# Topography, Phase Imaging, and Mechanical Property Investigation of Polyester Yarn Interaction with Silicone Gel Matrix

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**Abstract:** To better inform materials design strategies, it is desirable to use a technique capable of measuring both sample topography and mechanical properties. A composite of polyester yarn embedded in silicone gel was examined with a commercial atomic force microscope (AFM). Non-contact AFM was used for topography and phase imaging. Force-distance spectroscopy plus force-volume mapping was used for mechanical property characterization. The hardness of the yarn was revealed to be about 100 times greater than the gel it is embedded in. This investigation reflects the effectiveness of the AFM in exploring desirable macroscopic traits in samples.

## Introduction

The addition of mechanical property characterization to textile development can yield next-generation fabrics for new applications. Understanding the characteristics of the materials being integrated with existing fabric matrices is paramount in predicting how targeted enhanced properties will manifest in novel composites. For example, weaving fibers into tear-resistant fabric can significantly increase the fabric's strength without seriously increasing its weight. The aim for next-generation fabrics is to emulate the durability and weight advantages of fiber-reinforced plastics, within the domain of the textile industry, by looking for solutions at a much smaller scale. Atomic force microscopy (AFM) can be used to study the interactions between the fibers and the fabric matrix, their interfacial regions, and the composite's topography—all points of knowledge helpful in adjusting the product for maximum performance gain while minimizing production cost. This article describes how AFM was used to characterize the hardness, measured in newtons per meter, at multiple sites of a polyester fiber interaction with a silicone gel matrix.

## Materials and Methods

A silicone gel matrix sample was embedded with strands of polyester yarn. This composite combines several layers that mimic abdominal wall tissue of animals and is used for surgical training [1]. Characterization by AFM was conducted on that sample under ambient air conditions using a Park NX10 SPM system in non-contact AFM mode for topographic imaging and phase imaging. Both sets of data were acquired simultaneously. Topographical imaging is useful primarily for observation of three-dimensional (3D) features on the surface of the sample, while phase imaging yields data that can be correlated to the sample's elastic properties. The mechanical properties of the sample were characterized primarily using force-distance spectroscopy and force-volume mapping. These results were then correlated with data acquired by phase imaging.

**Force-volume mapping.** In force-distance spectroscopy, force-distance (f-d) curves are used to measure the force that an AFM probe applies vertically to a single point on a sample surface. Force-distance curves are plots of the cantilever's deflection, as measured by a position-sensitive photodetector, versus the extension of a piezoelectric scanner [2]. Force-volume mapping builds on f-d spectroscopy in that it takes an array of single measurement points and turns the collected f-d curves across the sample surface into a two-dimensional (2D) characterization map of hardness [2]. Hardness is defined as the slope of the f-d curve given in units of newtons per meter (N/m). Although the Park NX10 SPM system is capable of a 40 nm spatial resolution [3], a 300 nm spatial resolution was used in the interest of time. The system is also sensitive to changes in force as little as 60 pN and can detect up to 100 μN depending on the spring constant of the cantilever used.

**Phase imaging.** Phase imaging is an AFM technique that makes use of the shift in a cantilever's oscillation as its tip moves across different features on the sample. The difference in the input signal for tip oscillation versus the ensuing oscillation output signal is referred to as a shift in phase [4]. This particular signal can be correlated to several material properties such as elasticity.

## Results

Figure 1 shows a cross-section view of the silicone matrix (orange-red in color) with a strand of polyester yarn in

