

AFM Integrated with SEM and FIB: A Synergistic Union

Aaron Lewis,¹* A. Komissar,² A. Ignatov,² Oleg Fedoroyov,² E. Maayan,² Dalia Yablon,³ and H. Taha²

¹The Department of Applied Physics, Rachel and Selim Benin School of Computer Science and Engineering, The Hebrew University of Jerusalem, Jerusalem, Israel

²Nanonics Imaging Ltd, Har Hotzvim Hi Tech Park, 19 Hartum St., BYNET Bldg, Jerusalem, Israel

³SurfaceChar LLC, 8 Manns Hill Crescent, Sharon, MA 02067

*aaronl@savion.huji.ac.il

Introduction

Scanning electron microscopy (SEM) and ion beam milling techniques are mature nanoscale measurement technologies, whereas atomic force microscopy (AFM) is a developing technology generating intense interest in the scientific community for basic research and development. These techniques have generally existed in separate worlds. This article discusses a capability that marries these technologies through an instrument recently introduced by Nanonics, the 3TB4000.

Hybrid methodologies are becoming more important as research needs continue to push the development of characterization tools that provide a more complete understanding of materials. In combination with the growing and pervasive needs of nanotechnology, the use of hybrid methods to study material properties with nanoscale spatial resolution is at the forefront of instrumentation in both research laboratories and industry.

For the Nanonics 3TB4000, development focused both on instrumental compatibility and on the new research directions that such a combined tool could take for improved characterization of materials. The development of the Nanonics 3TB4000 has been a part of an integrated microscopy program, which included combining light optics with AFM [1], Raman chemical analysis and AFM [2], and mass spectroscopy with AFM functionality [3]. These technique combinations have resulted in unique capabilities unachievable with any of the above instruments alone. This article gives the reader a first glimpse at the emerging union of AFM with SEM/FIB.

Innovations in Instrument Design

The challenge has been, as in all such integrations, to maintain full functionality of each individual technique while at the same time enabling new materials characterization capabilities through integration that did not exist previously. Historically there have been several barriers to the effective merger of AFM methodologies with other microscopical or analytical techniques. This section describes the innovations in scanner, probe, and feedback loop technology and design to overcome these barriers.

Scanner. The AFM scanner controls the fine X, Y, and Z motion of either the sample or the tip in the instrument, depending on the instrument configuration. These scanners often employ cylindrical piezoelectric elements. These elements in the upright configuration possess limited Z range and a geometry that obstructs integration with an electron and ion beam. On the other hand, a cylindrical piezo device

permits large ranges of X and Y motion perpendicular to the long axis of the piezo. To this end Nanonics has developed a 3D UltraFlat Scanning stage with 4 cylindrical piezos (see schematic in Figure 1), enabling large ranges (85 μm or greater) in X, Y, and Z. This new stage allows for a variety of geometries of AFM systems in confined spaces such as electron and ion beam microscopes.

Probe. A second element that has limited hybrid tool advancement is the AFM probe. Specifically, the probe requires a design that enables the tip of the probe to be exposed to the electron/ion optical axis. Most AFM probes are manufactured from silicon-based materials that form an opaque barrier for visualization of the tip from above (Figure 2a). A few manufacturers do provide a geometry where the tip is slightly visible from the top (for example, AdvancedTecTM probes from Nanosensors); however, these probes are generally limited in their functional capabilities. Nanonics has developed the NanoToolKitTM of fused-silica-based probes with full visualization of the tip and full functionality for experiments, including electrical and thermal measurement [4], optical measurements [5], and material [6] and gas deposition. This enables new horizons of AFM combinations with electron/ion beams. A schematic of a fused silica probe with full visualization of the tip from the top is shown in Figure 2b.

