Nanoindenting, Scratching, and Wear Testing *with the Atomic Force Microscope*

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Introduction

Nanoindentation is an option for Digital Instruments MultiMode® and Dimension™ Series Scanning Probe Microscopes (SPMs). Using TappingMode™ AFM and an AFM diamond tip mounted to a metal-foil cantilever, you can indent a surface and immediately image the indentation. This eliminates the need to move the sample, switch AFM tips, relocate the indenting area for scanning, or use an entirely different instrument to image the indentation. Although indentation cantilevers have higher spring constants than typical imaging cantilevers, it is still possible to image soft samples with relatively low forces. This is possible using TappingMode AFM, which requires less force to image a sample than does contact mode AFM. The diamond tips are sufficiently sharp to provide good image resolution.

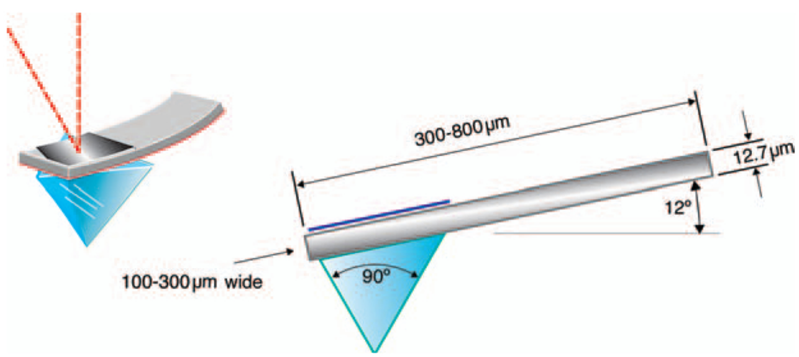

Figure 1. Typical indentation cantilever.

The same cantilever/tip also allows scratching and wear tests. This option allows characterization of mechanical properties of thin films, such as diamond-like carbon, using indentation to investigate hardness, and scratching or wear testing to investigate film adhesion and durability. Recent studies have been done on chemical mechanical polishing (post CMP) wafers, polymers, and biological samples such as bovine and human sperm nuclei.

How It Works

The indentation process is straightforward. First, engage on the surface in TappingMode AFM using an indentation cantilever. While scanning, the sample surface may be imaged to find the desired location for the indentation. Once the area of interest is located, select Indentation Mode; this halts the lateral scanning of the cantilever and lifts the tip slightly off the surface. Then, select the desired indentation parameters and execute the indentation. The cantilever oscillation is now turned off, and the scanner lowers the diamond tip towards the surface where the tip is forced into the sample surface until the cantilever deflects a specified amount. The tip is then retracted until it reaches the initial position above the surface. While indenting, a force displacement curve is recorded utilizing the DC displacement of the cantilever versus the extension of the scanner. Many indentations may be made using various forces, rates, etc. Upon exiting indentation mode, TappingMode AFM is turned back on and the instrument returns to imaging mode; the indentations made on the surface may then be imaged.

Scratching is done in a similar manner. First, engage on the surface with an indentation cantilever and image the surface to locate the desired position for the scratch. Next, select Scratch Mode where, once again, the lateral scanning halts and the tip is lifted slightly off the surface. Then, set the scratch parameters, such as force, rate, scratch length and direction, and the scratch is executed. During the scratching, the cantilever oscillation is turned off, the tip is forced into the surface until the specified cantilever deflection is reached, and the tip is then moved laterally according to the preset length, rate, and direction. Multiple scratches may be made using various forces, lengths, rates, and directions. Upon exiting Scratch Mode, TappingMode AFM is turned back on, and the instrument is ready to image the scratches made on the surface of the sample.