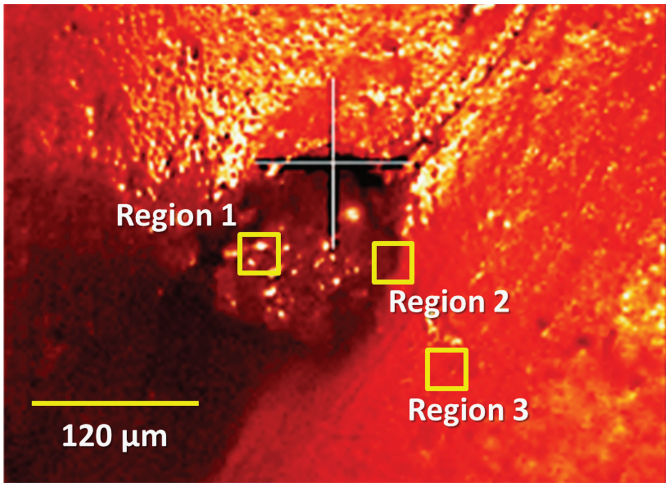


**Figure 1:** AFM system built-in light optical camera image of a polyester fiber embedded within a silicone gel matrix. Three regions on this sample were selected for hardness characterization: Region 1 = the fiber, Region 2 = the interfacial area between the fiber and gel, and Region 3 = the silicone gel matrix.




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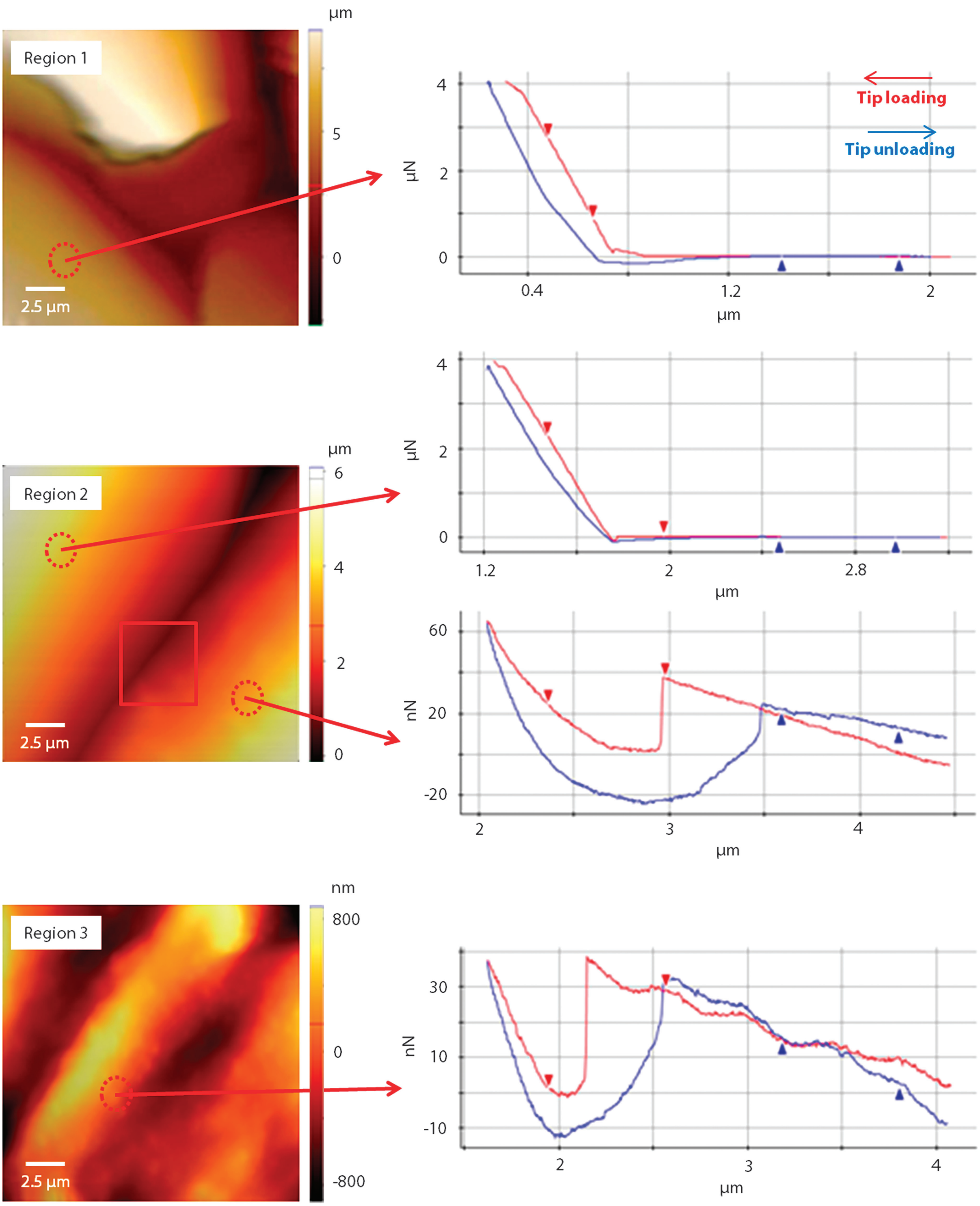