Feedback loop. The final barrier to AFM/SEM/FIB integration has been the feedback loop that uses the standard reflection of laser light, bounced off the back of a cantilever and directed toward a position-sensitive detector, to measure

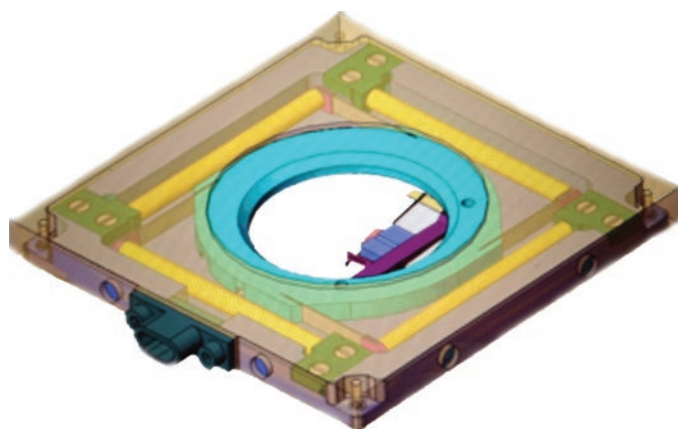


Figure 1: Schematic of an ultraflat scanner geometry design for confined spaces including electron and ion optical integration.

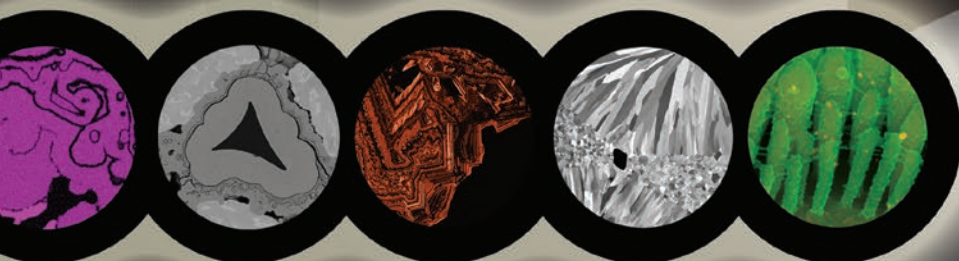
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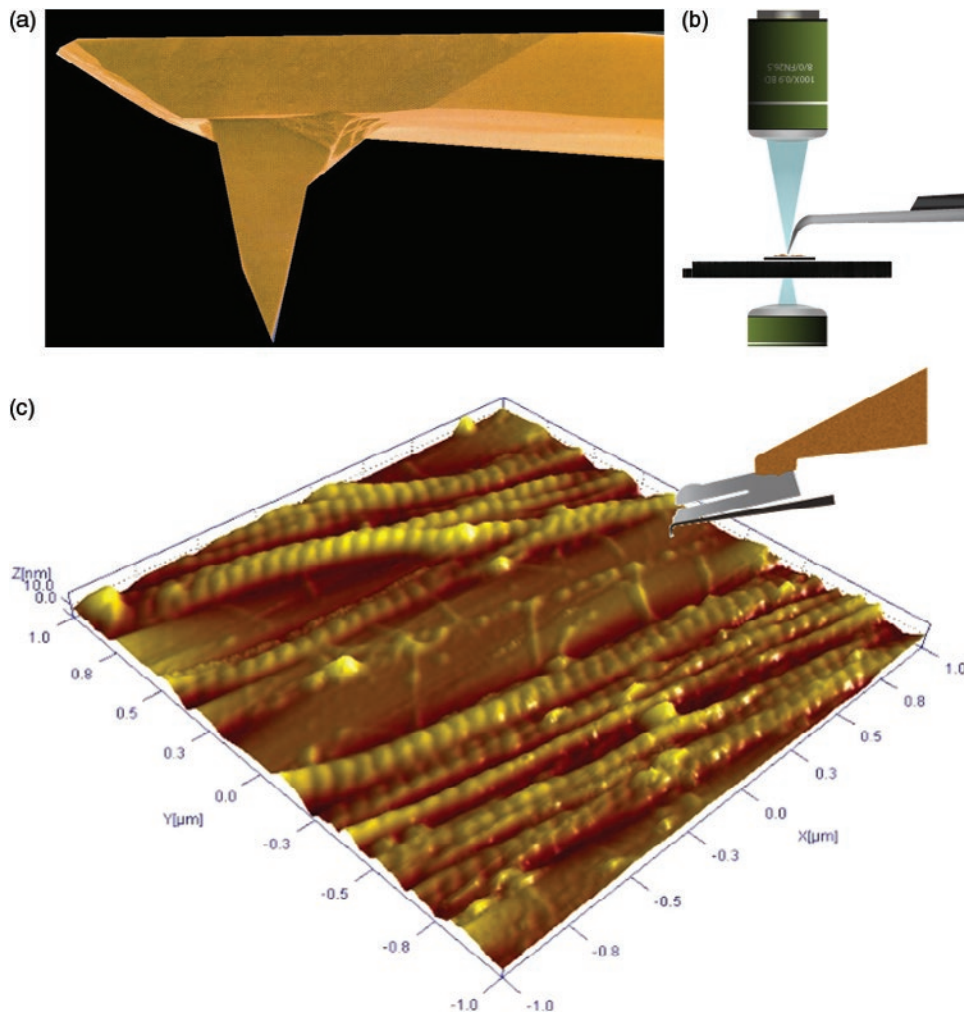