Indentation Cantilevers

Indentation cantilevers are thicker, wider, and longer than standard AFM cantilevers, and are composed of stainless steel. The typical range for spring constants of contact mode, TappingMode, and indentation cantilevers are 0.01-1.0N/m, 20-100N/m, and 100-300N/m respectively. The fundamental resonance frequency for indentation cantilevers is in the range of 35-60kHz, depending on the dimensions of the cantilever and the

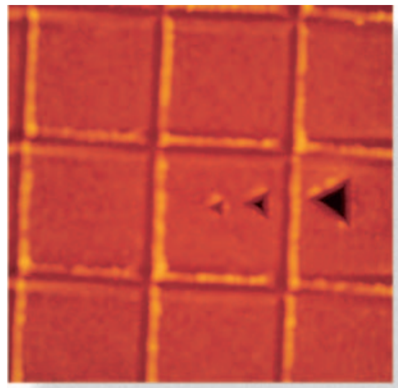

Figure 2. Indentations on a 1μm gold ruling at various forces used for testing the sharpness and orientation of the diamond tips mounted on the indentation cantilevers. 3μm scan.

size of the diamond. For comparison, the resonant frequency for standard TappingMode AFM cantilevers is usually about 300kHz.

A typical indentation cantilever has a spring constant of 150N/m; length, width, and thickness of about 350, 100, and 13μm, respectively (Figure 1); and a resonant frequency of 50kHz. Veeco Probes measures and supplies the customer with the spring constant of each cantilever purchased. The typical indentation force range available with our instruments is 1-100μN with resolution better than 0.5μN. Larger forces up to about 300μN can be accommodated using custom cantilevers with higher spring constants.

The diamond tip mounted to the end of the cantilever has a tip radius that is less than 25nm to assure good imaging resolution and nanometer scale indents and scratches. The diamond tip is the apex of three sides of a four-sided pyramid with an apex angle of about 90 degrees. To provide symmetric indents, the diamond is mounted such that the vertical axis of the pyramid is approximately normal to the sample surface when mounted on the microscope.

Unlike contact mode, our patented TappingMode technique allows use of the high spring-constant cantilevers required for nanoindentation, while still imaging the surface with minimal damage. This is illustrated by the indentations made in the 1μm gold ruling shown in Figure 2. The indentation cantilever that was used to indent and image the surface has a particularly large spring constant of about 400N/m. If this cantilever were used to image the gold in contact

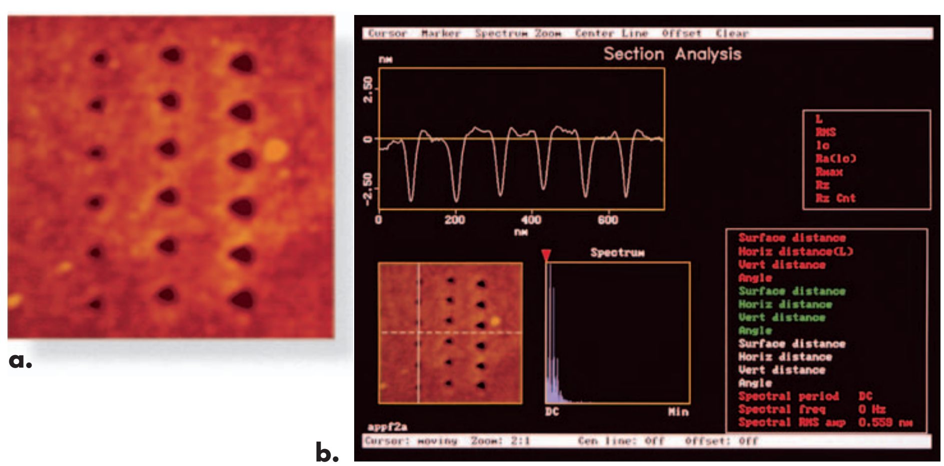

Figure 3. (a) Indentations on a 15nm thick diamond-like carbon (dlc) thin film using three forces of about 15, 20, and 25μN respectively. All indentations are less than 10nm deep. The smallest indentations are approximately 3nm deep as seen in the section view (b). 800nm scan.

mode, it would have damaged the surface sufficiently to prevent meaningful imaging of either the indentations or the rulings. Veeco Probes also provides an image of a gold ruling, similar to Figure 2, for each indentation cantilever sold. The indents and the image are captured with the same cantilever that is sold to the customer.

