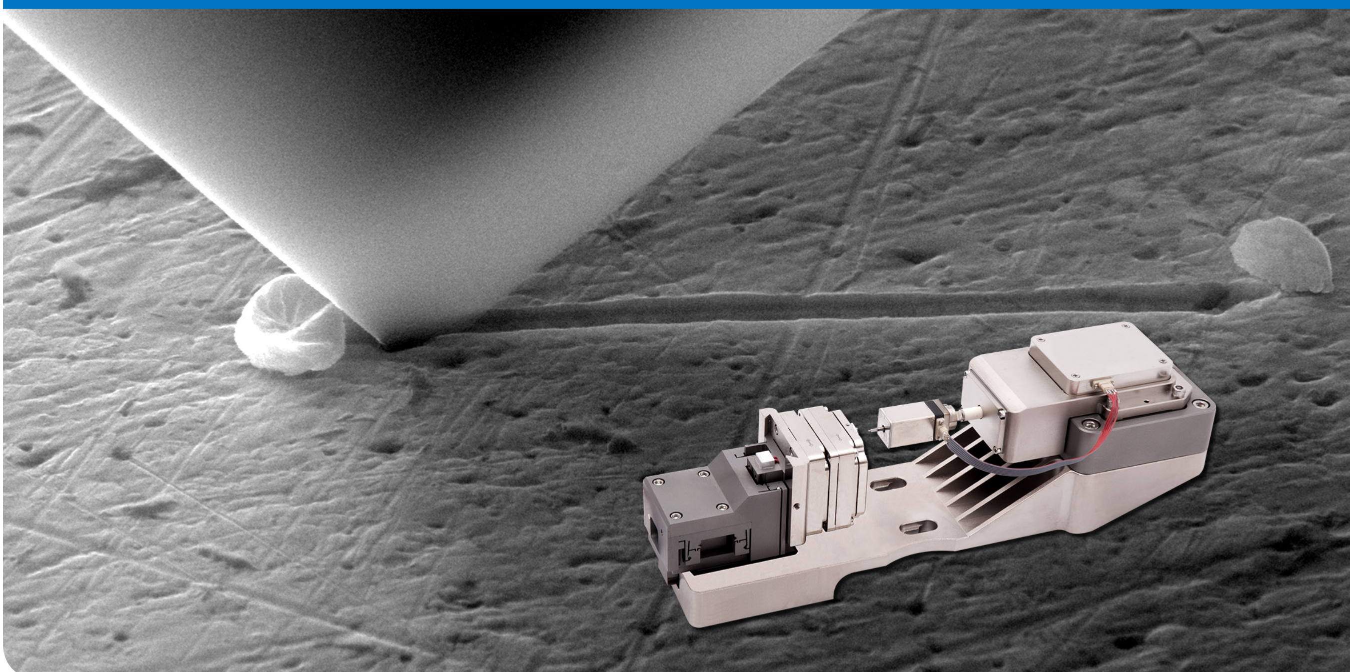

In-Situ SEM nanoScratch Module

Quantitative Tribology with the Hysitron PI 88 SEM PicoIndenter

Until recently, in-situ nanoscale mechanical testing has been limited to the application of purely normal forces. Quantitative nanoindentation, compression, bending, and tensile loading coupled with direct SEM observation are accomplished by varying the tip and sample geometry. Often however, testing in more than one dimension is required to fully understand the performance of a material system, especially when dealing with the tribological properties of a material. With the nanoScratch upgrade option for the Hysitron® PI 88 SEM PicoIndenter®, simultaneous normal and lateral force measurement is accomplished through a laterally oriented version of Bruker's capacitive force sensor. Lateral actuation is achieved through a specially designed long-range piezo actuator, which attaches to the standard XYZ sample positioning stages of the Hysitron PI 88.

Applications of In-Situ Scratch Testing

- **Films and Coatings**: Measure scratch resistance, coefficient of friction, and film-substrate adhesion, then correlate lateral force data to real-time SEM imaging to determine failure mode
- **Structures**: Apply shear forces to pillars, solder bumps, beams, MEMS devices, and oriented crystalline materials
- **Particle-Substrate Interaction**: Quantify the friction between a single micro- or nano-particle and the supporting substrate, and observe the damage initiation and wear processes
- **Atmospheric Control**: Minimize the influence of surface water layers and oxidation on frictional measurements, and allow for the in-situ characterization of tribological processes across a wide range of controlled conditions with ESEM

Case Study: Low-k Dielectric Film Failure

A Hysitron PI 88 SEM PicoIndenter equipped with the nanoScratch module and a diamond cube-corner probe was used to perform constant normal displacement and ramping normal force scratch tests on a silicon substrate under sliding conditions. The test goal was to quantify the performance of a low-k dielectric film. Figure 1 shows an SEM image of scratch tests where the normal displacement was held at a maximum of 750 nm, while the sample was moved laterally at a rate of 500 nm/s.