Figure 2: (a) Standard AFM probe geometry, (b) exposed tip geometry, and (c) exposed tip probe in a normal force tuning-fork geometry performing ultrasoft imaging of the protein collagen.

which is important to measure accurately in order to measure material elasticity. Tuning forks avoid these instabilities with very stiff cantilevers and thus have high Q , high sensitivity, and a narrow spectrum of oscillation frequencies, allowing the frequency rather than the amplitude to be the source of the feedback. Nonetheless, the amplitude can still be measured independently of the frequency feedback that is used. This gives the crucial point of contact without any calculation. This is a critical and essential element of AFM for the measurement of mechanical properties of materials, which no SEM or FIB can accomplish.

With this combination of ultra-thin scanners (Figure 2a), exposed-tip functional probes (Figure 2b), and normal force tuning forks with such probes (Figure 2c), the AFM hardware now allows unusual geometries with high sensitivity permitting new integrations with standard analytical tools such as SEM and FIB systems.

Results

The combination of these three capabilities into one instrument allows for new measurements not previously possible (Figure 3). We describe here a few examples exhibiting the 3TB4000's benefits and capabilities.

amplitude changes. This approach is so universally applied that it is rarely questioned. However, such an approach uses very soft cantilevers with low spring constants. When such cantilevers are oscillated in intermittent contact mode, the most common mode of non-destructive imaging in an AFM, they show a broad spectrum of oscillation frequencies with a low sensitivity or Q factor.

To obtain high Q factors one needs stiff cantilevers with a narrow spectrum of oscillation frequencies. Normal force tuning-fork-based geometries have been recognized as having stiff cantilevers with a sharp frequency spectrum [7]. Thus, the normal force tuning-fork-based geometry provides the ideal platform for the NanoToolKit™ of fused-silica-based probes. With such a combination of stiff cantilevers using tuning-fork-based feedback, the force of a photon has been measured to be 1.6 pN, which is a significant improvement over the best beam-bounce-based feedback results [8]. Furthermore, this combination overcomes the well-known instabilities in soft cantilevers, such as the jump to contact instability when a probe is approximately 20 nm from the surface or the adhesion ringing instability when a probe is lifted off a surface. These instabilities allow only an approximation of the point of tip-sample contact,

Improved field of view. One interminable problem with SPM technology is the limited field of view. Thus, an immediate improvement in this limitation of SPM technology is seen in Figure 4 where one is able to zoom into a specific ZnO nanowire from a field of 600 μm to 40 μm . This has recently allowed the measurement of the optical attenuation coefficient of a particular ZnO nanowire using the near-field optical capability of this hybrid technology [9].

