Asylum Research
Atomic Force Microscopes

The MFP Instrumented NanoIndenter for Quantitative Materials Characterization

Asylum's MFP NanoIndenter is a true instrumented indenter and is the first AFM-based indenter that does not use cantilevers as part of the indenting mechanism. These characteristics and the use of state-of-the-art AFM sensors provide substantial advantages in accuracy, precision and sensitivity over other nanoindenting systems.

Overview and Advantages

Unlike cantilever indenters, the MFP NanoIndenter (Figure 1) moves the indenting tip perpendicular to the surface. This vertical motion avoids the lateral movement and errors that are inherent in cantilever-based systems. Compared to conventional commercially-available instrumented nanoindenters, the MFP NanoIndenter provides lower detection limits and higher resolution measurements of force and indentation depth with the superior precision of AFM sensing technology.

The indenter is completely integrated with the AFM, providing the unique ability to quantify contact areas by performing AFM metrology of both the indenting tip and the resulting indentation (Figure 2). These direct measurements enable analysis of material properties with unprecedented accuracy relative to indirect calculation methods. The design uses passive actuation through a monolithic flexure, minimizing drift and other errors in depth measurement.

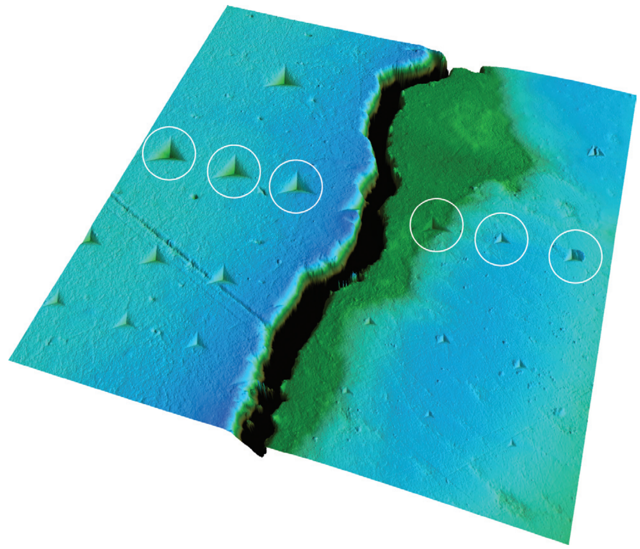


Figure 2: Indenting on dentin (left of crack), and enamel (right) on a human tooth sample. The indentations in each row (one row is circled) were all created with the same maximum force. The smaller indents on the enamel demonstrate that it is harder than the dentin, 70µm scan. Sample courtesy D. Wagner and S. Cohen, Weizmann Institute of Science.

The positioning accuracy in the sample plane is sub-nanometer using the MFP's closed loop nanopositioning sensors. The NanoIndenter Head utilizes advanced diffraction-limited optics coupled with CCD image capture for precision navigation of the tip to areas of interest on the sample.

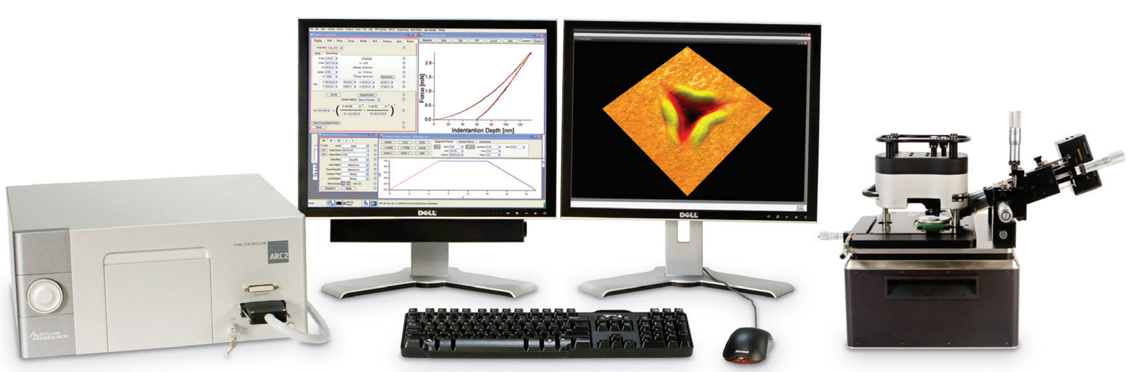


Figure 1: The MFP NanoIndenter integrates the quantitative capabilities of instrumented nanoindenters with the resolution of AFM/SPM to provide advanced materials characterization with enhanced accuracy, precision and sensitivity.