**Figure 2:** AFM topographical images (left) and corresponding f-d curves (right) from the three regions indicated in Figure 1. Force-distance curves were taken at the sample sites indicated by the dotted red circles in each AFM topographical image. The red inset square in Region 2 was selected for further force-volume and hardness mapping in Figure 3. In Regions 1 and 2 (upper), at sites corresponding to the polyester fiber, the change of the force load's slope is two orders of magnitude larger than in sites corresponding to the silicone gel in Region 2 (lower) and Region 3.

the center (dark circular feature, 100–150 µm in diameter). Here three regions were selected for investigation. Region 1 is situated directly on the exposed strand of embedded polyester yarn. Region 2 is the interface between the embedded polyester yarn and the silicone matrix surrounding it. Region 3 is an area composed entirely of the silicone matrix.

**Load vs. distance data.** Figure 2 shows non-contact AFM topographic images taken from each of the regions selected in

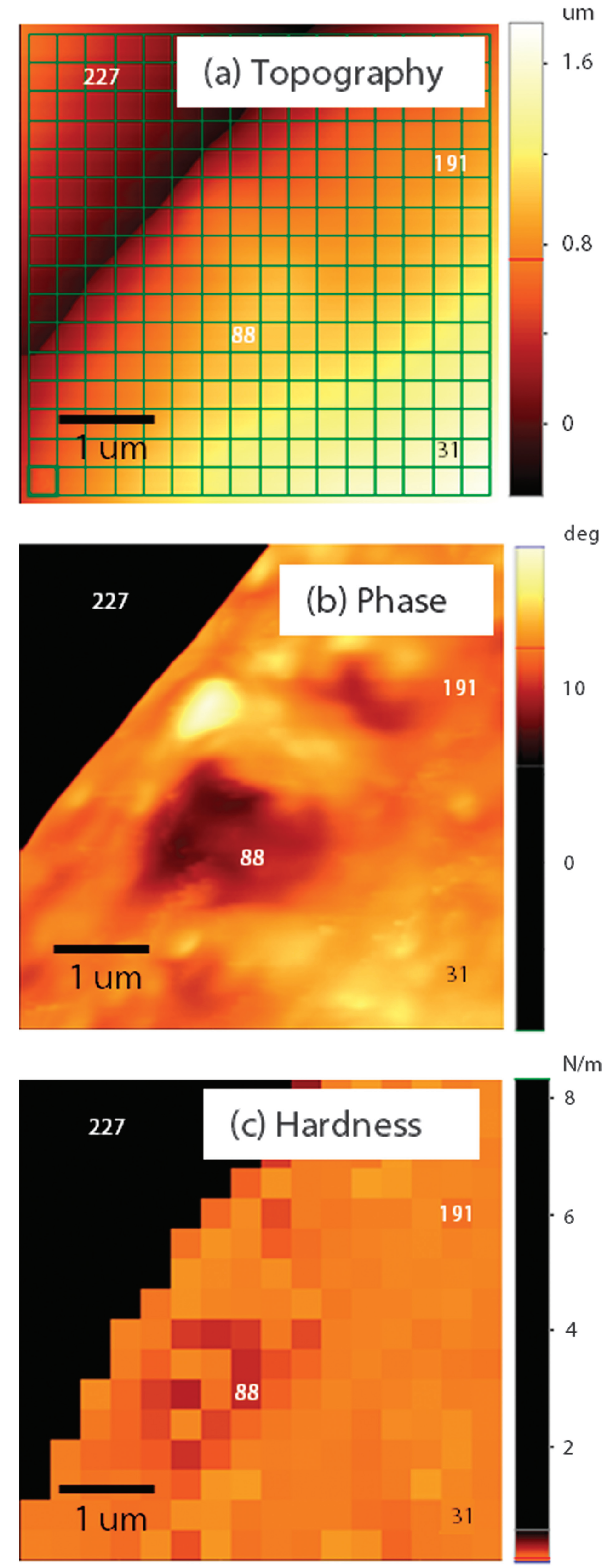


Figure 3: The topography-derived AFM force-volume mapping array (a), phase image (b), and hardness map (c) of the sample's interfacial region. Note the visual correlation between the phase image and hardness map.

Figure 1 as well as f-d curves measured at each of those regions. The shape of the f-d curves indicates the physical interaction between the tip and sample; in this case, the force load on the cantilever tip vs. the tip-sample distance. The slope of the f-d curve is steeper when the tip presses onto a harder sample.

Region 1 of Figure 1, a location on the polyester yarn fiber, was selected for f-d spectroscopy. The f-d curve at this site reveals that the force applied by the tip to the sample increases by about 4 µN over a distance of 0.5 µm as the tip pushes down onto the surface (Figure 2). In Region 2, two sites were measured at the sample's interfacial region: the first site was in the left half of the image (the polyester yarn), and a second site was in the right half of the image (the silicone matrix). The f-d curve for the polyester yarn side of the interfacial region again exhibits a force of about 4 µN being applied by the tip to the sample over a distance of 0.5 µm. The f-d curve at the site corresponding to the silicone matrix portion of the region yields different values. An initial increase of just under 40 