Imaging an FIB milled trench. Figure 5a shows an AFM image profiling a 25- μm -deep trench with the large Z range of the system, both in terms of the scanner and the long exposed tip. The example in the figure is a trench milled in silicon with FIB. In this measurement, the FIB beam was used to mill this feature, and it was followed immediately by AFM imaging without having to remove the specimen from the FIB chamber and search for the feature of interest with the AFM. Furthermore, the true depth of the trench was measured by the AFM in a cross-sectional profile (Figure 5b), while a 3D reconstruction revealed the geometry of the sidewall (Figure 5c). Sidewall imaging with long and exposed tips becomes feasible with the 3TB4000 (see Figure 5c). The red arrow in this image clearly shows a Pt decoration on this

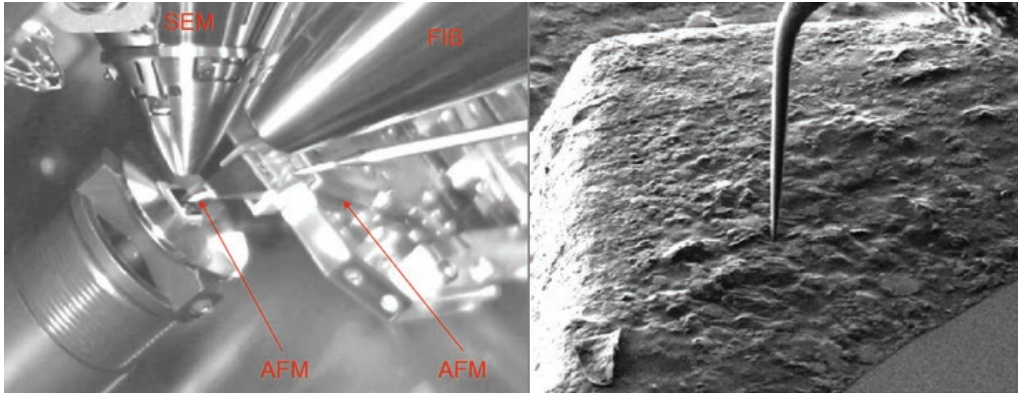


Figure 3: The 3TB4000 in position for on-line FIB nanofabrication and SEM imaging. (left) Location of AFM with respect to the SEM and FIB columns in the specimen chamber. (right) AFM probe tip imaged with the SEM. Image width = 210 μm .

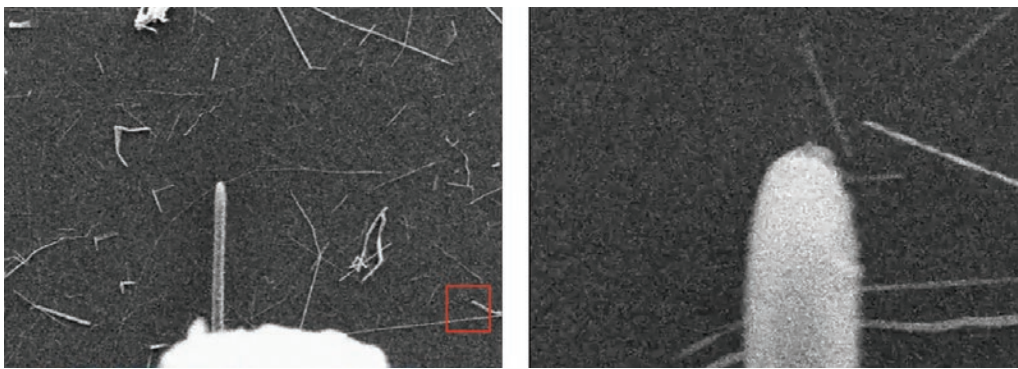


Figure 4: Zooming into a nanowire from a field of view of 600 μm to 40 μm . The boxed area in the left image is the zoomed area in the right image.

structure, and the white arrow shows an undercut. SEM is severely limited in such imaging tasks.