The integrated software provides a full complement of experimental control and analysis functions, including standard analysis method templates. The system kit includes a set of nanoindenting tips, three different sample mounts, two calibration standards for sensitivity and spring constant verification, as well as the tools and accessories necessary to perform indenting experiments on a full range of materials. This highly quantitative tool, combined with high-end AFM capabilities, breaks new ground in the characterization of diverse materials including thin films, coatings, polymers, biomaterials, and many others. The MFP NanoIndenter Module is available in standard (spring constant typ. 4,000N/m) and low force (spring constant typ. 800N/m) versions exclusively for the Asylum MFP-3D™ AFM.

Innovative, Robust Design

At the heart of the NanoIndenter is our exclusive sensored closed loop head, designed with a robust flexured transducer for quantitative measurements.

Monolithic Design Eliminates Drift and Errors in Depth Measurements

With conventional nanoindenters, electrical actuation typically causes small parts to heat up, resulting in drift and, consequently, measurement errors. The monolithic design (Figure 3) of the flexured and sensored Z axis of the MFP NanoIndenter eliminates these drift problems and provides for quantifiable results.

Nanopositioning for Accuracy and Precision

Displacement of the MFP's indenting flexure is performed with a piezo actuator and measured with our patented low-noise, sensored Nanopositioning System (NPS™). The force is computed digitally as the product of the spring constant and the measured indenter flexure displacement. This measurement is generated by converting the optical signal (measured at the MFP photodetector) into the displacement of the vertical indenting flexure. The indenter provides unprecedented resolution because the two quantities of interest – depth and force – are computed based on displacements measured with state-of-the-art AFM sensors. Unlike conventional instrumented nanoindenters that cannot quantitatively measure the force in real time, the optical lever detection enables high bandwidth, true force feedback. This allows repeatable imaging, quantitative feature measurement, quantitative force curves, and accurate positioning for manipulation and lithography.

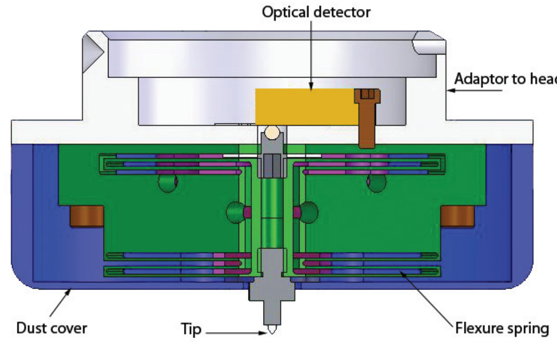


Figure 3: The NanoIndenter transducer is a flexured, sensored design for unprecedented precision and accuracy.

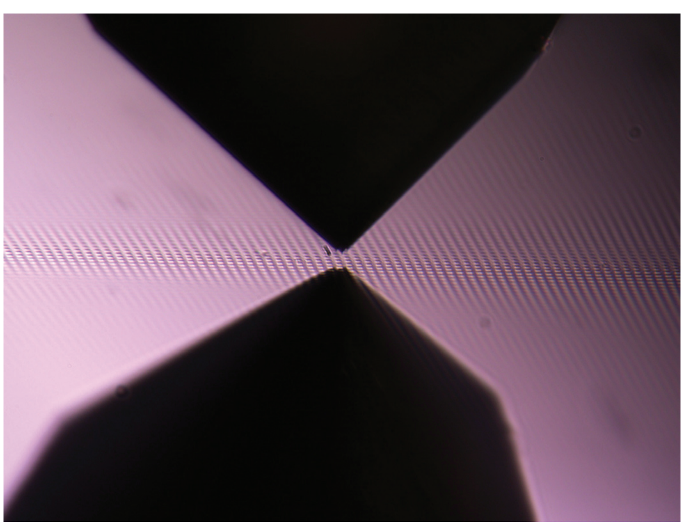


Figure 4: Optical view of 10µm calibration grating and cube corner tip. Reflection of the tip (bottom) can be seen on the sample.

Diffraction-limited Optics Provide High Resolution Viewing of Tip and Sample

The NanoIndenter optics and camera assembly provides viewing of the indenter tip and sample at an angle of 20 degrees from horizontal (Figure 4). The indenter tip can be positioned with an accuracy of 20 micrometers with a 5x objective on a reflective surface.

Different regions of the sample can be viewed with the optic's independent translation stage. The design allows for easy exchange of objectives to accommodate different sample requirements. A built-in iris diaphragm provides adjustable depth of field and the camera allows for adjustment of exposure time, gain, frame rate, saturation, and gamma.

Easy-to-use Pre-calibrated Setup and Calibration Verification

The spring constant is calibrated with three independent methods to minimize errors in your measurements – the added-mass, reference spring, and micro-balance methods. Calibration of the indenter flexure assembly is performed at the factory; hardware and software are provided for calibration checks by users, ensuring precision and accuracy over the life of the instrument.