Examples

Nanoindentation with an AFM cantilever is useful for investigating the hardness of films as thin as 5nm. Since the macroscopic properties of thin films of a material may be different from the microscopic properties, the ability to perform tests at the nanometer level is of great importance in many processes. The 15nm thick diamond-like carbon (dlc) film in Figure 3a was indented five times with three different forces: about 15, 20, and 25μN, respectively. The indentations were then imaged with the same tip, which did the indenting. In the cross-section image (Figure 3b), the depth of the 15μN indentations (measured after any elastic recovery of the film has occurred) is only about 3nm. The projected area of these indentations is approximately 1,600nm^2. The 20 and 25μN indentations have depths of about 5nm and 7nm and projected areas of about 2,000 and 2,500nm^2, respectively. Given the small size of these indentations, the ability to image immediately with the same instrument and tip is a major benefit.

Using indentation cantilevers, it is possible to indent various samples with the same force in order to compare hardness. Figure 4 shows two different dlc thin films that were indented four times with three different forces of 23, 34, and 45μN. The same forces were used for both films by using the same indentation cantilever under the same conditions. The indentations on the left film are smaller than the ones on the right film. For the film on the left, the average indentation depth for each of the forces used is about 1.5, 3, and 6nm, respectively. For the film on the right, the indentations are about twice as deep: 3, 7, and 13nm. The images of the two films also show a difference in the way that the material has piled up around the indentations.

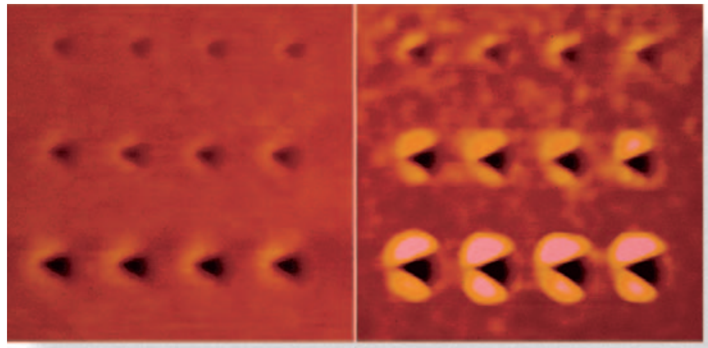

Figure 4. Indentations on two different diamond-like carbon thin films using three different forces (23, 34, and 45μN) with four indents made at each force. Each film was indented using the same forces and cantilever in order to compare hardness. 500nm scans.

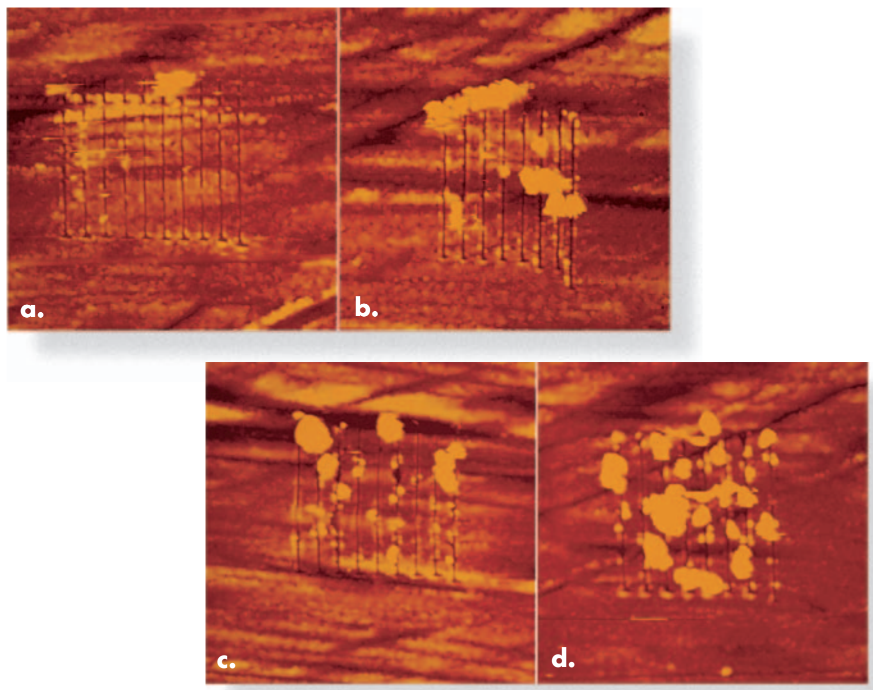

Figure 5. Array of scratches performed on four different 10nm thick diamond-like carbon thin films, all using the same force and cantilever in order to compare film adhesion and durability. The scratches are 1μm long and less than 10nm deep. 2μm scans.

