Cross-platform integration of AFM with SEM: Offering the best of both worlds

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INTRODUCTION

Hybrid instrumentation involving the combination of atomic force microscopy (AFM) with other characterization capabilities has been a field with much activity and new product entries over the past 5 years.

AFMs have recently been integrated with a wide variety of instrumentation and spectroscopies including: Raman spectroscopy, infrared spectroscopy, time-of-flight secondary ion mass spectrometry (TOF-SIMS), and scanning electron microscopy (SEM). The challenge in these hybrid solutions is to not compromise on the capability of each individual technique while enabling new characterization methods through an integration that provides an improved, more complete understanding of the sample.

SEM as a microscopy method has particular complementarity with AFM where integration of the two offers key unique advantages:

1. **Combination of quick screening and high resolution for 3D mapping.** The SEM as an individual technique is one of the most common forms of microscopy for rapid screening of materials through the ability to quickly scan over large areas. Although the sub-nm lateral resolution of the SEM is impressive, it does not provide true z resolution on the nm-scale when imaging the morphology and topography of the sample. In contrast, true topography or z information is precisely what the AFM offers, making a hybrid technique like the AFSEM™ ideal for mapping a surface in 3D.
2. **Complete visualization of surface properties** that fills in the gaps of each technique. On the one hand, the AFM maps topographic, mechanical, and electrical properties on the nanoscale, where these properties clearly represent a gap in the data provided by SEM. A shortcoming in the AFM's capabilities, however, is the absence of any chemically specific information. On the other hand, the SEM with its electron dispersive X-Ray spectroscopy (EDX) capabilities can provide elemental information of the surface. Through combination of EDX with the other surface properties mapped by AFM, this tool can now provide a complete 3D visualization of the surface including information on its elemental composition, true 3D topography, and mechanical and electrical properties.
3. **Tomographic imaging** through focused ion beam (FIB) and SEM. By combining the AFM with a dual-beam FIB-SEM system, the complete visualization described in #2 can now be extended in the depth axis. All the surface properties can now be mapped as a function of depth by using the FIB to etch away layers while the AFM/SEM information is collected, e.g., allowing for 3D mapping of mechanical properties.
4. **New possibilities for in-situ nanomechanical testing.** The design of the system employs a tip-scanner that permits integration with relatively heavy or specialized stages such as a tensile stretching stage or a nanoindenter. This enables completely new capabilities for correlative in-situ nanomechanical testing.

The AFSEM™ from Nanosurf/GETec provides a powerful new capability that joins the forces of AFM and SEM. AFM imaging in all the conventional AFM modes is now possible simultaneously under an SEM beam without disruption to either technique. Through its unique design and the use of self-sensing cantilevers, the unit is the first cross-platform AFM that can be integrated into all major commercial SEMs. Providing all the advantages described above, it represents an accessible innovation with significant capability enhancement for materials science research.

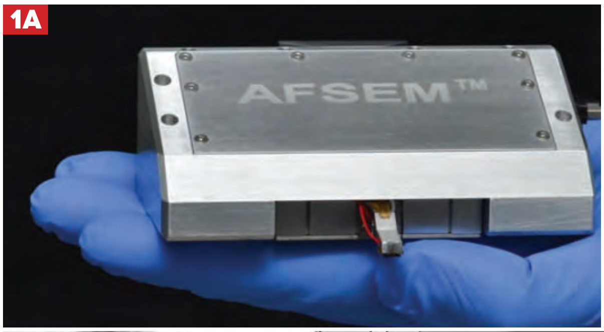
MATERIALS AND METHODS

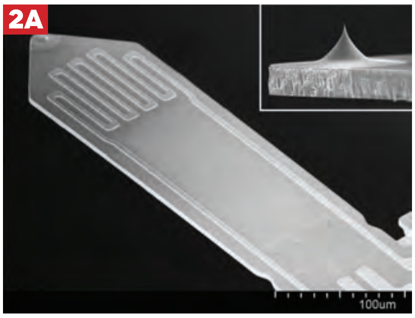
A: AFSEM™ SYSTEM DESIGN

This recently released product results from a joint collaboration between Nanosurf (Switzerland) and GETec (Austria) to build and design an AFM from the bottom up with the expressed purpose of integration into an SEM chamber.

The compact scanner fits into the palm of a hand as seen in Figure 1a below. A schematic of the instrument is shown below in Figure 1b where the light blue region is the coarse stage, the scanner is in orange, and the adapter is in the light gray section under the coarse stage. Thus, the AFM is integrated in a non-obstructive way with the electron column and other techniques on the right.