Push and Turn Adjustments Maintain Calibration

The advanced design of the NanoIndenter head includes push-and-turn adjustments that maintain the pre-set calibration parameters and protect against accidental changes.

Direct Measurement for Tip Characterization and Accurate Results

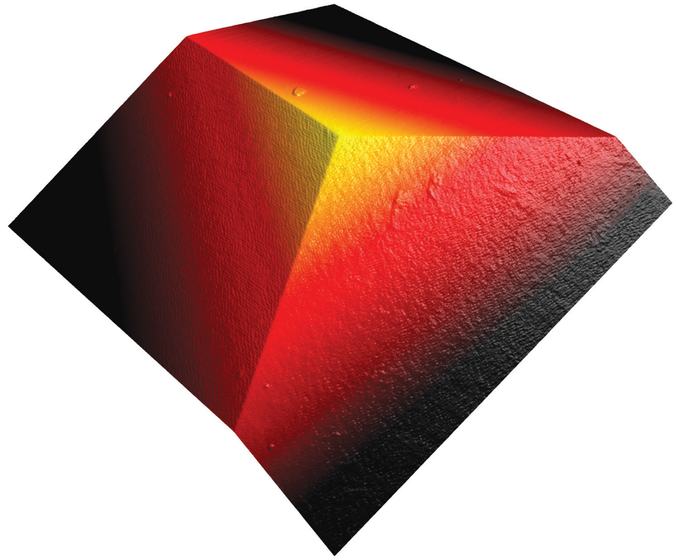
Tip characterization is extremely important for quantitative analysis in nanoindenting applications. Conventional nanoindenters must use indirect methods to evaluate the effect of the indenter tip geometry on the indentation results, such as indenting on a standard sample (fused silica) with application of theoretical and experimental assumptions. In contrast, the MFP NanoIndenter allows *direct* tip metrology using standard AFM techniques (Figures 5). This method specifically avoids the theoretical assumptions and associated experimental errors inherent in conventional methods (e.g. Oliver-Pharr). Similarly, AFM metrology of resulting indentations (Figure 6) provides additional experimental data to improve upon the accuracy of theories for data analysis. In addition, damaged or worn tips can be identified through AFM imaging and discarded before invalid data are collected.

Applications

The NanoIndenter is ideal for a variety of nanoindenting applications including:

- Elastic behavior of metals, ceramics, polymers, etc.
- Dislocation phenomena in metals
- Fractures in ceramics
- Mechanical behavior of thin films, bone, biomaterials
- Residual stresses
- Time-dependent mechanical characteristics of soft metals and polymers
- Combined nanoindenting with current-voltage measurements (IV)
- Combined nanoindenting and Piezoresponse Force Microscopy (see PFM Application Note)

The MFP-3D AFM platform allows accurate estimation of elastic rebound, pile-up and sink-in material volumes. AFM imaging is key to identification of cracks, displacement, and failure zones in indented samples, as well as imaging of features revealing physical phenomena.



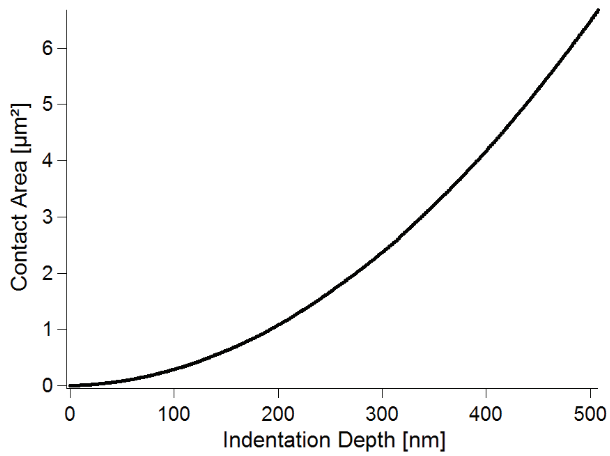


Figure 5: Direct measurement of tip (Berkovich) area function shows the relationship between depth (AFM Z-sensor data, top) and contact area (numerical integration of the data, bottom), 5µm scan.

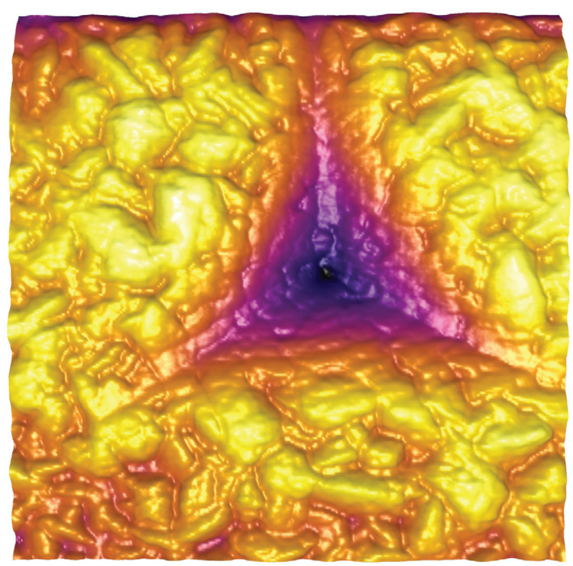


Figure 6: Granular materials have contact areas that are discontinuous and need to be measured for a correct estimation of material properties. Indium tin oxide, 800nm scan.

