NANOVEA
A Better Measure.

Tooth Hardness Evaluation Using Nanoindentation

Prepared by
Camille Murray

6 Morgan, Ste156, Irvine CA 92618 · P: 949.461.9292 · F: 949.461.9232 · nanovea.com

INTRO

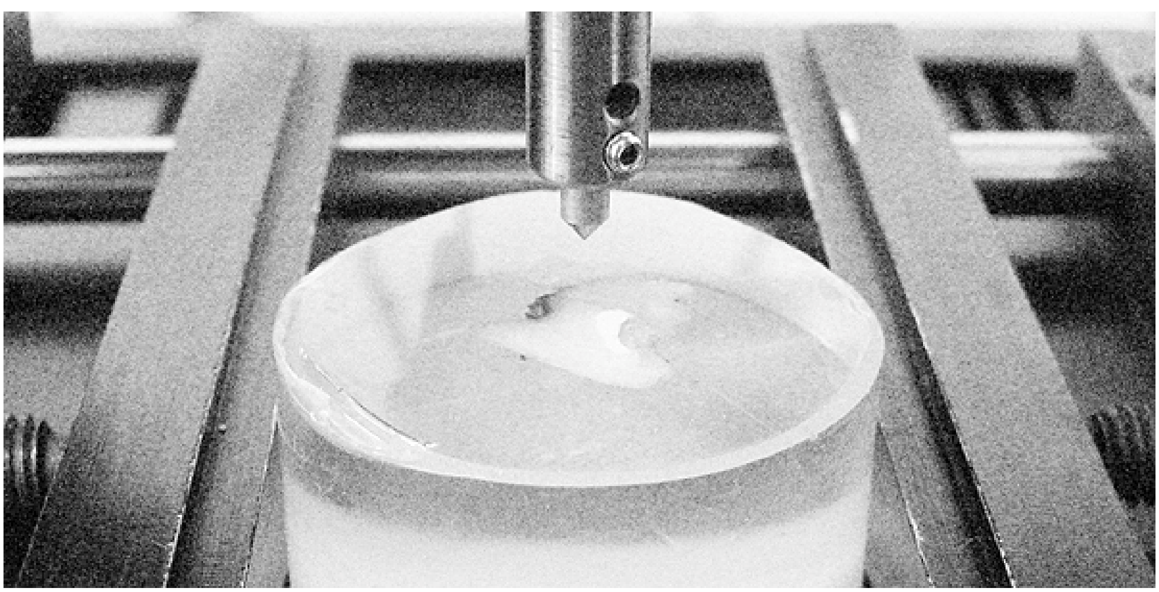
Biological material that has naturally developed over time is the inspiration for many new material developments. These biological materials of interest are made up of complex organic and inorganic structures resulting in superior mechanical properties. To reproduce a high-performance man-made version will require comparable properties. It is thus critical to understand, control and evaluate the fabrication of these bio-inspired materials for intended mechanical property results.

IMPORTANCE OF NANOINDENTATION FOR BIOMATERIALS

With many traditional mechanical tests (Hardness, Adhesion, Compression, Puncture, Yield Strength etc.), today's quality control environments with advanced sensitive materials, from gels to brittle materials, now require greater precision and reliability control. Traditional mechanical instrumentation fails to provide the sensitive load control and resolution required; designed to be used for bulk materials. As the size of material being tested became of greater interest, the development of Nanoindentation provided a reliable method to obtain essential mechanical information on smaller surfaces such as the research being done with biomaterials. The challenges specifically associated with biomaterials have required development of mechanical testing capable of accurate load control on extremely soft to brittle materials. In addition, multiple instruments are needed to perform various mechanical tests which can now be preformed on a single system. Nanoindentation provides a wide range of measurement with precise resolution at nano controlled loads for sensitive applications.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Nanoindentation mode, is used to study the hardness and elastic modulus of the dentin, decay and pulp of a tooth. The most critical aspect with Nanoidentation testing is securing the sample, here we took a sliced tooth and epoxy mounted leaving all three area of interest exposed for testing.



