<td>0</td>
<td>F</td>
<td>0</td>
</tr>
<tr>
<td>pH</td>
<td>8</td>
<td>Na+</td>
<td>287.59</td>
<td>Cl</td>
<td>378.55</td>
</tr>
<tr>
<td>Color (HCl)</td>
<td>253</td>
<td>K+</td>
<td>33.95</td>
<td>NO3</td>
<td>43.00</td>
</tr>
<tr>
<td>Total suspended solids (mg L-1)</td>
<td>15.3</td>
<td>Ca2+</td>
<td>57.37</td>
<td>Br</td>
<td>0</td>
</tr>
<tr>
<td>Particle size (nm)</td>
<td>566.9</td>
<td>Mg2+</td>
<td>126.64</td>
<td>SO4</td>
<td>383.55</td>
</tr>
<tr>
<td>Zeta Potential</td>
<td>-7.59</td>
<td>Mg2+</td>
<td>126.64</td>
<td>NO3</td>
<td>117.41</td>
</tr>
<tr>
<td>Chemical Oxygen demand (mg L-1)</td>
<td>74.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total Organic Carbon (mg L-1)</td>
<td>19.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Electrical conductance (μS cm-1)</td>
<td>2.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 1. Schematic illustration of the formation of the nanoscavengers MND, MCH, and MCCH.

Figure 2. Characterization of Magnetic nanoscavengers by: FT-IR, a) MND, b) MCH, c) MCCH, TGA, d) MND, d) MCH, f) MCH, d) MCCH, DLS/Zeta Potential vs pH: g) MND, h) MCH, i) MCCH, and STEM: j) MND, k) MCH, l) MCCH. The white line represents a scale bar of 200 nm.

Figure 3. a) MW, b) MND, c) MCH, d) MCCH, e) Traps, f) MCH, g) MCCH; and SEM: h) MND, i) MCH, j) MCCH. The white line represents a scale bar of 200 nm.

Figure 4. a) pH, b) MND, c) MCH, d) MCCH, e) Traps, f) MCH, g) MCCH; and STEM: h) MND, i) MCH, j) MCCH. The white line represents a scale bar of 200 nm.

Figure 5. a) pH, b) MND, c) MCH, d) MCCH, e) Traps, f) MCH, g) MCCH; and STEM: h) MND, i) MCH, j) MCCH. The white line represents a scale bar of 200 nm.

Figure 6. a) pH, b) MND, c) MCH, d) MCCH, e) Traps, f) MCH, g) MCCH; and STEM: h) MND, i) MCH, j) MCCH. The white line represents a scale bar of 200 nm.
Armando Melgarejo, Ben Schoenek, Jiali Zhang, and Byong Kim
Park Systems, Inc., Santa Clara, CA, USA

Introduction
The field of nanotechnology has diversified into different areas of research, from materials science to biotechnology. Many of these applications are based on the capability to fabricate or manipulate nanostructured materials [1]. One convenient technique for structuring, manipulation, and fabrication at the nanometer scale is atomic force microscopy (AFM) nanolithography. All AFM nanolithography techniques can be classified into two general groups in terms of their operational principles: force-assisted and bias-assisted nanolithography [2]. The force-assisted method consists of applying a large force to a sharp tip to mechanically modify the surface atoms or molecules of the sample and produce trenches on the surface. In this case, the interaction between the tip and the sample is purely mechanical [2]. On the contrary, the bias-assisted method entails applying a voltage between the AFM tip and the substrate in contact with the sample. The tip-sample voltage induces an electrochemical reaction that produces oxide on the surface of the substrate [3]. This application note demonstrates an electrochemical process called anodic oxidation using the bias-assisted lithography method to create oxide patterns on the surface of a silicon substrate. The success of this technique relies on using the AFM tip as a biased cathode to the sample surface. Also, the water meniscus around the sample acts as an electrolyte for the chemical reaction. The environment humidity directly influences the size of the meniscus [1]. Therefore, this application shows nanoscale oxide line formation using AFM-analyzed lithography on a Park NX10 AFM system using Park SmartLitho, the new nanolithography software developed by Park Systems. Park SmartLitho [3] can also be used for nanomanipulation, scratching techniques, and high voltage lithography.

Figure 1. Park SmartLitho™ software. A) Vision & Monitoring view. B) Nanolithography mode panel. C) Lithography design area. D) Object list. E) Objects edit panel.

Figure 3. a) DLS (black line) and Zeta Potential (blue line) of the RW from the WWTP “La Morita”. The pH was adjusted using solutions of 0.001M HCl and 0.001 M of NaOH. b) Alkalinity(purple) and hardness(yellow) measurements for RW from the WWTP “La Morita” at pH 7, 8, and 9, expressed as mg L-1 of CaCO3.

Figure 4. DLS measurements of untreated RW (purple), and RW treated with MNP (black), MND (gray), MCH (yellow), and MCCH (orange) at pH of 7, 8, and 9. d) Comparison of the Zeta Potential measurements of untreated RW (purple), and RW treated with MNP (black), MND (gray), MCH (yellow), and MCCH (orange) at pH of 7, 8, and 9.

Figure 5. a) Comparison of the hardness removal of MNP (black), MND (gray), MCH (yellow), and MCCH (orange). At pH of 7, 8, and 9, expressed as percentage in mg L-1 of CaCO3. b) Comparison of the alkalinity removal of MNP (black), MND (gray), MCH (yellow), and MCCH (orange). c) Comparison of the COD removal of MNP (black), MND (gray), MCH (yellow), and MCCH (orange).