This cross-platform AFM can





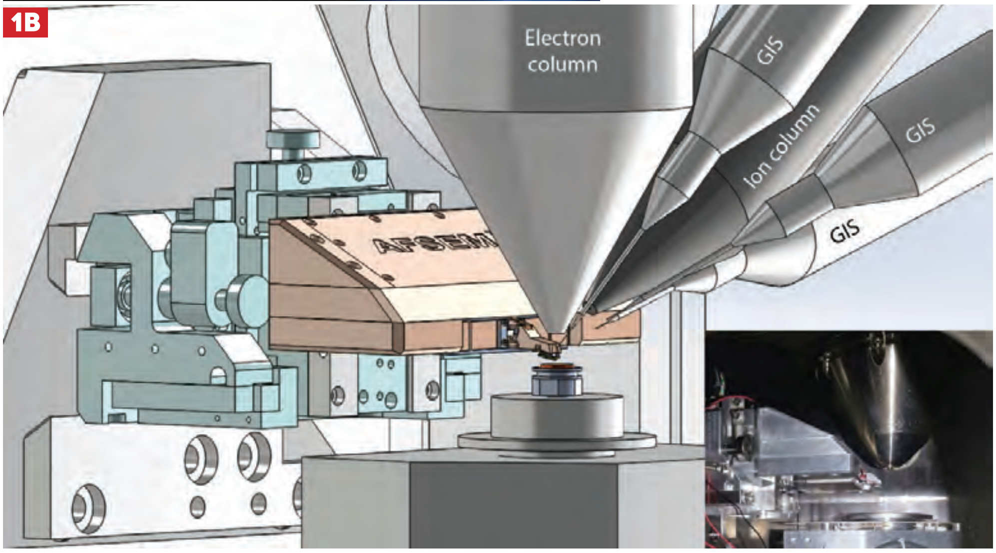


FIGURE 2A, above left, Example of a piezoresistive self-actuating probe manufactured by SCL Sensortech. These kinds of cantilevers operate through a piezo-resistive Wheatstone bridge which measures the strain through a change in resistivity under cantilever deflection
FIGURE 2B, above, Example of a specialized probe for conductive AFM (c-AFM) measurement where the cantilever has a gold/chromium track and 3D printing is used to deposit a pure metallic high-aspect needle probe on the end

FIGURE 1A, top, AFSEM™ module that fits directly into the SEM chamber
FIGURE 1B, above, Schematic of the instrument. There is unobstructed access to the electron beam above and to the right

be integrated with all main SEM manufacturers. This is in contrast to previous AFM-SEM systems that were collaborations between specific AFM and SEM vendors and thus could fit into a very limited subset of chambers. Moreover, the compatibility list includes conventional SEMs as well as field emission SEM and dual-beam FIB-SEM instruments.

The mechanical integration is SEM vendor-specific and involves mounting an adapter piece (shown in light gray in the schematic in Figure 1b) using screws or slots already present in the SEM door and stage. The instrument then seamlessly fits into this adapter piece. It does not require any modification to the SEM chamber (i.e. no new holes or drilling) so that any service contract for the SEM remains intact. The integrated instrument operates with two computers where the SEM uses its own computer and the AFM runs off a dedicated computer/controller system with the Nanosurf AFSEM software.

The tip-scanning design is key to providing maximum sample flexibility within the SEM chamber. This design is in contrast to a sample scanning design where the sample is scanned under the AFM tip, which limits the size of the sample that can be imaged. In addition, the cantilever tip itself represents the lowest physical point in the scanner design. Thus, with this setup, any sample that can fit into the SEM chamber can be imaged. The system design also permits tilting of the AFM in combination with the sample, a common feature employed by SEM users. Finally, similar to other modern stand-alone AFMs, this AFM is closed-loop in x and y utilizing novel MEMS position sensors[1] that provide accurate and distortion free AFM imaging and measurements in the x and y direction.

B: CANTILEVER TECHNOLOGY

As is the case for all AFM technology, the cantilever/tip (probe) assembly is the heart of the instrument through the myriad of ways it interacts with the sample. The common way to detect cantilever motion in AFM is through the optical beam deflection method where a laser is reflected off the back end of a cantilever and directed towards a position sensitive detector.