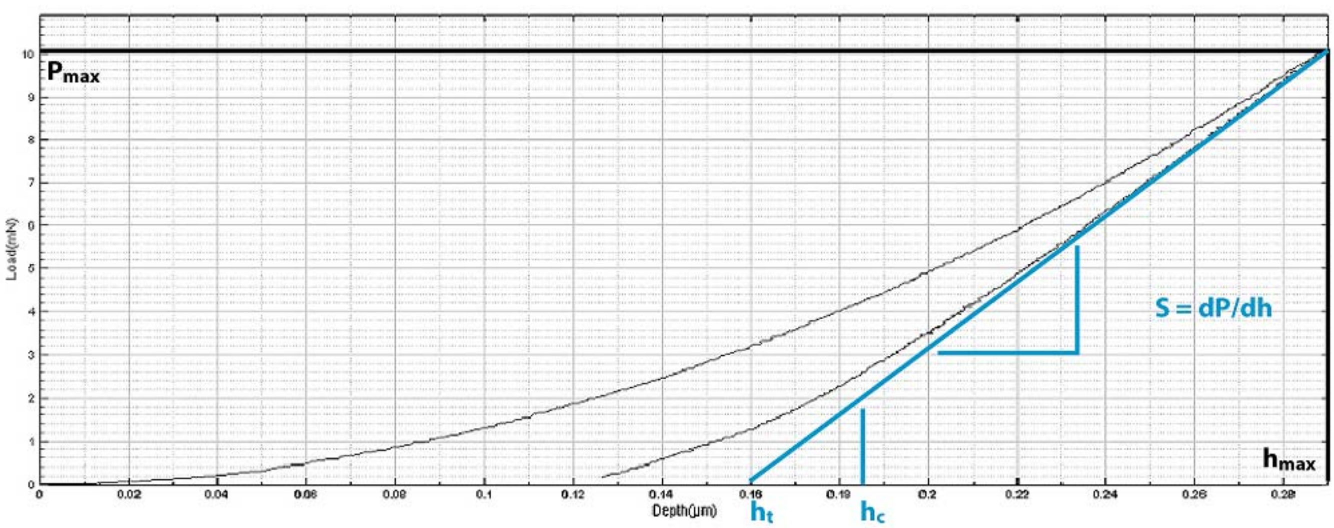
MEASUREMENT PRINCIPAL

Nanoindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an already established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set maximum value, the normal load is reduced until complete relaxation occurs. The load is applied by a piezo actuator and the load is measured in a controlled loop with a high sensitivity load cell. During the experiment the position of the indenter relative to the sample surface is precisely monitored with high precision capacitive sensor. The resulting load/displacement curves provide data specific to the mechanical nature of the material under examination. Established models are used to calculate quantitative hardness and modulus values for such data. Nanoindentation is especially suited to load and penetration depth measurements at nanometer scales and has the following specifications:

Maximum displacement (Dual Range)	: 50 μm
Depth Resolution (Theoretical)	: 0.003 nm
Depth Resolution (Noise Level)	: 0.4 nm
Maximum force	: 400 mN
Load Resolution (Theoretical)	: 0.03 μN
Load Resolution (Noise Floor)	: 1.5 μN

Analysis of Indentation Curve

Following the ASTM E2546 (ISO 14577), hardness and elastic modulus are determined through load/displacement curve as for the example below.



Hardness

The hardness is determined from the maximum load, P_{max}, divided by the projected contact area, A_c:

$$H = \frac{P_{\max}}{A_c}$$

Young's Modulus

The reduced modulus, E_r, is given by:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}}$$

Which can be calculated having derived S and A_C from the indentation curve using the area function, A_C being the projected contact area. The Young's modulus, E, can then be obtained from:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}$$

Where E_i and ν_i are the Young's modulus and Poisson coefficient of the indenter and ν the Poisson coefficient of the tested sample.

How are these calculated?

A power-law fit through the upper 1/3 to1/2 of the unloading data intersects the depth axis at h_t. The stiffness, S, is given by the slope of this line. The contact depth, h_c, is then calculated as:

$$h_c = h_{max} - \frac{3P_{max}}{4S}$$

The contact Area Ac is calculated by evaluating the indenter area function. This function will depend on the diamond geometry and at low loads by an area correction.