References
AFM cantilever probe is mounted on a piezo-electric bimorph shaker, attached to the AFM head 2 piezoelectric scanner. The shaker vibrates the AFM cantilever probe at its resonant frequency, which is automatically selected along with the vibration amplitude. As the probe nears the sample surface, the vibration amplitude decreases. Likewise, as the probe moves away from the surface, the amplitude increases. The 2 piezo scanner moves up and down and adjusts its height to maintain a constant vibration amplitude. The height adjustment of the scanner thus reflects the height change on the sample surface. The AFM raster scan detects the height change point-by-point in the X-Y direction to map out the three-dimensional surface topography. The tip approaches the surface, and the image is retaken promptly with the same parameters as the baseline AFM image. The newly drawn line is clearly visible and parallel to the previously drawn line (Figure 2, Top Left). Notice the image position remains the same, showing the control of the XY scanner, separate from the Z scanner, even at smaller scan sizes. In this example, the lithography process is done in contact mode, and therefore, imaging in non-contact mode is an option. However, after the manipulation, it is important to take the image using non-contact mode to avoid disturbing the particles present on the sample.

After verifying the success of lithography process, the final image is taken and exported to the analysis software called Park XEI®. In this case, we can see that the previously drawn line is around 35 nm in width, and the newly-drawn one is 47 nm (Figure 2, Bottom). Both lines are drawn using the same lithography parameters. Nevertheless, the line on the right side, drawn first, is observed to be narrower than the oxide line on the left. This might be due to performing the lithography in contact mode. In contact mode, the tip is rubbed against the sample surface as the lines are drawn. The rubbing can cause the end of the tip to wear out and blunt. The blunted tip can fabricate wider oxide lines than the ones drawn with a sharper tip (1.5).

We can see that the resulting line height is approximately 1.4 nm for both lines when adding another cursor. Park XEI® software is capable of showing a three-dimensional rendering of the image taken (Figure 2, Top Right). With the 3D representation, we notice that the lines drawn during this oxide growth process have a lower height than the nanoparticles on the surface.

Conclusion
This application note discusses how to use Park SmartLitho™ software to easily design and customize nanoscale oxide patterns using Park NX10 AFM. With only a few seconds of runtime, this simple test case demonstrates a bias-assisted nanolithography process, which successfully generates oxide patterns as narrow as 35 nm in width. Although the process itself employs contact mode, true non-contact mode before and after the operation validates that the new oxide line is indeed parallel to the line in the baseline image. This test case also demonstrates the scanning and imaging precision of Park’s XY scanner. Overall, this study shows that mode in Park SmartLitho software is excellent tool for generating well-defined nanoscale patterns and features.

References
Polytetrafluoroethylene (PTFE), commonly known as Teflon, is a fluorocarbon solid that has one of the lowest coefficients of friction. Therefore, it is widely used as a material with low adhesion or as an inert coating. Despite its chemical simplicity, PTFE exists in four different crystalline phases that have been studied with electron diffraction techniques. Interestingly, no high resolution AFM data with electron diffraction techniques can easily be acquired using a 3rd eigenmode of Multi75Al-G cantilever at 1.1Mhz and set point of 12.5mN. The outermost surface layer, is able to be observed in Figure 1c and d, which is always confined to the utmost/outermost surface layer, is able to accurately reproduce results obtained from volume average techniques. Thus, proving that AFM is an important tool in the investigation of the molecular structure of polymers. In fact, AFM is able to provide more structural information by shining a light onto the amorphous phase of the polymer. These crystalline domains exhibit flat terraces as observed in Figure 1c and d. On the following height and phase images (Figure 2), we examine these flat terraces at a bit closer. A 100 nm x 100 nm height image (Fig. 2a) shows ~5Å steps with sharp edges. Corresponding phase images (Fig. 2b and c) reveal true molecular nature of these flat steps – we can clearly resolve single molecules with a period of 5.6Å. A cross-section (Fig. 2d) gives a clear periodic structure of the terrace. From this cross-section, we can also measure the width of a single line at full width at half maximum being 3.5Å. This determines the maximum resolution achieved on this sample. When comparing the observed period of 5.6Å to the reported diffraction data of PTFE unit cell a = 5.66Å, we can note a remarkable agreement.

**Figure 1.** Large-scale height and phase tapping mode images of a Teflon surface. a – height image, 512 x 512 px, scan rate 0.5 Hz; b – phase image acquired simultaneously with image a – phase image, 512 x 512 px, scan rate 4 Hz; c – high resolution phase image showing both crystalline and amorphous regions, 512 x 512 px, scan rate 4 Hz. Captured by Park NX20 Large Sample AFM.

**Figure 2.** High resolution AFM images of a Teflon surface showing single PTFE molecules. a – height image, 512 x 512 px, scan rate 0.5 Hz; b – phase image, 512 x 512 px, scan rate 4 Hz; c – phase image, 512 x 512 px, scan rate 6 Hz; d – high resolution phase image showing both crystalline and amorphous regions, 512 x 512 px, scan rate 4 Hz. Captured by Park NX20 Large Sample AFM.

**Conclusion**

To conclude, we have demonstrated a straightforward practical approach for high-resolution imaging using higher eigenmodes of a standard cantilever in tapping mode on a commercial large-scale NIX0 Park Systems AFM achieving molecular resolution of a Teflon sample. AFM, a surface sensitive technique which is always confined to the utmost/outermost surface layer, is able to accurately reproduce results obtained from volume average techniques. Thus, proving that AFM is an important tool in the investigation of the molecular structure of polymers. In fact, AFM is able to provide more structural information by shining a light onto the amorphous regions of Teflon – something that diffraction techniques cannot easily achieve. For instance, a phase image (Fig. 2b) shows that PTFE molecules extend further into the amorphous region of the polymer. The highly localized and high resolution nature of AFM places it in a unique position to investigate the structure of polymers in real space.

**References**


**Conference Topics**

- Nanophotonics, nanoelectronics and photonics applications
- Advanced materials: organic, inorganic and hybrid
- Nanoscience and nanotechnology
- Nanomechanical and electrical characterization of soft materials
- Overcoming barriers in AFM
- Advanced imaging
- High-resolution imaging

**SPM Methods:**

- Nanomechanical and electrical characterization
- Characterization of soft materials
- Advanced imaging
- High-resolution imaging

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Vladimir Korolkov

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- Park NX-Wafer
  Low noise, high throughput atomic force profiler with automatic defect review

- Park NX-3DM
  Innovation and efficiency for 3D metrology
Abstract

Carbon-based materials are being used increasingly in various applications recently. This is primarily attributed to their customizable nature and multiple allotropes. Carbon could be sourced naturally as well as produced by synthetic methods for the further processing steps into customized forms such as sheets, wires, quantum dots, powder, granules, pellets, rods, tubes, composites, etc.