Properties such as film adhesion and durability can also be studied using indentation cantilevers. Figure 5 shows four different 10nm thick dlc films (coated on computer hard disks) on which a series of scratches were made at a force of 15μN. The average depth of the scratches for each film is 3, 6, 6, and 3nm for Figures 5a to 5d, respectively. The material deposits, which have piled up near the scratches are likely peels or shavings

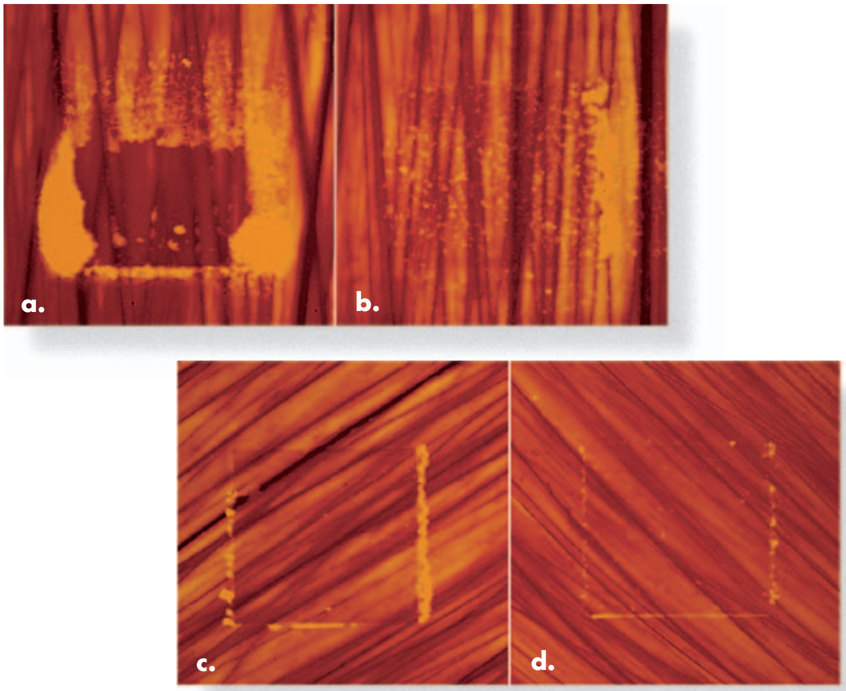

Figure 6. Worn areas on four different 10nm thick diamond-like carbon thin films generated using the same force and cantilever in order to compare film adhesion and durability. The worn areas are 5μm square. 9μm scans.

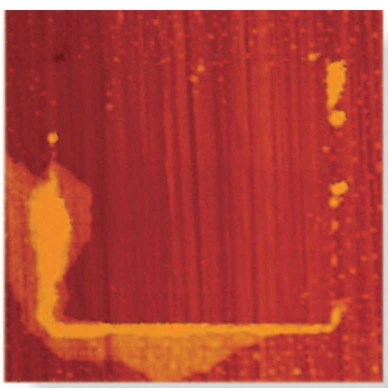

Figure 7. Worn area on a diamond-like carbon thin film in which the film delaminated from the substrate. The worn area is 2μm square. 3μm scan.

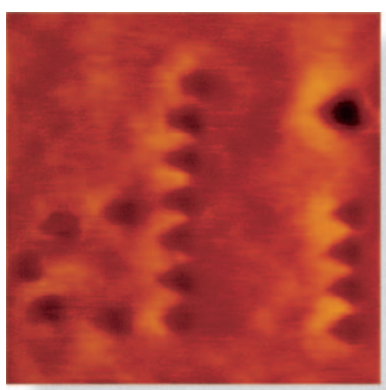

Figure 8. Digital Instruments logo indented on a 15nm thick diamond-like carbon thin film. The small indents are 1nm deep and the large indent is about 2.5nm deep. The forces used for the indentations were about 5μN and 10μN respectively. 250nm scan.

of the film, which were scratched away from the surface. The films may also have delaminated from the substrates causing taller features (brighter colors) in the images.