In situ milling measurements. The ability of AFM to quickly and easily follow FIB milling with high Z resolution provides a straightforward and convenient method for checking FIB milling results. This capability is demonstrated in Figure 6 where several FIB-milled features in Si were moni-

tored during the milling process. The grayscale SEM image in Figure 6a can easily locate these features and measure their widths but cannot provide direct information on their depths. The AFM delivers an equivalent top-view image (Figure 6b), a 3D reconstruction (Figure 6c), and a cross-sectional profile (Figure 6d) that together provide a true topographic map of specimen features. The depth profiles demonstrate the linearity of the etching process with time.

In situ measurements of AFM tip condition.

The benefits of AFM also can be seen in Figure 7 where the SEM image shows a fused silica AFM probe in contact with a surface under a controlled force. Figure 7 (left) shows that the AFM probe is in contact with the surface, depressed, and even deformed by this interaction. In Figure 7 (right), the probe has been removed from the surface, and so it has relaxed and recovered its

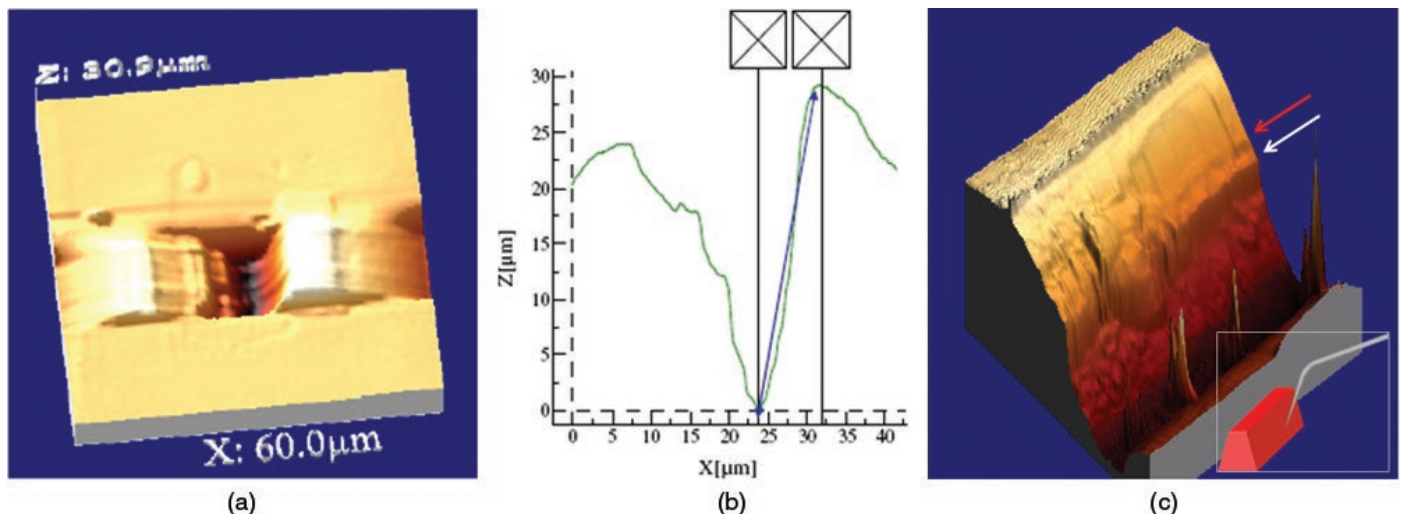


Figure 5: Exposed side walls in a deep trench FIB-milled in silicon. (a) AFM topographic image, (b) AFM Z-height profile, and (c) 3D image showing the geometry of the sidewall.