From the viewpoint of the system working cycle, behavioral characterizations are essential to substantiate the applicability of a material. Several analytical and characterization techniques are effectively put into use towards qualitative as well as quantitative estimation, measurement, and verification. In this study, powdered activated charcoal (activated carbon), lampblack (carbon black), and powdered graphene (graphene) are subjected to investigations such as x-ray diffraction (XRD), light microscopy (LM), scanning electron microscopy (SEM), infrared (IR) thermal imaging, electrical probing (DL), and chemical sampling, and the observations are assessed and correlated with pertinent applications.

Keywords: Characterization, Carbon black, Activated carbon, Graphene, Material.

Introduction

Carbon exists abundantly in various forms which could be acquired, synthesized, and tailored towards their interesting physicochemical, thermal, mechanical, electrical, magnetic, and optical properties. The current study attempts to underline the efficacy of various characterization methods and relate them to carbon’s appositeness in multiphased applications [1,2,3,4].

Materials and Methods

Lampblack is synthesized from Panchadeepa oil, a heterogeneous lipid constituted by ghee and plant oils, subjected to incomplete combustion and vapor-deposition upon copper. Activated charcoal (medicinal grade) was procured from Tudor Laboratories [5]. Materials and Methods

For testing solubility, 5 mg of each solute was mixed in 100 mL of each solvent - water, 90% THF, 90% DMF, and IP liquid was mixed in 100 mL of each solvent - water, 90% THF, 90% DMF, and IP liquid. The forward-looking infrared images in Figure 3 indicate a uniform temperature distribution across carbon black and graphene. However, activated carbon coated glass slide exhibits two different temperature distributions, the other distribution being congruent in dimensions but differing by an average temperature of +5K at a distance of 20 µm, respectively.

Carbon samples were refrigerated at 277K, 20 µm, respectively. XRD scan was conducted on Bruker XRD, light microscopy (LM), scanning electron microscopy (SEM), infrared (IR) thermal imaging, electrical probing (DL), and chemical sampling, and the observations are assessed and correlated with pertinent applications.

Table I. XRD Peak Analysis

<table>
<thead>
<tr>
<th>Carbonaceous Material</th>
<th>Crystalline peaks (°2θ)</th>
<th>Amorphous peaks (°2θ)</th>
<th>Crystallite size (nm)</th>
<th>% Crystallinity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Activated carbon</td>
<td>50</td>
<td>24, 44, 79</td>
<td>34.33</td>
<td>0.06</td>
</tr>
<tr>
<td>Carbon black</td>
<td>30</td>
<td>24, 44</td>
<td>15</td>
<td>2.13</td>
</tr>
<tr>
<td>Graphene</td>
<td>26.8</td>
<td>44, 79</td>
<td>13.54</td>
<td>85.56</td>
</tr>
</tbody>
</table>

Table II. Solubilities of carbons in different solvents

<table>
<thead>
<tr>
<th>Carbonaceous Material</th>
<th>Solubility in Water</th>
<th>Solubility in Liquid paraffin</th>
<th>Solubility in DMF</th>
<th>Solubility in THF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Activated carbon</td>
<td>Hydrophobic</td>
<td>Suspension</td>
<td>True solution</td>
<td>True solution</td>
</tr>
<tr>
<td>Carbon black</td>
<td>Hydrophobic</td>
<td>Suspension</td>
<td>Colloidal</td>
<td>True solution</td>
</tr>
<tr>
<td>Graphene</td>
<td>Hydrophobic</td>
<td>Suspension</td>
<td>True solution</td>
<td>True solution</td>
</tr>
</tbody>
</table>

Table III. Ohmic measurements [5]

<table>
<thead>
<tr>
<th>Carbonaceous Material</th>
<th>Resistance (Ω)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Activated carbon</td>
<td>5.6 * 10^3</td>
</tr>
<tr>
<td>Carbon black</td>
<td>0.25 * 10^3</td>
</tr>
<tr>
<td>Activated carbon</td>
<td>1.42 * 10^3</td>
</tr>
</tbody>
</table>

Results and Analyses

XRD of carbon materials corroborates the crystalline nature. From Table I, it is deduced that the % crystallinity in Graphene >> Carbon black > Activated carbon. The crystallite size of carbons is in accordance with the amorphousness, i.e., Activated carbon > Carbon black > Graphene.

SEM images in Figure 2 offer more quantifiable and scalable data than LM. Secondary electron detectors demonstrate topographical-visualizations - clustery porous activated carbon and carbon black, fractal periodic flaky graphene. Graphene and activated carbon have voids, whereas carbon black consists of aggregates. Backscattered electron detectors construe the elemental composition as image contrast, and the carbonaceousness in Graphene > Activated carbon > Carbon black.

The forward-looking infrared images in Figure 3 indicate a uniform temperature distribution across carbon black and graphene. However, activated carbon coated glass slide exhibits two different temperature distributions, the other distribution being congruent in dimensions but differing by an average temperature of +5K at a distance of 20 µm, respectively. This might be due to the amorphous nature of activated carbon and phonon scattering.

The images in Figure 4 correspond to those in Figure 3, with the former being a three-dimensional representation of the latter. The plane into the paper/screen corresponds to the two-dimensional surface of the carbon-coated glass slide, and the normal axis to it represents the temperature variation map. Graphene > Activated carbon >> Carbon black.

 Materials and Methods

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Results and Analyses

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LM resolution limits its depth of analysis, and hence, properties such as particle size distribution, porosity, surface topography cannot be quantified to a large extent. From Figure 1, the morphology is very uniform for Graphene > Activated carbon > Carbon black. Carbon black spreads erratically, forming layers and shadows in imaging. Activated carbon and carbon black have a higher surface area than graphene since, for an equivalent quantity, graphene is less scattered. Powdery form yields a more pronounced airgap in activated charcoal [5].