nN in the force applied by the tip to the sample occurs over a distance of approximately 1.25 µm, at which point a steep drop-off of about 40 nN in the tip-applied force was observed. It is possible that the drop-off in applied force is due to the tip being pulled onto the surface of the silicone matrix. A second increase in tip-applied force occurs as the probe continues pressing onto the matrix. Here the observed increase in force is measured to be about 50 nN over a distance of 0.5 µm. In Region 3, a location on the sample's silicone matrix, we observed a two-stage force load increase similar to the one exhibited by the silicone matrix half of Region 2. The initial tip-applied force builds up to about 40 nN over a distance of 1.5 µm just before plummeting to about 0 nN. Again, this sudden decrease in applied force is speculated to be the moment in which the tip has snapped onto the surface of the silicone matrix. Shortly thereafter, a second increase in tip-applied force is observed as the probe continues to be pushed down onto the matrix resulting in a load increase of about again 40 nN over a distance of about 0.5 µm. In this investigation, f-d curves taken from a region within a strand of polyester yarn show that the force load on the cantilever tip is approximately 100 times greater than the load shown on an f-d curve from the silicone matrix at similar tip-sample distances.

**Force-volume mapping.** In order to increase the area of observation from single points to whole sections of the sample's surface, force-volume mapping was applied as depicted in Figure 3a. This technique produces a 2D map of a mechanical property—in this case, hardness (Figure 3c). To begin, a small area within Region 2 (Figure 1) was selected for hardness mapping (the interfacial region). Here we can map effects on both the polyester yarn and the silicone matrix in one map. This location is indicated by the inset red square in the non-contact AFM topography image for Region 2 (Figure 2). The area for force-volume mapping measured 5 µm × 5 µm and was the same for all the images of Figure 3. A 16 × 16 grid was placed over the location to be mapped, creating an array of 256 sites. Force-distance curves were measured at the midpoint of each of these sites, and the data were translated into a 256-pixel hardness map where each pixel represents the sample hardness detected at the midpoint of each site. Note the sharp difference in the color