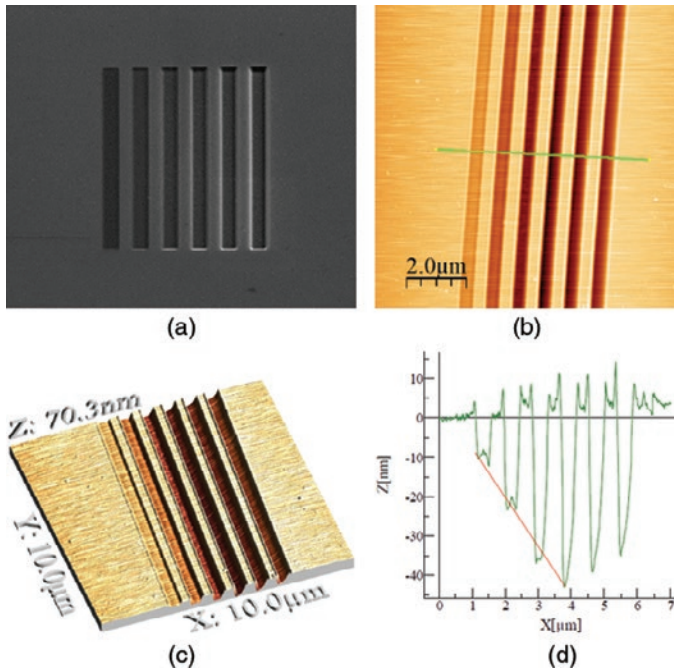


Figure 6: Features milled in a focused ion beam (FIB). (a) SEM image, (b) AFM image, image width = 10 μm, (c) 3D reconstruction showing the cross-section, (d) cross-sectional profile along green line in (b) demonstrating the linearity of the etching process with time.

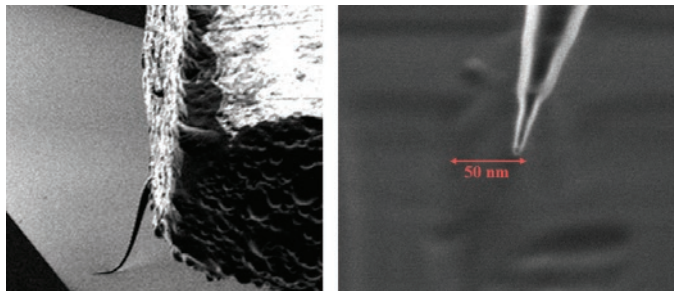


Figure 7: Monitoring the AFM tip with the SEM. (left) An AFM tip in contact with a surface, (right) assessment of tip condition with the SEM.

interaction with the surface. Furthermore, the lateral resolution in AFM is the convolution of the probe tip geometry with the sample geometry; this can now be directly assessed on-line at high resolution.

If the tip has been contaminated during the imaging process, the FIB can be used to remove the contamination, and both the FIB and the on-line SEM can be employed to see the results of the cleaning. Figure 8 shows such a cleaning through imaging in top down images before and after cleaning (8a and 8b) and side view images before and after cleaning (8c and 8d).

Material property measurements. In addition to important imaging tasks, the AFM is capable of measuring various properties of materials. One of these features, which is especially relevant in SEM and FIB, is the measurement of the elasticity within hybrid structures. An example of this measurement is shown in Figure 9 where the SEM was used to guide the AFM probe (Figure 9a). The AFM measures a lower elasticity in soft copper metal, which differentiates