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Abstract

Carbon-based materials are being used increasingly in various applications recently. This is primarily attributed to their customizable nature and multiple allotropes. Carbon could be sourced naturally as well as produced by synthetic methods for the further processing steps into customized forms such as sheets, wires, quantum dots, powder, granules, pellets, rods, tubes, composites, etc.

From the viewpoint of the system working cycle, behavioral characterizations are essential to substantiate the applicability of a material. Several analytical and characterization techniques are effectively put into use towards qualitative as well as quantitative estimation, measurement, and verification. In this study, powdered activated charcoal (activated carbon), lampblack (carbon black), and powdered graphene (graphene) are subjected to investigations such as x-ray diffraction (XRD), light microscopy (LM), scanning electron microscopy (SEM), infrared (IR) thermal imaging, electrical probing (DL), and chemical sampling, and the observations are assessed and correlated with pertinent applications.

Keywords: Characterization, Carbon black, Activated carbon, Graphene, Material.
and activated carbon exhibit a near- symmetrical heat flux profile. Activated carbon exhibits an enclosed heat variation with the outward heat flux getting flattened, whereas graphene exhibits a similar variation with the heat flux curving outwards in a continuous fashion as it stems from the inward. The heat flux from carbon black is distorted and is asymmetrical.

Soluble-solvent molecular interactions vary based on miscibility, homogeneity, and affinity of materials. All carbons repelled water molecules, floating on water in spite of repeated stirring. The three carbons are heterogeneous in liquid paraffin, forming a suspension. Carbon black, after ultrasonication in THF, and graphene exhibit homogeneity with DMP and THF. Activated carbon is active in THF and DMP. Despite molecular agitation by ultrasonic waves, it exhibited only partial solubility. The observations are briefed in Table II.

Discussion
Material crystallinity, the fundamental of solid-state physics [6], is perceptible from XRD results yielded in Table I. Paramount insights into the morphology, surface topography, stacking, and carbon composition were obtained from the micrographs in Figures 1 and 2, which could be applied in interfacial surface tension in antenna coating [7], platelet formation in layered-piercing [8], and conductive filling in voids of ultraparacap carbon electrode [9]. From Figures 3 and 4, the thermal flux and superficial heat distribution of carbons after cooling were understood, and these would be crucial in passive cooling [10], adsorptive heat flux from carbon black is distorted in fashion as it stems from the inward. The heat flux becomes flattened, whereas graphene exhibits a near-symmetric heat flux profile. Activated carbon and activated graphene exhibit a near-symmetric heat flux profile. The current amplifier is necessary to amplify the current signal. The current amplifier both increases the current magnitude, therefore, a current amplifier is needed to amplify the current signal. The current amplifier both increases the current magnitude, therefore, a current amplifier is needed to amplify the current signal.

Introduction
Sophisticated, high-performing technology components often require electrical properties that are relatively similar. When DC bias is applied, the current flow between the conductive tip and sample is monitored, and the electrical properties are measured. The conductive tip acts as an electrode. Generally, the current has a very low magnitude; therefore, a current amplifier is needed to amplify the current signal. The current amplifier both increases the current magnitude, therefore, a current amplifier is needed to amplify the current signal.

Conclusion and Future Scope
In the present work, observations from XRD, LM, SEM, IR imaging, solubility, and O were scrutinized and related to carbon applications. The outcomes and impact could be furthered by conducting other analyses such as TEM, AFM, EDX, Raman spectroscopy, ICP-OES, NMR, TGA, BH-BET, and molecular dynamics for cumulative supplementation.

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Disclosures

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Figure 2. Schematic Autonomous L-AFM and SSRM-AFM

C-AFM and SSRM operate in the Contact mode regime, and their imaging principles are relatively similar. When DC bias is applied, the current flow between the conductive tip and sample is monitored, and the electrical properties are measured. The conductive tip acts as an electrode. Generally, the current has a very low magnitude; therefore, a current amplifier is needed to amplify the current signal. The current amplifier both increases the current magnitude, therefore, a current amplifier is needed to amplify the current signal.
Figure 2. Topography and resistance images of Sample 1 obtained in ambient air and high vacuum using SSRM mode. The topography images in Figures 2a and 2b show a polished surface with a height difference of 159 nm on the cross-section. However, they do not contain significant information related to the device pattern. The measurement positions in both images are the same in ambient air and in high vacuum, as can be seen in their matching topography line profiles in Figure 2c. Therefore, environmental compensation is possible. Figures 2d and 2e are the resistance images taken simultaneously with the topography images in Figures 2a and 2b, respectively. By Figures 2d and 2e, the resistance image in high vacuum shows more features and details than the one taken in air. In fact, line profiles of the resistance images, as shown in Figure 2f, show a significant difference in the resistance distribution in air and vacuum. Since there is no oxidation or water layer in the vacuum, the SSRM shows a higher sensitivity in a vacuum than in air due to the improved electrical contact between the tip and sample. Characterizing electrical properties with this level of detail is essential for understanding the functionality of the MOSFET device.

Figure 3 presents the current images of Sample 2 obtained in ambient air and high vacuum using C-AFM mode. Small grain-structures with distinct contrast are visible in the current image obtained in high vacuum, while the current image in air shows less detail. The maximum current measured in high vacuum is 827 nA, while it is 59 nA in air. The line profiles of the current images, as shown in Figure 3c, confirm that a consistently larger current is measured in high vacuum than in air. This improved electrical sensitivity in high vacuum is likely due to the removal of water and contaminants that are typically present on the sample surface in air.