The cantilevers used for nanoindentation and scratching can also be used for wear testing. Figure 6 shows four different dlc films (coated on computer hard disks) on which an area was worn by scanning the surface – in this case using contact mode – at a preset force using a raster scan pattern. The worn areas result from only a single pass (scan) from top to bottom over the square area. Using this type of testing, the durability of materials can be compared. In Figure 6a, the film has been completely worn away from the substrate, whereas the films in Figures 6c and 6d appear to have worn only slightly with small amounts of debris shown at the edges of the scan. The film in Figure 6b exhibits wear similar to the topmost portion of Figure 6a; however the film did not fail entirely. The image shows that films 6c and 6d are tougher than films 6a and 6b. Figure 7 shows a dlc film, which appears to have delaminated from the substrate of the hard disk during the wear test.

The image shown in Figure 8 is a good illustration of the indent size and image resolution possible with the indentation cantilevers. The Digital Instruments logo was indented on dlc film using the indenting software/hardware and then imaged with the same tip. The scan size is about 250nm and the z-range (or full height range) is 4nm. The indentation depth, measured from the image, is about 1nm for the small indents and about 2.5nm for the indent used to dot the "i". For the small indentations, the projected area is approximately 400nm^2, with spacing of 25-30nm and indent width of about 20-25nm. The force applied during the indent was about 5μN for the small indents and 10μN for the large indent.

Figure 9 shows an array of indentations, which were performed on two polymer films using five different forces. Figure 9a is PMDA-ODA polyimide and Figure 9b is BPDA-PDA polyimide. The smallest indents are about 20nm deep and the largest about 200nm deep for both samples. The average depth of the indentations on the polymer in Figure 9b is about 85% of the depth for the polymer in Figure 9a, suggesting that the BPDA-PDA polyimide is harder than the PMDA-ODA. This is consistent with the chemical structures of the two polyimides, which indicate that the BPDA-PDA has stronger bonding than the PMDA-ODA. There is also an obvious difference in the amount of material, which has piled up at the edges of the indents for each of the polymers. The PMDA-ODA polyimide, which has deeper indentations, has less accumulated material than the BPDA-PDA.

The bovine and human sperm nuclei, shown in Figure 10, were indented using the same forces in order to compare the hardness of each. Both samples were indented using forces of about 8 and 16μN, and then imaged in air using the same indentation cantilever. The hardness of the bovine and human sperm was calculated as 0.9 and 0.5GPa, indicating that bovine sperm is about twice as hard as human sperm. This result is consistent with what is known about the different proteins, which compact the DNA in bovine and human sperm. Figure 10c and 10d show full size images of the sperm nuclei, which were imaged using the indentation cantilever.

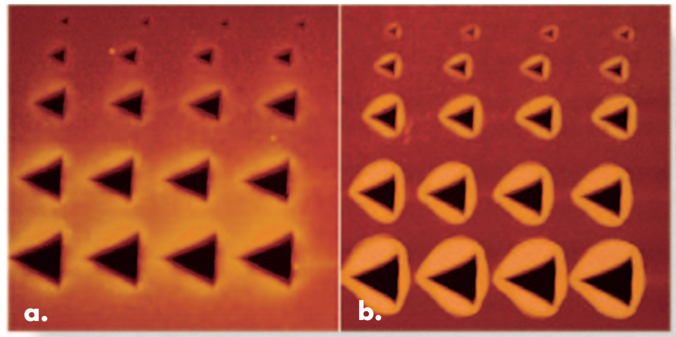

Figure 9. Indentations on two different polymers using the same forces to compare hardness. Each sample was indented four times using each of five forces. The first sample (a) is a PMDA-ODA polyimide, and the second sample (b) is a BPDA-PDA polyimide. The indentation depths vary from about 20-200nm and are deeper for the softer PMDA-ODA polyimide. 3μm scans.