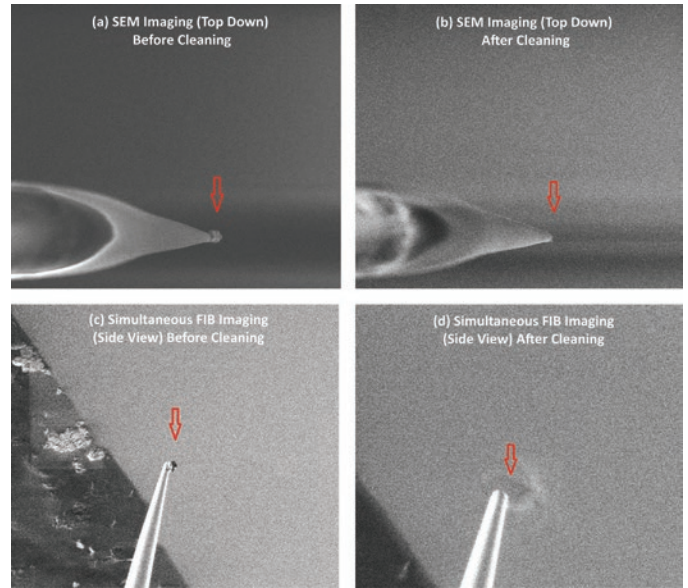


Figure 8: Cleaning a contaminated probe. Top-down image (a) before and (b) after cleaning (image width = 43 μm). Side view images (c) before and (d) after cleaning (image width = 59 μm).

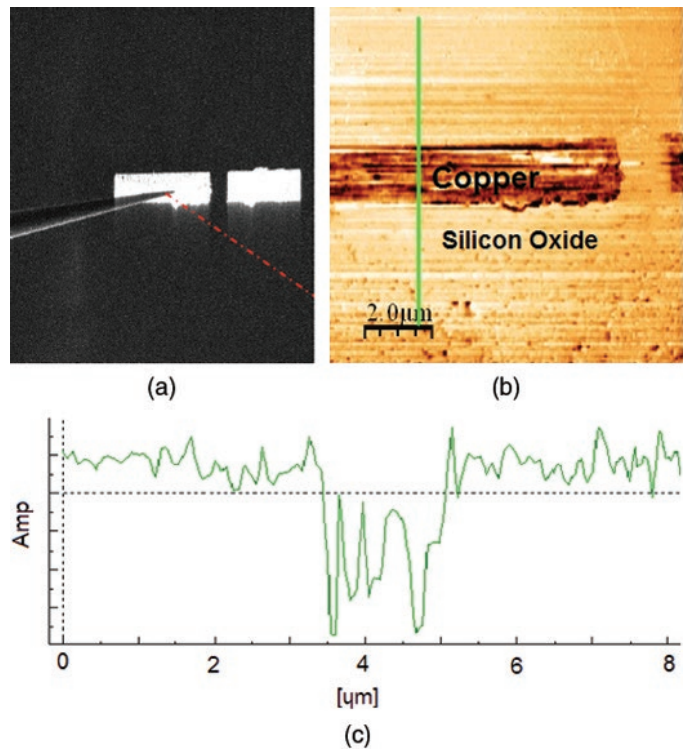


Figure 9: Imaging the elasticity of copper on silicon. (a) SEM used to locate measurement point, (b) AFM image distinguishes soft Cu (red) from stiff silica.

it from the surrounding stiffer silicon oxide in Figure 9b. The softer copper and the harder silica also have the effect of a trampoline on the probe; the probe experiences larger amplitudes on the harder surface. The line profile of the amplitude in Figure 9c was taken from the green cross-sectional profile in Figure 9b. The tuning fork in normal force mode has amplitude as a measured parameter that is independent of the feedback. With such probes one can

effectively monitor mechanical damping on-line without the need for digitization or further analysis of the intermittent contact mode amplitudes where the probe is contacting the surface at some defined frequency.

Monitoring nanofabrication processes. The FIB is increasingly used for nanofabrication with the requirement for accurate Z control. The ability of an on-line AFM is useful in defining the effectiveness of such fabrication processes.

Conclusion

The Nanonics 3TB4000 incorporates innovations in the design and technology of atomic force microscopy enabling its integration with SEM and FIB technologies into a single powerful capability. Innovations in scanner design, probe manufacturing, and feedback loop have enabled this useful integration, resulting in a tool more powerful than its individual components. Several examples are shown where the SEM, FIB, and AFM are used together to provide complimentary information about the material. This will likely lead to the next generation of 3D functional analysis of devices, highlighting the disruptive nature of this technological synergism.

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