**Abstract**—Thin films are ubiquitous, with uses ranging from optoelectronics to antibacterial coatings. Unfortunately, precisely quantifying how the choice of substrate influences epitaxial growth remains an unsolved problem. Here, a novel thin film of holmium oxide with record-high magnetic saturation was grown on a variety of substrates. Conventional attempts to extract epitaxial information and characterize the growth mechanism were ineffective, due to the unique size regime of the product. Instead, a signal-processing inspired Fourier method was used to elucidate information on epitaxial ordering from purely topographical data, avoiding the pitfalls of atomic-level diffraction. Further, we define and utilize an inner product-based metric termed a q-score that can quantify the relative degree of ordering of epitaxial crystallites. The q-score provides a direct measure of epitaxy, enabling more quantitative future studies of thin film growth.

**INTRODUCTION**—Magnetic resonance force microscopy (MRFM) is an emerging technology that offers the promise of single nucleon detection in individual biological samples or nanodevices [1]. Such a device functions like a nanoscale magnetic resonance imaging (MRI) machine, applying a radiofrequency (RF) current and measuring response with a magnetic cantilever, all against the background of a strong magnetic field [2,3]. Effective MRFM demands a large signal to noise ratio (SNR). From [3], this is

$$SNR = \frac{N_{\text{RF}}}{2k_{\text{B}}T} \left( \frac{q}{pb} \right) \left( \frac{\gamma B}{\gamma B_0} \right) \left( \frac{k_{\text{B}} T}{k_{\text{B}} T_0} \right)$$

Since the SNR is quadratically dependent on the magnetic field gradient $\beta/\alpha$, developing strong, new magnetic materials is important for optimizing MRFM. Recently, a new crystal phase of holmium oxide thin film with record-high magnetic saturation (Figure 2 Tesla) was synthesized using a thermal physical vapor deposition technique [4]. Holmium films grown on different substrates (A-plane sapphire, C-plane sapphire, and amorphous quartz) under otherwise identical deposition conditions have different morphological properties, suggesting that the substrate controls film growth. This direction is not a chemical process, as the two sapphire substrates are chemically identical, and growth occurs in a regime well below the vaporization of all the substrates [5,6]. Rather, the different crystal structures and orientations of the substrates visually appear to direct holmium growth in different ways. For example, in Figure 1, we see the holmium oxide film grows into crystalline triangles regardless of surface, but on the Sapphire C substrate (Figure 1b), they seem to be aligned with one another. When thin films grow, individual atoms land on the surface and then move around until they lose their kinetic energy and reach a thermodynamic minimum [7]. This can occur in different ways depending on how the landing atom aligns with the substrate crystal atoms. When films grow on a substrate, the substrate can direct their growth to align with its structure, creating a periodic result [8]. This is termed epitaxial growth.

To determine whether different substrates are directing growth of different crystal phases of holmium oxide, we attempted a variety of standard diffraction techniques, including coarse x-ray diffraction (XRD) and more fine-grained elastic recoil detection (ERDA) and transmission electron microscopy (TEM). Most of these techniques determine bulk crystal structure and elemental composition, ultimately determining that we have a new phase of holmium oxide. However, they operate at the wrong size scale to determine whether the crystallites themselves are oriented. XRD and ERDA are too coarse to detect individual crystal grains reliably [9,10]. Available TEM only operates at a small domain size (~50 nm), while the crystallites in question are on the order of 100 nm. Although TEM has been successful at studying epitaxial growth in the past, it has traditionally been done on much thinner and smaller nanostructures [11] or been focused on defects [12]. In other words, no techniques were successful at revealing the orientation of individual crystal grains.
The following notation will be used throughout the manuscript:

- $f(x,y)$ denotes the z-height/brightness at a given spatial coordinate of an AFM image.
- $\mathcal{D}(f)$ denotes the transform of $f(x,y)$, this is the FFT in all cases except in section V.
- $\mathcal{D}(f)$ denotes a Frobenius inner product.
- $A^*$ denotes the complex conjugate of $A$.

**TOPOGRAPHICAL CRYSTALLINITY**

It is well known that using different substrates results in the growth of different types of crystal grains. There are three main cases of thin film growth, reviewed extensively in [8] and [13].

**Epitaxial crystalline growth.** In the case of epitaxial growth, the crystal grains align perfectly with the substrate’s crystal structure, and the edges of crystal grains also align with the substrate and thus align with each other. Topographical data of this type of growth presents as a perfect tessellation of crystal edges. The shape of the grains depends on the shape of the substrate’s exposed crystal plane. The FT described in Equation (2), with translation and multiplication accounted for, can be expressed as a rotation of the quantification algorithm.

**Non-epitaxial crystalline growth.** In the case of non-substrate directed crystalline growth, nucleation and growth occurs in random directions. Although crystals with hard edges can still form, they will not be aligned with each other. This can be modeled as an assortment of randomly oriented crystals, where the shape of the crystal depends mostly on the thin film material’s intrinsic crystal properties.

**Random crystalline growth.** In the case of non-epitaxial, non-crystalline growth, the thin film grows haphazardly on an unordered substrate, forming unordered structures. This case is largely non-instructive, and has no consistent morphological ordering.

In this experiment, holmium oxide film was grown on two different crystal planes of sapphire, A-plane, and C-plane, depicted in Fig. 2. The A-plane atomic cross section is a rectangular lattice, while the C-plane cross-section contains a hexagonal lattice. Meanwhile, holmium naturally grows into a hexagonal lattice [21] that is a close match in terms of lattice strain to C-plane sapphire, so it is expected that the C-plane sapphire will clearly direct its growth, whereas holmium will grow into randomly oriented triangular or hexagonal crystallites on the A-plane sapphire. The amorphous quartz substrate is not crystalline, and thus cannot epitaxially direct growth.