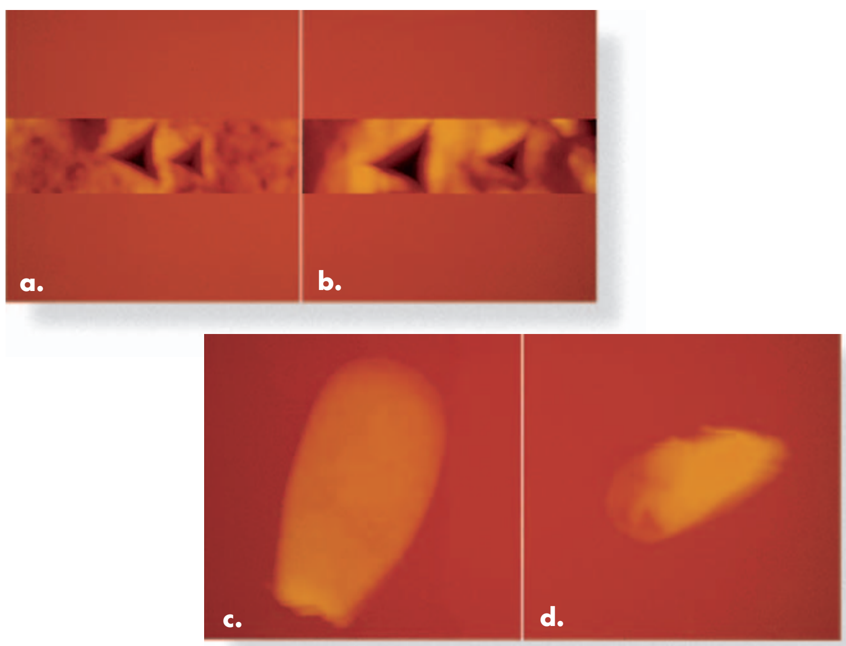

Figure 10. Indentations on bovine (a) and human (b) sperm nuclei, imaged in air and indented at forces of about 8 and 16μN. 1μm x 0.25μm scans. The hardness for the bovine and human sperm were calculated at about 0.9 and 0.5GPa, respectively. Full size images of bovine (c) and human (d) sperm nuclei were also imaged with the indentation cantilever. 10μm scans.

It is also possible to investigate the variation of mechanical properties of a surface by indenting or scratching at various locations on the same sample. Figure 11 shows an aluminum sample after CMP, which was indented at multiple locations. The force was the same for the top two rows, then increased for the next two, and increased again for the bottom two rows. The forces used were about 4, 5.3, 6.7μN, respectively. The top two rows of indentations are very similar in size with an average depth less than 1nm. But the bottom and middle two rows contain indentations of various size and depth. Within the bottom two rows the large indents are 6-7nm deep; whereas the smaller indents are 2-3nm deep. Other tests (not included here) suggest that the deeper indentations may have been located near grain boundaries on the sample surface and that the indents on the top two rows were either not proximate to such boundaries or were not deep enough (or made with sufficient force) to be affected by the boundaries.

Summary

Nanoindenting, nanoscratching, and wear testing with AFM hardware and software, in conjunction with our patented TappingMode AFM imaging technique, is a powerful tool for characterizing mechanical properties of the sample on, or near, the surface. An important advantage over other instruments is that the same tool, the AFM cantilever, can be used to (1) image the sample prior to testing the sample, (2) indent, scratch or wear the sample surface, and (3) image immediately afterwards to observe the change. Compared to other AFM techniques, Veeco's TappingMode AFM technique allows use of a high spring constant cantilevers to indent, scratch and image, eliminating the need to change tips and the difficulty of relocating the indentation or scratch. The addition of indentation cantilevers, a special cantilever holder, and the dedicated software is all that is needed, to add the nanoindentation, scratching and wear testing capability to a new or existing MultiMode or Dimension Series SPM equipped with a NanoScope® III, IIIa or IV controller.

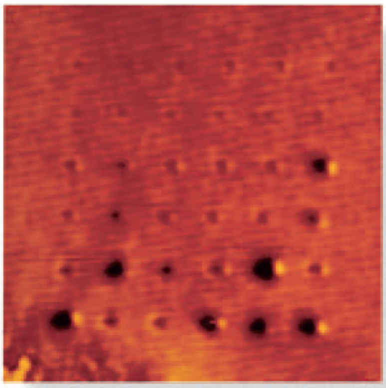

Figure 11. Indentations on aluminum sample after CMP show variations in the indentation size and depth at different locations when using a constant indentation force. Indents were made using three different forces for the top, middle, and bottom sets of two rows each. See text for additional discussion. 1μm scan.