Specifications

MFP NanoIndenter System Configurations

Combination AFM and Nanoindenting System

This configuration provides maximum flexibility and capability for both nanoindenting and AFM imaging. The system includes a full MFP-3D system (with closed loop AFM scanner), a dedicated nanoindenter head and the nanoindenter optical system. This configuration is ideal for customers doing frequent nanoindenting and for multi-user facilities where users commonly use the MFP-3D for both nanoindenting and other applications.

The system above includes Asylum's ARC2™ all-digital control system (controller, computer and monitors), user choice of base for viewing of the tip (either Top View or Dual View), user choice of Standard or Low Force nanoindenter head, two diamond tips (one cube corner and one Berkovich) and one sapphire sphere, a ruby learner's tip, a spring constant calibration reference, and sample holders for small, medium and large samples.

NanoIndenter Upgrade

The NanoIndenter Head is available as a separate option for use with the user's existing MFP-3D AFM. The head can be used interchangeably with the existing closed loop MFP-3D scanner.

NanoIndenter Head

Piezo driven Z flexure with NPS sensor carries an optical lever system with low-coherence SLD for detection of nanoindenter flexure displacement. Push and turn adjustments on top. Optional 40µm Extended Head available.

NanoIndenter Optics

Diffraction-limited optics with adjustable focus, depth of field, saturation and gamma provide high-resolution optical imaging of tip and sample from a 20 degree angle. Micrometers on three axes allow adjustment of focus and viewing position.

NanoIndenter Flexure

Two flexure models are available – Standard and Low Force.

	Standard	Low Force
Spring constant (typ.):	4,000N/m	800N/m
Mass:	250mg	200mg
Resonant frequency:	700Hz	300Hz

Force Resolution

Measured in a 1kHz bandwidth.

	Standard	Low Force
Maximum load:	20mN	4mN
Load Noise Floor*:	75nN	15nN
Displacement Noise Floor*:	0.3nm	0.3nm

**Measured in a 0.1Hz to 250Hz bandwidth*

NanoIndenter is a Class 1M Laser Product

Range

Z range: 15µm standard, 40µm (optional).
Load rate range (V_{lr}): 1µN/s $< V_{lr} <$ 10mN/s.

Sample Size/Holders

Small: 12.7mm diameter SEM stubs.
Medium: 2.0" maximum diameter.
Large: 4.0" x 2.4" maximum.

Stage

Micrometer driven stage for mechanical alignment of the indenter tip and sample.

Scan Axes

X-Y: 90µm travel in closed loop. Closed loop position control with sensor noise <0.6nm average deviation (Adev) in a 0.1Hz-1kHz bandwidth (BW) and sensor non-linearity <0.5% (Adev/full travel) at full scan.
Z: >15µm sensored travel in closed loop (40µm optional). Sensor noise <0.3nm Adev in a 0.1Hz-1kHz BW and sensor non-linearity less than 0.2% (Adev/full travel) at full scan.
Z height: noise <0.06nm Adev, 0.1Hz-1kHz BW.

Software

Based in IGOR Pro by WaveMetrics, a powerful scientific data acquisition and analysis environment. The software is customizable.

For nanoindentation, features include:

- Feedback on force (stress control) and indentation depth (strain control)
- Force vs. time
- Force vs. displacement
- Constant loading rate
- Quasi-static test methods: indentation creep, stress relaxation
- Dynamic techniques

Nanoindenting Tips

Asylum Research offers a variety of nanoindenting tips with standard or custom geometries. Each system ships with two diamond tips (one cube corner and one Berkovich). The tips are calibrated in compliance with the requirements of the International Standard ISO/IEC 17025.1. Dimensions and angles comply with the ISO 14577-22 standard, which defines internationally accepted micro- and nanoindenter tolerances.

Numerous geometries are available for the indenter shape, such as three-sided pyramids, four-sided pyramids, wedges, cones, cylinders or spheres. The tip end of the indenter can be made sharp, flat, or rounded to a cylindrical or spherical shape. Asylum carries Berkovich, modified Berkovich, cube corner, and Vickers as standard traceable nanoindenters. These indenters are inspected and measured with equipment and standards traceable to the NIST or PTB. We also offer conductive diamond tips and blank tip holders to allow users to mount their own tips. Contact Asylum Research for additional nanoindenting tip models and custom options.

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