**Epitaxial crystalline growth**

- $
abla \cdot \mathbf{u} = 0$
- $\mathbf{u} = (u_x, u_y)$
- $\mathbf{F} = (F_x, F_y)$
- $\mathbf{X} = (X, Y)$
- $\mathbf{Y} = (Y, Z)$
- $\mathbf{X} = (X, Y, Z)$
- $\mathbf{Y} = (Y, Z, X)$
- $\mathbf{Z} = (Z, X, Y)$

Fig. 3. Theoretical Fourier domain images of ordered (epitaxial) vs. disordered (non-epitaxial) triangles, (a, 6), and (g) input random images, while (b, 6), (f) and (h) and their respective FFTs, (e) and (h), the unordered triangles create a mess of overlapping, rotated hexagons, in (a) and (e), uniformly, perfectly oriented tessellation of identical equilateral triangles creates honeycomb patterns out of discrete data. In (a) and (e), translations of triangles of varying height and size both create different patterns.

**Fig. 4.** Holmium oxide thin films grown on three different substrates under identical conditions and (a) under microscope, (b) a close up of grown triangular patterns on sapphire A (1100), (c) and (d) C sapphire (1100), while (e) and (f) contain triangular crystallites, has no clear ordering in its FFT (b), however, the thin film on sapphire C (1100), which also contains triangular crystallites, has hexagonal ordering in its FFT (d), suggesting crystallite alignment. To confirm, there is no clear ordering in either the AFM image (e) or FFT (f) of the film grown on amorphous quartz, only in (g) and (h) two-order patterns and symmetry, qualitatively compared to Fig. 3c.

Quantifying epitaxial growth at present, two tools are available to quantify the presence of epitaxy on nanostructure and thin film growth.

Direct measurements using TEM, as in [11] and [12]. This reveals the degree of lattice mismatch at the substrate-film interface and is thus the ideal tool for quantifying epitaxial growth. However, this tool is ineffective when the resulting thin film does not perfectly match up with the sapphire crystal lines (as with the holmium oxide), and when the size regime of interest extends beyond a couple nanometers (in our case, the relevant regime is hundreds of nanometers; see Fig. 1).

Theoretical lattice mismatch [22]. Every material has a theoretical lattice mismatch, and the difference between lattices is quantified by the Lattice strain $\varepsilon$. In the case of the sapphire-holmium oxide system, this strain is determined by the crystal spacing of the substrate, $d_{\text{substrate}}$, and the crystal spacing of the material $d_{\text{material}}$ in Equation (5). Typically, a lattice strain of below 1% (0.01) suggests epitaxial thin film growth.
However, this technique is not a true “measurement” of ordering, but rather a convenient percentage that can be reported along with qualitative images of clearly ordered growth (i.e. aligned nanowires). Lattice mismatch does not always determine epitaxial growth and vice versa [23]. For example, the lattice strain between holmium oxide and sapphire C is |0.053|, but it is clear by inspection that choice of substrate influences growth (Fig. 4a,c,e).

As a result, development of a quantification scheme beyond lattice is paramount to understanding partial quantifier, we could simply take a Frobenius inner product. Careful when comparing different results to make sure the same alignment is as follows:

1. Load the images and take their FFTs.
2. Remove uniform background noise.
3. Remove outliers above an experimentally determined high quantifier. In the future, an LFP could be used.
4. Normalize each FFT F by dividing by its Frobenius norm, ∥F∥F, where the inner product is given in (6).
5. Compute what we here define as the q-score, defined as the inner product of the processed FFTs A′Q′.

\[ q = \langle A', Q' \rangle \]  

\( q = \langle A', Q' \rangle \) (7)

6. In addition, compute the error, defined below. Note that low quantifiers are correlated with high errors.

\[ \epsilon = \sum |q_{ij} - Q_{ij}|^2 \]  

\( \epsilon = \sum |q_{ij} - Q_{ij}|^2 \) (8)

There is one further challenge: rotating an image will also rotate the FFT of that image. For instance, rotating the quantifier triangle tessellation image by 10 degrees will also rotate its Fourier transform by 10 degrees. The proof is below.

Working in polar coordinates, set \( \rho \cos \theta \), \( \rho \sin \theta \) quantifiers of \( \rho \cos \theta \), \( \rho \sin \theta \) (Fig. 1c), multi-color, size image (Fig. 18), and multi-color image (Fig. 14). Recall that a high q-score is indicative of high matching with the ideal pattern and subsequent high ordering. While the particular q-score changes depending on the choice of quantifier, the ordering remains the same. Sapphire C is the most ordered, followed by sapphire A. Amorphous quartz is the least ordered.

In addition, note that the multi-color, multi-size triangle tessellation quantifier offers the greatest distinguishability. This is expected, given that it is most similar to the AFM pattern that would actually be observed in the case of epitaxial growth crystallites are all oriented, but can vary in height and lateral size, represented by variations in color and size respectively.

\[ \langle A, Q \rangle = \sum |a_{ij} - q_{ij}|^2 \]  

\[ \langle A, Q \rangle = \sum |a_{ij} - q_{ij}|^2 \] (6)

However, in order for this computation to work, the FFTs must be preprocessed first. Otherwise, images that are very bright will automatically have a very large inner product, or triangles that are not exactly aligned with those of the quantifier may image have a lower inner product despite perfect alignment between triangles in the AFM image.

The quantifier algorithm is as follows:

1. Load the images and take their FFTs.
2. Remove uniform background noise.
3. Remove outliers above an experimentally determined high quantifier. In the future, an LFP could be used.
4. Normalize each FFT F by dividing by its Frobenius norm, ∥F∥F, where the inner product is given in (6).
5. Compute what we here define as the q-score, defined as the inner product of the processed FFTs A′Q′.

\[ q = \langle A', Q' \rangle \]  

\( q = \langle A', Q' \rangle \) (7)

In order to avoid accidental mismatch between potential perfect tessellations of the substrate and quantifier (i.e. the substrate hexagonal pattern could, by chance, be rotated 30 degrees away from the quantifier FFT hexagonal pattern), we will rotate the quantifier by small increments, going through the above algorithm and computing the q-score and error for each rotation. We can perform this rotation in real space and then take the Fourier transform, as rotating in real space is equivalent to (7). Then, we select the highest q-score (i.e. the best match) and set that as the true q-score, along with its concurrent error. Note that for a 6-fold symmetric image such as a tessellation of triangles, we only have to rotate through a total of 60 degrees before the periodic pattern returns, decreasing computational time. In addition, the amount of each rotation can be tuned according to computational demands.

