

# Agilent Continuous Stiffness Measurement (CSM) Option

Data Sheet

## Features and Benefits

- Accurate, repeatable results compliant with ISO 14577 standards
- Dynamic properties characterization via continuous measurement of stiffness by indentation depth
- Seamless compatibility with Agilent Nano Indenter XP and DCM indentation heads
- Ability to fully characterize dynamic properties in the nanometer range as well as accurately characterize viscoelastic materials
- Ability to control indentation tests with a constant strain rate

## Applications

- Semiconductor, thin films, MEMs (wafer applications)
- Hard coatings, DLC films
- Composite materials, fibers, polymers
- Metals, ceramics
- Biomaterials, biology

## Overview

Nano Indenters from Agilent Technologies make it possible to characterize mechanical properties (such as modulus of elasticity, loss factor, and fracture behavior) in the nanometer range using both quasi-static and dynamic depth-sensing indentation methods.

The accuracy and reliability of mechanical properties characterization depends on more than the accuracy of the tip geometry and the force and displacement measurements. To accurately calculate the contact area and mechanical property values, the stiffness of the contact between the indenter tip and the sample material must also be accurately determined.

## Quasi-Static and Dynamic Methods

Agilent Nano Indenters support both quasi-static and dynamic depth-sensing indentation methods. In conventional quasi-static indentation testing, the stiffness of contact is determined by analyzing the force vs. displacement curve during unloading. This depth-sensing method provides a single measurement for the given indentation depth. The Agilent



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Continuous Stiffness Measurement (CSM) option, which is compatible with both the Agilent Nano Indenter XP and DCM indentation heads, satisfies application requirements that must take into account dynamic effects, such as strain rate and frequency.

With the CSM option, the Agilent Nano Indenter applies a load to the indenter tip to force the tip into the surface while simultaneously superimposing an oscillating force with a force amplitude generally several orders of magnitude smaller than the nominal load. The CSM option offers a means of separating the in-phase and out-of-phase components of the load-displacement history. This separation provides an accurate measurement of the location of initial surface contact and continuous measurement of contact stiffness as a function of depth or frequency, thus eliminating the need for unloading cycles. Since the contact stiffness is determined directly, no assumptions (such as mechanical equilibrium) are required to correct for elasticity.

As a result, property measurements are inherently more accurate using CSM. Obtaining the same data would require tens or even hundreds of

tests using the conventional method. This makes CSM a powerful tool not only for stiff materials such as metals, alloys, and ceramics but also for time-dependent materials like polymers, structural composites, and biomedical materials.

The state-of-the-art CSM option provides the only means available to both fully characterize dynamic properties in the nanometer range and accurately characterize viscoelastic materials providing values such as storage modulus. Indentation tests using CSM can be controlled with a constant strain rate, a critical test parameter for material systems such as pure metals or low-melting-point alloys, and polymer films and film/substrate systems. This level of control is not possible with the conventional method.

## Nano Indenters

The culmination of decades of research and development, Agilent Nano Indenters are the world's most accurate, flexible, and user-friendly instruments for nanoscale mechanical testing. Electromagnetic actuation allows Nano Indenters to achieve unparalleled dynamic range in force and displacement. These advanced

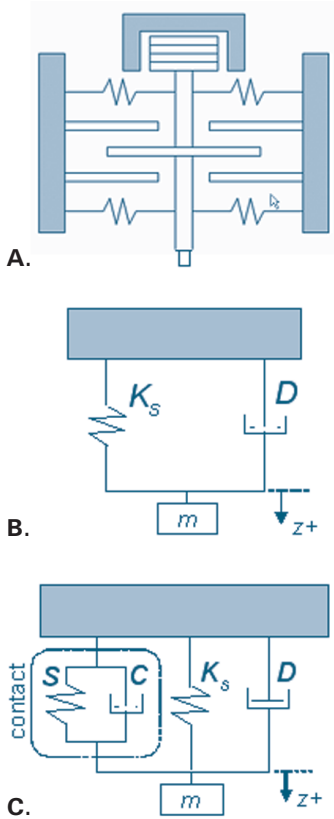


Figure 1. A. Schematic of free-hanging indenter. B. Dynamic model of instrument alone (no contact). C. Dynamic system model during testing.

$$m\ddot{h} + D\dot{h} + kh = F(t), \text{ where } h = h_0 e^{i(\omega t - \phi)} \text{ and } F(t) = F_0 e^{i\omega t}$$

$$S_{\text{contact}} = \left[ \frac{F_0}{h_0} \cos \phi + m\omega^2 \right]_{\text{coupled}} - \left[ \frac{F_0}{h_0} \cos \phi + m\omega^2 \right]_{\text{inst. (freespace)}}$$

$$E' = \frac{\sqrt{\pi}}{2} \frac{S_{\text{contact}}}{\sqrt{A}}$$

$$E'' = \frac{\sqrt{\pi}}{2} \frac{(D\omega)_{\text{contact}}}{\sqrt{A}}$$

$$\tan \delta = \frac{E''}{E'} = \frac{D\omega}{S}$$

Figure 2. CSM can accurately characterize viscoelastic materials reporting values such as storage modulus and  $\tan \delta$ .