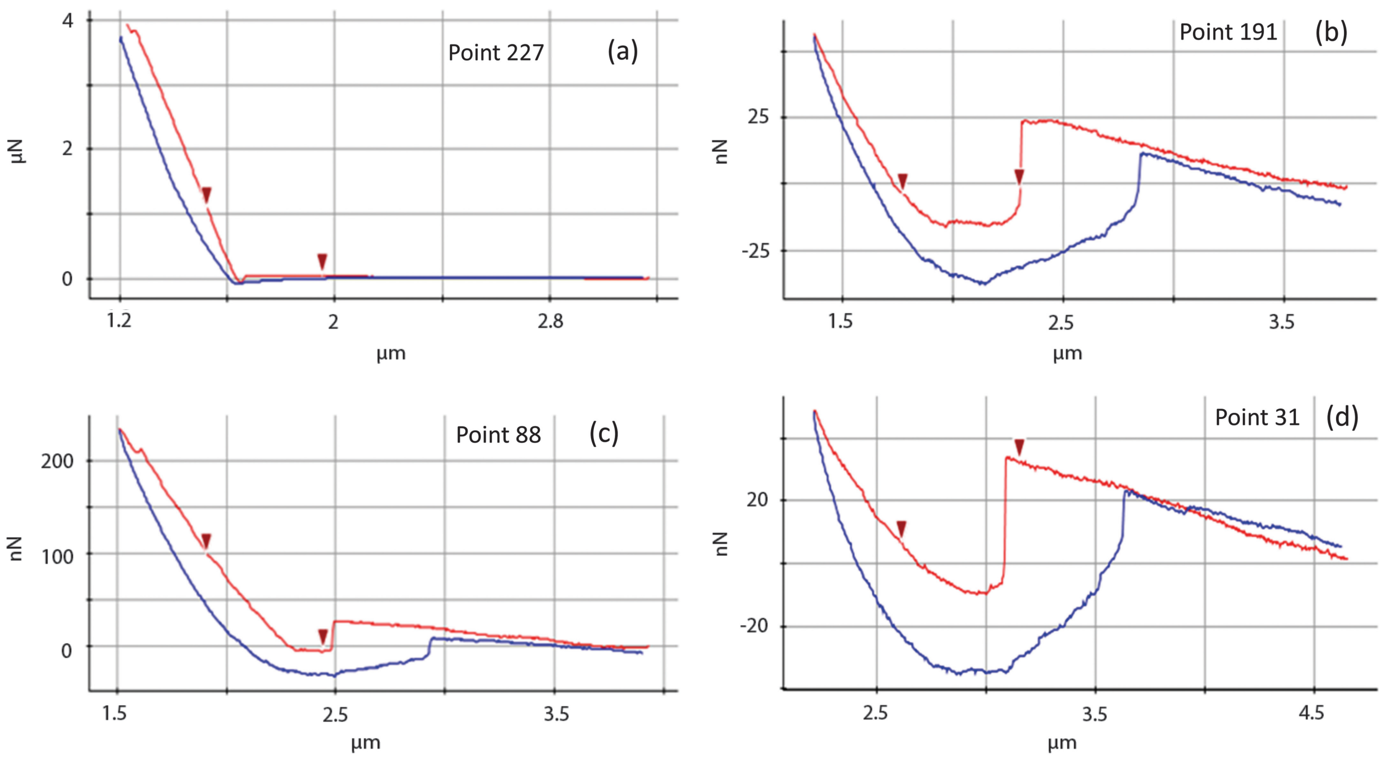


**Figure 4:** Force-distance curves of targeted points from the hardness map of the sample's interfacial region. The tip-applied forces measured at the polyester fiber at Point 227 (a) are about two orders of magnitude greater than forces measured at silicone gel matrix sites at Points 191 (b), 88 (c), and 31 (d).

of the pixels in the hardness map across the field of view, which closely follows the border of the interfacial region between the polyester yarn and the silicone matrix. A similar phenomenon was observed in the phase image—the upper-left portion of the phase image corresponds to the embedded yarn and has a different phase signal (black, no signal) from the remaining portion of the image depicting the silicone matrix. This indicates that phase shifts in the cantilever's oscillation are markedly different in the yarn compared to the matrix, suggesting a difference in material elasticity. While it is premature to directly correlate coloration differences in the phase imaging to directly identifying differences in sample hardness, the visual similarities in the hardness map and phase image are noteworthy and may warrant further study.