A quick internal calibration of the quantification algorithm is that it should return 1 if Q = 0, this is indeed the case.

Next, we test this algorithm on our images, depicted in Figure 5. The different quantifiers used are the mono-color image (Fig. 1c), multi-color, size image (Fig. 18), and multi-color image (Fig. 14). Plotting this into Eq. 2, we see Fig. 6.

Using 3 images from each of sapphire A and sapphire C, each image taken 5 mm from the last along the same line of translation, we compute the q-scores between each permutation of the 3 images. Zero rotation of FFTs is used, as rotation occurring over the course of translation would indicate that the long-range ordering is broken. The resulting q-score for sapphire C is much higher than that of sapphire A (Fig. 6). Thus, the film grown on sapphire C is well aligned across enormous spatial jumps (5 mm >> 5 microns, the span of each image). This preservation of ordering over a long range is highly unlikely to occur unless the underlying substrate is directing at each point, making this a direct and quantitative measure of epitaxy.

The error terms, computed using Equation (8), are small for sapphire C, but large for sapphire A (Fig. 7), confirming a perfect match in Fourier space between different images from sapphire A and consequent non-epitaxial growth.

One interesting result is that the q-score, when comparing shifted images approaches 1 for the aligned growths on sapphire C (Fig. 6). This approach utilizes the shifted AFM images as quantifiers. However, the q-score is nearly 1000 times smaller when using an abstracted perfect tessellation as the quantifier (Fig. 5). As a result, the best use of the quantifier algorithm is likely to compute self q-scores, i.e. quantify the difference in Fourier transforms between different regions of a single thin film to detect long-range ordering. This use alleviates one of the biggest problems with the quantifier algorithm: the choice of the ideal quantifier or FFT to compare our signal to. From Figure 5, it is apparent that altering quantifiers can change q-scores by nearly 50%, and that differentiability is sometimes a challenge (i.e. the q-scores of sapphire A and sapphire C are very close for all but the multi-color, multi-size quantifier). Comparing images to their downstream counterparts adds an internal control, which is desirable as the exact pattern of epitaxial growth is unknown due to uncertainties at nanoscale.

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The DISCRETE COSINE TRANSFORM: ANOTHER WEAPON IN THE ARSENAL? The success of the using the q-score to quantify long-range ordering relies on the transformation of real space to a form of frequency space. Although the Fourier transform was used in the quantifier algorithm, alternative transforms could potentially be used. For example, the discrete cosine transform (DCT) is often
used for lossy image compression, relying on an orthogonal basis of cosine functions (rather than as simple linear functions, as with the FT). The DCT is defined below, omitting normalization factors:

$$F_{nx} = \sum_{x=0}^{2^m-1} f_{x} \cos \left( \frac{\pi}{2^m} x \right)$$

We define our DCT quantifier algorithm (DQA) as a version of the DCT, except taking a 2D DCT in all places where a 2D FFT was previously performed. Applying the DQA to a few test cases shows that it is potentially useful, but quantitatively challenging to optimize.

Fig. 8. Applying DCT to epitaxial characterization. (b) depicts the DCT of (a), while (d) depicts the DCT of (c). The ideal ordered triangles have much more “striped” DCT than the randomly oriented triangles. Here, the ideal DCT distinction disappears when comparing thin films grown on sapphire A (e) and sapphire B (f); (i) depicts the q-scores for the DQA on all substrates, they are virtually identical.

When looking at the discrete cosine transforms of ideal ordered vs unordered triangles (Fig. 8a,b), not immediately apparent. The DCT of the unordered triangles in (8a) appears random (8b), while that of the triangular low-resolution topographical images alone.

CONCLUSION
Because of the broad applications of epitaxially grown films, mechanistically understanding their growth is critical to developing and characterizing new materials. In particular, a set of holmium oxide thin films grown on three different substrates under otherwise identical conditions were found to be exceptionally paramagnetic, but the degree of epitaxy was not measurable via traditional differential techniques, inhibiting mechanistic understanding of their growth. However, it was possible to collect extensive topographic data via AFM, and it was obvious that the holmium oxide crystals appear to exhibit symmetry akin to that of a triangular tessellation.

Here, we developed a qualitative theoretical framework for transformations of ordered vs unordered triangles into k-space, demonstrating f-fold symmetry for both ordered case and symmetric spreading for unordered triangles. We compared the FFT of holmium oxide crystals grown on various substrates to the transforms of these ideal cases, which suggested that growths on sapphire C are epitaxially aligned.

Next, we developed and tested an algorithm for calculating gas sensors to a modified inner product between FFTs that quantifies the amount of similarity (and thus epitaxial ordering), confirming that sapphire C was indeed more ordered than sapphire A. Since this q-score relies on comparing two images, two, approaches can be used: comparing a theoretically ideal transform to any image or comparing differences from the reference transform. The latter allows comparison and potential identification of triangular vs other types of symmetry, while the latter also allows direct quantification of long-range epitaxy with a robust internal control. This algorithm was modified to operate using DCTs and DFTs for further tests but little practical quantification success.

Ultimately, the C-sapphire substrate is most effective at epitaxially directing holmium oxide thin film growth, making it a candidate for future experiments. More broadly, utilizing Fourier space enables quantification of epitaxial growth in the absence of crystal data. The q-score is a tool measures and quantifies epitaxial growth, allowing mechanistically guided development of new materials.