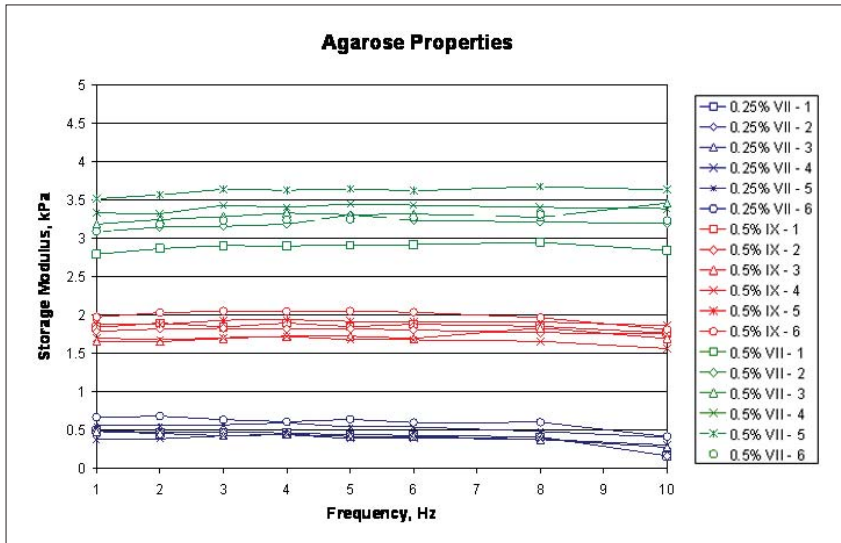


Figure 3. Accurate measurement of mechanical properties of Agarose is very important because it has been found that the storage modulus of these gels affect the form and function of the resulting cells. Three different gels were tested and as seen in the plot above viscoelastic characterization of the Agarose gels was achieved down to  $E' = 500\text{Pa}$ .

instruments not only enable users to measure Young's modulus and hardness in compliance with ISO 14577 standards but also enable measurement of deformation over six orders of magnitude (from nanometers to millimeters).

Agilent Nano Indenters are capable of characterizing even the most compliant materials. These measurements are possible because Nano Indenters are designed so that they can be modeled by the simple harmonic oscillator model, which makes system characterization simple. When testing compliant samples, Agilent characterizes the indenter in free air so that the machine contribution can be subtracted from the system response where only the desired sample properties remain.

Nano Indenters are carefully designed to account for the dynamics of indentation testing. Each system is individually calibrated and characterized over its full dynamic range of operation to ensure maximum accuracy and reliability.

Every Nano Indenter is backed by highly responsive Agilent Technologies customer service personnel. Knowledgeable and experienced regional applications engineers are available to guide users through more advanced testing, provide outstanding technical support, and offer unmatched applications expertise.

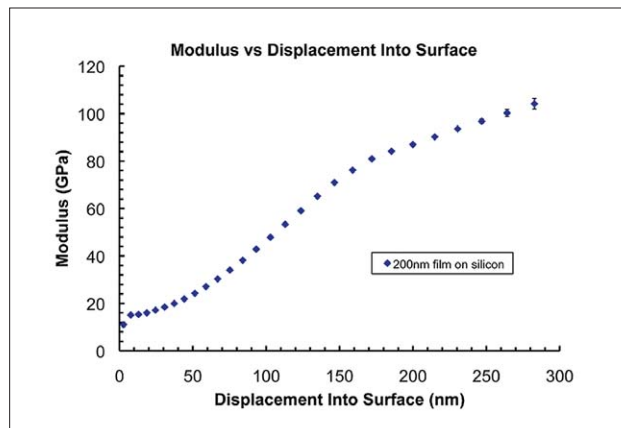


Figure 5. Characterization of function of depth on thin film that was applied to sample using CSM.

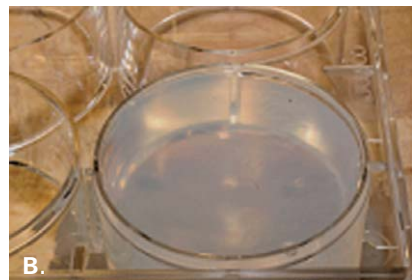
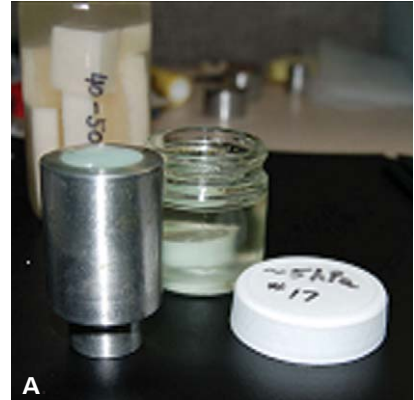


Figure 4. A. Concentric-cylinder sample holder. B. Agarose gel as supplied in a well plate.

## Specifications

Frequency Range	0.001 Hz to 120 kHz
Load Coupled Frequency Range	0.001 Hz to 1 kHz
Voltage Sensitivity	2 nV to 1 V full scale
Typical Operating Frequency Range	1 Hz to 300 Hz

### The Min and Max Load Amplitudes

DCM Max	0.9 mN
DCM Min	0.9 nN
XP Max	5.0 mN
XP Min	1.0 nN

*Note: There is a complex interplay between the frequency, the transfer function, the sample properties, and the depth of contact and contact area of the indentation. There is no simple answer as to “can I test my sample at a given frequency”. The answer is “it depends on the sample, the depth of indentation, contact area of indentation, frequency, and the transfer function.”*

**Nanoindentation instruments from Agilent Technologies conform to ISO 14577 standards, delivering confidence in test accuracy and repeatability. These state-of-the-art solutions ensure reliable, high-precision measurement of nanomechanical properties for research and industry.**

## Nano Mechanical Systems from Agilent Technologies

Agilent Technologies, the premier measurement company, offers high-precision, modular nano-measurement solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Agilent’s leading-edge R&D laboratories ensure the continued, timely introduction and optimization of innovative, easy-to-use nanomechanical system technologies.

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