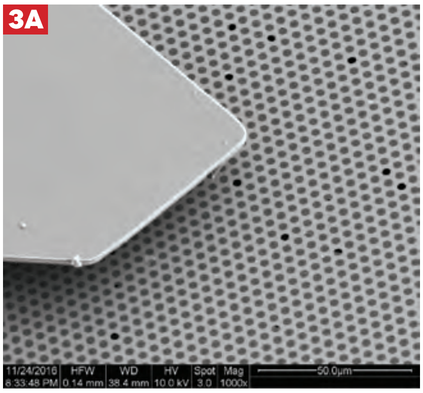
This method is accurate and sensitive. However, there are application areas where having a laser beam and detector apparatus placed above the cantilever is cumbersome or even impractical. An example of this is the integration of an AFM into an SEM where space is extremely limited and complicated laser alignment procedures are undesirable for the user.

Other cantilever technologies that do not rely on the optical beam deflection method have been developed since the early 1990's. A common alternative is piezoresistive self-sensing or self-actuated cantilevers where strain sensing elements are incorporated into the cantilever obviating the need for an external, space-consuming readout. These kinds of cantilevers operate through piezo-resistive strain-sensors connected in a Wheatstone bridge configuration which measures the strain-induced change of the sensors' resistivity when the cantilever is deflected. An example of this cantilever is shown in Figure 2a.

There are obvious advantages to employing this kind of cantilever technology in an integrated AFM/SEM. The primary advantage is that the area over the cantilever is unencumbered, leaving it accessible to the electron beam and other analysis techniques such as EDX. In addition, the ability to operate the AFM and cantilever without laser alignment enables a more facile user experience, especially when the cantilever is secured inside a vacuum chamber.

The piezoresistive probes designed for this instrument are fabricated in-house by GETec's sister company SCL-Sensortech. These highly sensitive self-sensing cantilevers have recently been shown to surpass standard optical beam deflection in low noise topographic imaging[2]. These cantilevers tend to be stiff (high spring constant) with application primarily to topographic imaging. Additionally, cantilever probes with a broad variety of resonant frequencies, spring constants, and tip-modifications are available for the AFSEM™.

Cantilevers for electrical property measurements through conductive AFM (c-AFM), for example, require



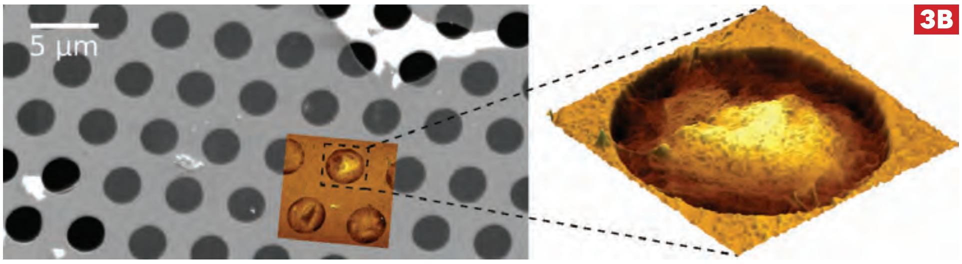


FIGURE 3B, above, An array of unsupported single and double layer graphene membranes visible with the SEM. The AFM has imaged an area covering several of these membranes in the orange square and provide high-resolution 3D info as shown in the zoomed in image on the right of one of the membranes displaying the sub-nanometer resolution capability

FIGURE 3A, above, The SEM screens a series of single and double layer graphene membranes. The AFM tip is then positioned at the region of interest to probe a specified area of interest further at higher resolution

FIGURE 4 Graphene membranes from Figure 3 imaged at different loads. With higher loads, the membranes are further mechanically displaced downwards. This displacement is quantified in the cross-sectional measurements below the images

specialized self-sensing cantilevers with a conductive coating. A chromium/gold track is laid down outlining the edges of the cantilever, as shown in Figure 2b. This track provides a connection between the tip and the PCB connector. Then, using a 3D nanoprinting method called focused electron-beam induced deposition (FEBID), high-aspect ratio pure metallic AFM tips are "printed" onto the self-sensing cantilevers. In this way, the probe provides a sharp, 10-nm diameter tip suitable for AFM imaging as shown in the inset in the top right of Figure 2b. These unique cantilevers are also supplied by SCL-Sensortech.

C: AFM CAPABILITY

Preserving all the powerful modes of AFM while integrating it into an SEM chamber has previously been a challenge. Either because of the scanner design or cantilever technology employed (such as optical fiber based probes or quartz tuning forks), the AFM portion often operates only with a limited subset of modes available to conventional AFM, especially in the realm of electrical characterization. By employing the piezoresistive cantilever technology, all major AFM imaging modes are now possible including major topographic imaging (dynamic/amplitude modulation mode and static/contact mode), force spectroscopy and phase imaging for nanomechanical measurements, and conductive AFM for electrical measurements.