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A COMPARATIVE STUDY OF ATOMIC FORCE MICROSCOPY BETWEEN AM-KPFM AND SIDEBAND KPFM, PRINCIPLES AND APPLICATIONS

Introduction
Since the development of Atomic Force Microscopy (AFM), development of measurement techniques to optimize both spatial resolution and sensitivity has been an important issue. Various measurement modes have been developed that characterize electrical, mechanical, magnetic, and thermal properties. Among those, Kelvin Probe Force Microscopy (KPFM) [2] is a well-known mode that enables the analysis of both surface morphology and surface potential distribution properties on a nanometer scale [1]. Several techniques have been utilized extensively to investigate the localized charge distributions on a surface layer [3], local surface potential distributions [4] variations of surface work functions [5] and ferroelectric domains [6], in a variety of research fields. KPFM shows a simultaneous delivery of surface topographical information and surface potential/work functions of samples by applying AC and DC voltage. Using the same configuration as electrostatic force microscopy (EFM), KPFM monitors the surface potential and work function of samples by applying a DC bias (VDC) to nullify tip-sample interactions. The VDC can be used: comparing a theoretical surface potential map to the conventional method, amplitude modulation (AM- KPFM) [7] the method which relates to the electrostatic force between the sample surface and tip via the amplitude of the AC frequency signal (Figure 1a). This implies that AM-KPFM is mainly affected throughout the AFM cantilever deflection signal and the work function of the sample can be estimated. Unlike AM- KPFM, SIDEKPFM is mainly affected by the AFM tip-sample interactions due to an averaging effect [7][8].

However, SIDEKPFM has an advantage in terms of spatial resolution of the surface potential measurement, amplitude modulation (AM- KPFM) and SIDEKPFM, the method which relates to the electrostatic force between the sample surface and tip via the amplitude of the AC frequency signal (Figure 1a). This implies that AM-KPFM is mainly affected throughout the AFM cantilever deflection signal and the work function of the sample can be estimated. Unlike AM- KPFM, SIDEKPFM is mainly affected by the AFM tip-sample interactions due to an averaging effect [7][8].

In this study, we present results obtained with AM-KPFM and SIDEKPFM on the same samples with extended areas of different surface potentials. From these results, we directly compare the spatial resolution of AM-KPFM and SIDEKPFM under identical conditions.

Materials and Methods
SIDEKPFM
SIDEKPFM is an optional AFM mode used to measure electrical properties of the sample surface. Figure 2 shows the connection diagram of SIDEKPFM which uses two lock-in amplifiers to measure the amplitude and phase of each sideband. On lock-in amplifiers 2 and 3, a signal with a frequency of 60 kHz is used as the reference signal (lock-in amplifiers 2) to detect the sideband signals with a frequency of 60 kHz in the detection lock-in amplifier 2. The decoupled signals from the two lock-in amplifiers are averaged and used for feedback into DC voltage. The AFM controller applies a corresponding VDC so the average sideband peak size becomes zero (AC tip bias set in Park ScanSmart™).
Test samples and AFM cantilever
ZYH grade highly-ordered pyrolytic graphite (HOPG, SPI Supplies) was utilized for showing layer by layer surface potential differences. A silicone substrate with low conductivity polymer dots was used to display the surface potential contrast of two different materials. An IFX dopant calibration sample (NPD, Infineon) was chosen to monitor the stepwise electrical signal. This sample consisted of 5 different n-type implant areas in order to compare the electrical signals. All imaging was performed using an Au coated NSC36-C (resonance frequency, 65 kHz; spring constant, 0.6 N/m; length, 130 µm) with the same AFM parameters.

Results and discussion
This study analyzes three different samples to compare the electrical resolution performances of AM-KPFM and Sideband KPFM. Figure 3 shows surface potential images of HOPG by AM-KPFM and Sideband KPFM. Sideband KPFM confirms the sharp contrast due to the HOPG layer, whereas AM-KPFM shows blurry edges. As shown in the line profile analysis, Sideband KPFM has an approximately two-fold higher surface potential difference on layers (~70 mV) than AM-KPFM (~35 mV, red arrow). Also, Sideband KPFM displays small HOPG fragments very clearly, while AM-KPFM shows a blurry image.

Next, the analysis of polymer dots on a silicone substrate was performed by AM-KPFM and Sideband KPFM. Additionally, Sideband KPFM both in and out of lift mode were compared to investigate the non-contact feedback performance for Sideband KPFM. In Figure 4, all images show contrasts between the polymer dots and the silicone substrate. However, AM-KPFM indicates a lower surface potential contrast than Sideband KPFM including lift mode. For Sideband KPFM, there is no significant difference between the no-lift mode and a 5 nm lift mode, which the line profile analysis confirms. Surface potential differences between the polymer dots and the silicone substrate are approximately 180 mV in AM-KPFM, ~300 mV for the no-lift Sideband KPFM, and ~330 mV for the 5 nm lift Sideband KPFM.

Lastly, AM-KPFM and Sideband KPFM were used on a IFX dopant calibration sample to compare stepwise electrical resolution. In AM-KPFM, different doping level of n-type implant are recognized; however, checking the electrical signal step was difficult. In contrast, the stepwise electrical signal is displayed in both no-lift and 5 nm lift Sideband KPFM. Based on several repetitive tests, all Sideband KPFM analyses show better resolution than those of AM-KPFM. If the line profile suggests, Sideband KPFM can monitor step-by-step surface potential differences, though AM-KPFM can detect a gradual signal decrease.

Conclusions
In this study, we compare AM-KPFM and Sideband KPFM using a variety of samples. Those results indicate that Sideband KPFM has superior resolution relative to AM-KPFM. In surface potential images, Sideband KPFM shows a clear electrical contrast and detects relatively small changes from a line profile analysis, the surface potential differences in Sideband KPFM are higher than that of AM-KPFM, which implies the superior performance of Sideband KPFM. However, the comparison between no lift and 5 nm lift in Sideband KPFM shows no difference for all tested samples. Due to the accuracy of Park Systems’ AFM feedback system, there is no tapping between the AFM tip and sample surface. Thus, it is possible to obtain the true surface morphology and a clear KPFM signal as well. Since the development of KPFM, it has become one of the more useful AFM options utilizing surface material science and in semiconductor engineering. It is a unique technique for surface potential or work function mapping on the nanoscale, and the Sideband KPFM option offers superior spatial resolution and improved electrical sensitivity for material characterizations.

References
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