Clear evidence of adhesive failure is shown in the image, which happens in a very periodic manner. These delamination events are also followed by spallation of the delaminated material, as evidenced by the residual material that has been ejected from the scratch path. Figure 2 plots the normal displacement and lateral force versus time from one of these tests. The lateral force data clearly shows many large, periodic load drops that can be easily correlated to the corresponding delamination and spallation events shown in the static, post-test SEM image.

Often, the lateral force data from a scratch test contains more subtle events that can only be tenuously correlated to features in post-test optical or SEM imaging. However, by performing the test in-situ, a real-time SEM video of the test can be captured and synchronized to the mechanical data. This means that the entire deformation process can be revisited and properly correlated to the measured lateral force data, as shown in Figure 3.

As the reported data shows, the Hysitron PI 88 equipped with the nanoScratch module delivers quantitative nanoscale mechanical data in both the normal and lateral loading directions. By coupling this multidimensional testing capability with high-resolution SEM imaging, even the most transient events can now be captured and fully quantified. The in-situ nanoScratch module for Hysitron PI 88 represents a major step forward in the characterization of nanoscale wear processes for a wide variety of materials, such as protective coatings like diamond-like carbon (DLC), sliding components like bearings or MEMS devices, or for interpreting the performance of constituent thin film layers of electronic devices, such as low-k dielectric films.

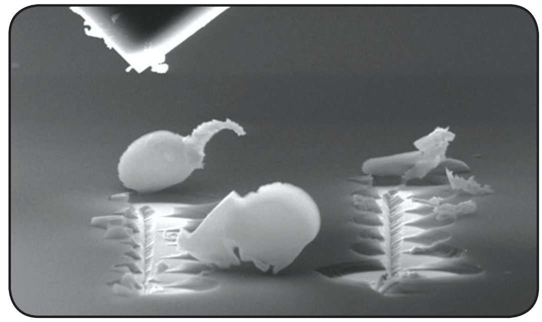

Figure 1. SEM Image from constant depth scratch tests on a low-k dielectric film.

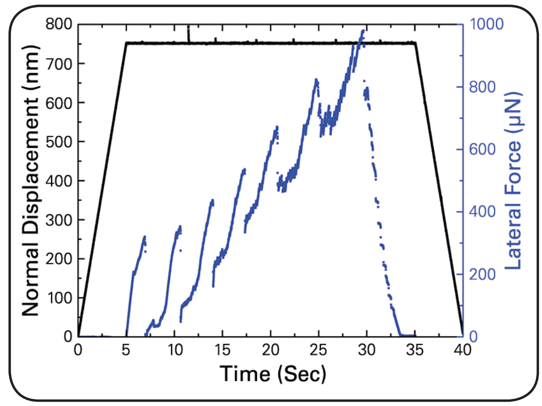

Figure 2. A plot of normal displacement and lateral force vs. time from a constant depth scratch test on a low-k dielectric film.

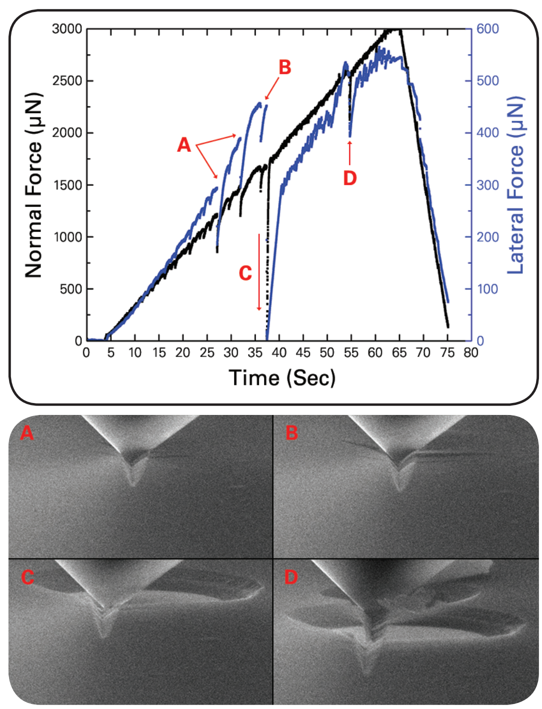

Figure 3. A plot of normal and lateral force data (top) recorded during a ramped normal load scratch test, along with frames (bottom) from the synchronized SEM video showing the various stages of deformation and failure:

A. Two pairs of cracks form perpendicular to the scratch path.
B. Delamination event and visible buckling ahead of probe.
C. Spallation/ejection of delaminated region.
D. Second spallation/delamination event once the probe reaches the far rim of the first spalled region.

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