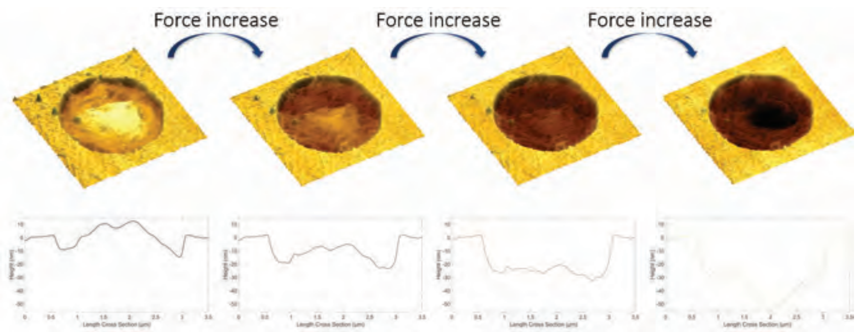
RESULTS AND DISCUSSION

A: COMBINING TWO WORLDS: LARGE AREA SCREENING SEM WITH LOCALIZED HIGH-RESOLUTION AFM

One of the most powerful applications of a combined AFM and SEM is the ability to use the SEM to quickly screen the surface for an area or feature of interest and then switch to AFM for high-resolution 3D imaging of that area.

While an SEM can image an area of 1-2 mm at low resolution very quickly at video frame rates, a medium-resolution AFM image takes on the order of 2-3 minutes to collect where the maximum scan size is ~100 µm for a large-sample instrument; the maximum scan size is 35 µm. Given these constraints, looking for a feature of interest with AFM is often like looking for a needle in a haystack as the user has to painstakingly image and then stitch together multiple areas at a time in order to cover a large surface area.

This integration is smooth and unobtrusive. The user identifies an area of interest with the SEM and analyzes this region with high resolution. There is no "re-positioning" from the SEM to the AFM; in other words, it is not a system that uses positioning actuators to move to the "same" position between the AFM and SEM. Instead, the AFM scans while actually under the electron beam such that the SEM views the AFM while the latter scans. At the same time, this also circumvents the need to expose the sample to air during the transfer from SEM to AFM, which is an important issue for air-sensitive samples (e.g. oxidizable surfaces).



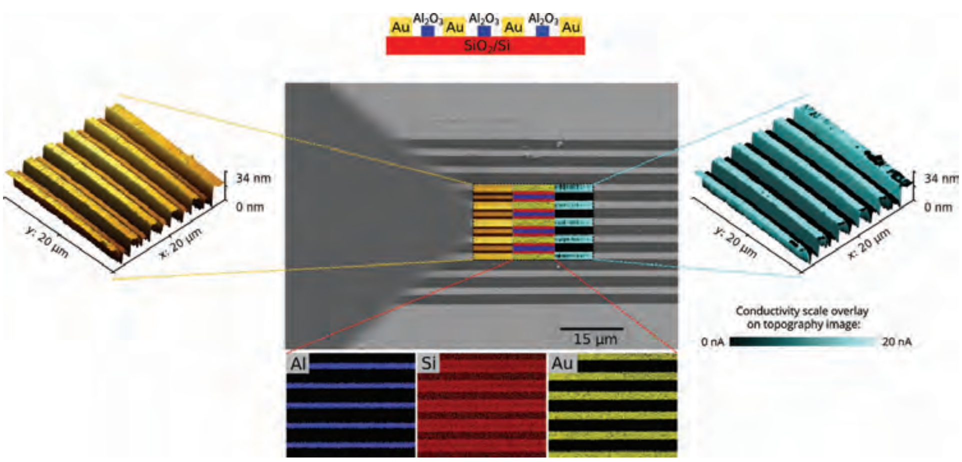


FIGURE 5 A periodic structure consisting of alternating gold and aluminum oxide steps. The SEM image is provided in the middle in gray tones. The EDX data of this structure is shown below, clearly delineating where the aluminum oxide and gold features are. The AFM topography measurement is displayed in the image on the left clearly showing alternating steps of various height. The conductivity image, collected by conductive AFM, is overlaid onto the 3D topography on the right revealing high conductivity on the gold and lower conductivity of the aluminum oxide and silicon oxide surface

AM

When conducting hybrid AFM/SEM measurements, it is important to keep in mind effects from beam-related contamination such as deposition of material (e.g. carbon) in the area that the beam has been scanning. High-resolution AFM measurements should precede high-resolution SEM measurements to ensure that the SEM beam contamination effects do not induce artifacts on the surface for the high-resolution AFM imaging. Low-resolution SEM scanning such as for screening applications minimize on beam contamination effects and should not pose a problem for subsequent AFM imaging.

