### TECHNIQUE

# **Rigid Probe Indentation**

Comparison of Hysitron TriboIndenter®/TriboScope® and AFM-based Nanoindentation

#### Nanomechanical characterization is used to measure and evaluate numerous mechanical properties of materials, including modulus, hardness, fracture toughness, wear resistance and friction coefficient. Nanomechanical characterization, as well as visualization of surface topography, provides crucial information concerning the performance of materials.

Many of these demanding characterization requirements can be met with nanoindentation and scanning probe microscopy (SPM) imaging. Quantification of these properties at the nanoscale requires accuracy in the measurement of both load and displacement during testing to ensure precise calculations of the mechanical properties of a material.

Two very different techniques for nanoindentation have emerged. These are AFM indentation and rigid probe indentation. Understanding the mechanics behind these different techniques will ensure the most accurate measurements are made for reliable results.

# Force Range

Nanoindentation is commonly used to characterize materials that have moduli spanning several orders of magnitude. An effective instrument for testing this diverse range of materials must possess a broad force range. AFM-based nanoindentation is limited by the stiffness of the cantilever on which the indenter probe is placed. Typically AFM-based nanoindentation is limited to about 100 $\mu$ N maximum force. In contrast, Hysitron offers a force range from 100nN to 10N.

Hysitron also offers load and displacement control indentation testing. This allows for both creep and stress relaxation tests. The displacement control also ensures that indents have precisely controlled indentation depths which is crucial for thin film research.



Figure 1. Schematic of AFM indentation (left) and Hysitron indentation (right) showing bending forces on AFM cantilever which can be difficult to quantify. The Hysitron indenter comprises of a rigid probe which prevents bending motion resulting in more accurate measurements.

## **Machine Compliance**

Machine compliance, or load frame stiffness can be a significant source of error. This should be minimized in order to obtain accurate, quantitative nanoindentation results. Machine compliance is the additional displacement measured during indentation due to the deformation of the testing instrument or the indenter probe. Typically, the machine compliance can be measured and compensated for by using a software correction function. This is an exacting challenge in AFM-based nanoindentation as the machine compliance is non-linear and not well characterized as the indenter probe is placed on the end of a long cantilever that flexes under load (Figure 1). Consequently, corrections for the machine compliance of AFM systems require complex modeling that do not guarantee accurate results. In contrast, nanoindentation instruments utilize a rigid probe that is normal to the surface and eliminates cantilevered motion. This minimizes machine compliance and provides more reliable results.





## **Testing Modes**

Nanoindentation using a rigid probe can be used in numerous testing modes supporting the measurement and monitoring of many nanoscale properties and events. AFM testing was primarily developed as a surface imaging tool. Hysitron has the ability to perform many nanoscale techniques through an abundance of testing modes in addition to its imaging capability. These techniques include, **nanoDMA**<sup>®</sup> for viscoelastic analysis, **TriboAE**<sup>TM</sup> for fracture studies and **TriboImage**<sup>TM</sup> for tribological research.

### Lateral Force

Acquiring quantitative force and displacement data in the lateral direction allows for quantification of friction coefficient, scratch resistance and wear parameters. AFM-based nanoindentation can not accurately quantify lateral forces due to the inability to determine the lateral stiffness of the cantilever. Hysitron utilizes the same transducer used for indentation to actuate laterally, bringing the same high-performance characterization capabilities to lateral force testing. Scratch testing is routinely used to investigate, thin film delamination, mar resistance and lubricant efficiency.

#### Imaging

The advantage of Hysitron nanoindentation systems over other nanoindentation instruments is the capability to provide *in-situ* imaging of the sample surface. This ensures precise test placement, as well as the ability to view the resultant deformation without moving to move the sample to an external instrument. Minimizing this time between testing and imaging is particularly crucial for viscoelastic materials where the observed deformation depends on the amount of recovery time.

Hysitron exclusively offers quantitative rigid probe nanomechanical testing as well as *in-situ* imaging as standard. The sensitivity and high bandwidth of Hysitron's testing systems allow nanomechanical characterization and *in-situ* imaging to be accomplished with the same transducer and tip.





Figure 2. Left—*In-situ* SPM image of residual indent showing fracture events in the form of crack propagation from the corners of the indent tip. This image was made using the same indentation tip that performed the testing. Right—Quasistatic nanoindentation curve of imaged indent showing fracture events as indicated by blue dots.

#### HIGHLIGHTS

- Force range from 100 nN to 10 N
- *In-situ* imaging and quantitative indentation as standard
- Quantitative measurement of stiffness, hardness and modulus
- User definable tip area function
- Displacement control for precise control of indentation penetration depth
- Creep and stress relaxation measurements
- Quantitative lateral force and scratch measurements
- Simple calibration for machine compliance and tip area function

# APPLICATIONS

- Hard materials and films which require higher loads.
- Heterogeneous materials such as composites with large mechanical variations
- Multilayer films that require displacement control to ensure testing stays within each film
- Viscoleastic materials where recovery time alters residual indent impression