**Force-distance curves at specific pixels.** The final leg of the investigation repeated the f-d curve comparison that was depicted in Figure 2; however, this time the sites selected for analysis were exclusive to the interfacial region (Figure 3). Four sites were selected from the array of 256 pixels shown on the topography image in Figure 3a. These sites are referred to as sites 31, 88, 191, and 227. The first three sites were located within the silicone gel matrix area of the interfacial zone, whereas site 227 was located on the polyester yarn. Figure 4 shows the f-d curves for each of the four sites selected. The f-d curve of site 227 is shaped as expected given our previous data for Region 1 (polyester yarn) shown in Figure 2. Again, a force load of about 4 μN over a distance of about 0.5 μm is observed. Sites 31, 88, and 191 have f-d curves that were anticipated from the measurements on Region 3 (silicone matrix) of Figure 2. The second force load increase is again measured to be around 40 nN over a distance of 0.5 μm. This data indicate that the polyester fiber is around two orders of magnitude harder (in N/m) than the silicone gel matrix. Thus, these 2D mapping data are consistent with the single-point f-d spectroscopy data acquired earlier in the investigation.

## Discussion

Given that the fibers of polyester yarn have been observed here to be around 100 times harder than the gel matrix in which they are embedded, it is reasonable to conclude that the gel's resistance to certain types of damage has been positively augmented by the embedded yarn. The mechanical property examined during this investigation was that of hardness. In fiber-reinforced plastics, embedded fibers allow the novel composite to remain in one piece despite having a large force applied to it. Likewise, it is possible the embedded fibers in the investigated composite material have positively augmented the matrix's damage resistance; further study is required. A fiber specifically selected for its hardness may conceivably increase a composite fabric's damage resistance leading to immediate uses in ballistics as well as other applications that require clothing with heightened durability. Also the novel composite here potentially has significant weight savings when compared to the material it is being designed to replace. When applied to the domain of textiles and apparel, fibers such as the ones investigated here can be embedded in more than just gel matrices and have been woven into existing fabrics such as cotton to confer traits such as increased aerosol filtration [5].

## Conclusion

Topographic imaging, phase imaging, and mechanical property mapping (based on f-d curve data) of a silicone gel and polyester yarn sample were acquired. The data collected reveal that the strands of polyester yarn are approximately 100 times harder than the silicone gel matrix they are embedded in. All data acquisition was performed with the forces on the order of nano-newtons observed across distances of a few micrometers. Performing measurements at this scale is an effective demonstration of AFM's capability to characterize key properties of materials used in novel composites such as next-generation fabrics. Understanding of the behavior of such materials enables educated speculation on the enhancement of properties of the composite material.

## References


[1] SurgiReal, "Our Team." http://www.surgireal.com/our-team (accessed September 19, 2016).

[2] Park Systems, "Park AFM Nanomechanical Mode," http://www.parkafm.com/index.php/park-afm-modes/nanomechanical-modes (accessed April 22, 2016).

[3] M Hong et al., *MRS Bull* 41 (2016) 512–13.

[4] "Phase Imaging" in *Springer Handbook of Experimental Solid Mechanics*, ed. WN Sharpe, Springer Science & Business Media, New York, 2006, p. 430, section 17.3.3.

[5] "Nanofibers as Filters" in *Intelligent Textiles and Clothing for Ballistic and NBC Protection: Technology at the Cutting Edge*, eds. P Kiekens and S Jayaraman, Springer Science & Business Media, New York, 2012, p. 210, section 10.3.


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