An example of this dual-benefit from the AFSEM™ is described below in the nanomechanical study of unsupported graphene sheets. An array of unsupported graphene sheets was prepared and is shown below in the 140 µm x 140 µm SEM image in Figure 3a. Tilting the SEM stage during operation enables partial visibility of the AFM tip for convenient positioning. Based on the larger-scale SEM image, the AFM tip is precisely positioned for further AFM analysis as shown in Figure 3b. The inset in Figure 3b nicely illustrates the true 3D topography of the graphene membrane with sub-nm z-resolution, which is not accessible with the SEM

The AFM then imaged the displacement of these free-standing graphene layers as a function of different forces or loads in Figure 4 (where the load is controlled by the setpoint for the feedback loop). As can be seen in Figure 4, the graphene layers continue to “bend downwards” with a continually increasing force load. The graphene displacement is quantified in the cross-sections below where the displacement (or in this case indentation) of the graphene layer is measured relative to the reference material around the edge. In this way, nanomechanical properties of these layers can now be measured with nanometer precision.

B: REACHING THE FULL POTENTIAL: COMBINED SEM/EDX/AFM MEASUREMENTS

The full power of this hybrid instrument provides a complete visualization of the chemical, mechanical, electrical and topographical properties of the sample with nanoscale resolution. An example of this exciting hybrid characterization is shown in Figure 5 with the image of a patterned gold/aluminum-oxide sample on silicon. The SEM provides a quick overview scan of this patterned grating showing periodically spaced lines. However, we are not sure which part of this grating is aluminum-oxide and which is gold. We are also not sure of the exact heights of the gold and aluminum oxide features and of the electrical conductivity of the different sample regions.

All of these different properties are evaluated in one correlative measurement. First, the EDX measurement immediately shows that the dark striped regions in the SEM image are in fact the aluminum steps. There is a very thin space of silicon around the aluminum steps. The light gray features in the SEM image are the gold steps. We can then turn to the AFM for step thickness or height information. On the left in the yellow colored image is the topographic image where the alternative gold and aluminum-oxide steps are seen in the alternating yellow and orange steps. The yellow gold steps measure 30 nm high and the orange aluminum-oxide steps measure 15 nm high. Since a conductive self-sensing cantilever was used for AFM imaging, we can collect all information concerning conductivity simultaneously with the topography information. Conductivity overlaid onto topography is displayed on the right in the blue 3D image, so that light blue and black represent high and low conductivity, respectively. This conductivity image clearly shows that the light gray areas – corresponding to the gold regions from the EDX – not surprisingly have high conductivity while the interstitial areas represent the electrically insulating aluminum-oxide and silicon oxide surfaces.

SUMMARY AND CONCLUSIONS

The AFSEM™ offers a powerful integration of AFM and SEM capabilities that is compatible with instruments from all main SEM vendors. With an unobtrusive integration requiring no modifications to the SEM chamber, this system combines the best of the AFM and SEM worlds. It takes advantage of the SEM’s fast screening capabilities over large areas as well as its ability to provide chemically sensitive information through EDX. The AFM side is able to provide high-resolution nanoscale information on true 3D topography, materials contrast, and mechanical and electrical properties. By combining these two into one instrument, the best of both worlds provides a complete, high-resolution, and unparalleled visualization and understanding of the material surface.

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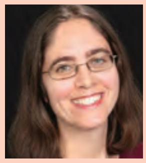
BIOGRAPHY



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ABSTRACT

We introduce the AFSEM™, a novel hybrid atomic force microscope – scanning electron microscope (AFM-SEM) that provides simultaneous acquisition of AFM and SEM measurements without compromising either the performance or power of either technique. The fast screening and chemical information from SEM and EDX are coupled with the topography and surface property characterization of the AFM to provide a complete understanding and visualization of the surface. The instrument is compatible with SEMs of all major SEM manufacturers requiring a simple insertion into the SEM door without any modifications to the SEM chamber. By taking advantage of high signal to noise piezoresistive AFM probes, the system provides a complete suite of AFM methods including topography, phase imaging, force spectroscopy, and electrical methods. This novel capability is applied to characterization of 2D materials and patterned nanostructures through the interactive combination of AFM, SEM and EDX.

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