For a perfect Berkovich and Vickers indenters, the area function is $A_c=24.5h_c^2$ For Cube Corner indenter, the area function is $A_c=2.60h_c^2$ For Spherical indenter, the area function is $A_c=2\pi Rh_c$ where R is the radius of the indenter. The elastic components, as previously mentioned, can be modeled as springs of elastic constant E, given the formula: $\sigma = E\varepsilon$ where σ is the stress, E is the elastic modulus of the material, and ε is the strain that occurs under the given stress, similar to Hooke's Law. The viscous components can be modeled as dashpots such that the stress-strain rate relationship can be given as, $\sigma = \eta\frac{d\varepsilon}{dt}$
where σ is the stress, η is the viscosity of the material, and dε/dt is the time derivative of strain.

Since the analysis is very dependent on the model that is chosen. Nanovea provides the tool to gather the data of displacement versus depth during the creep time. The maximum creep displacement versus the maximum depth of indent and the average speed of creep in nm/s is given by the software. Creep may be best studied when loading is quicker. Spherical tip might be a better choice.

Other tests possible includes the following:

Puncture Resistance, Stress-Strain & Yield Stress, Fracture Toughness, Compression Strength, Fatigue testing and many others.

TEST CONDITIONS

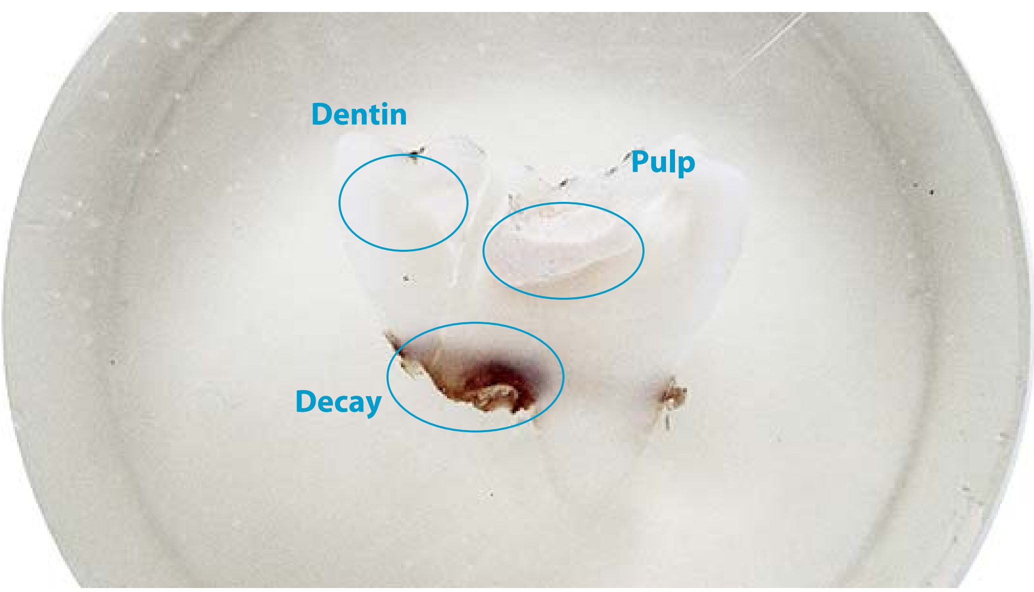
	All Samples
Maximum force (mN)	30
Loading rate (mN/min)	60
Unloading rate (mN/min)	60
Creep (s)	20
Computation Method	Nanovea
Indenter type	Berkovich Diamond

RESULTS

This section includes a summary table that compares the main numerical results for the different samples, followed by the full result listings, including each indentation performed, accompanied by micrographs of the indentation, when available. These full results present the measured values of Hardness and Young's modulus as the penetration depth with their averages and standard deviations. It should be considered that large variation in the results can occur in the case that the surface roughness is in the same size range as the indentation.

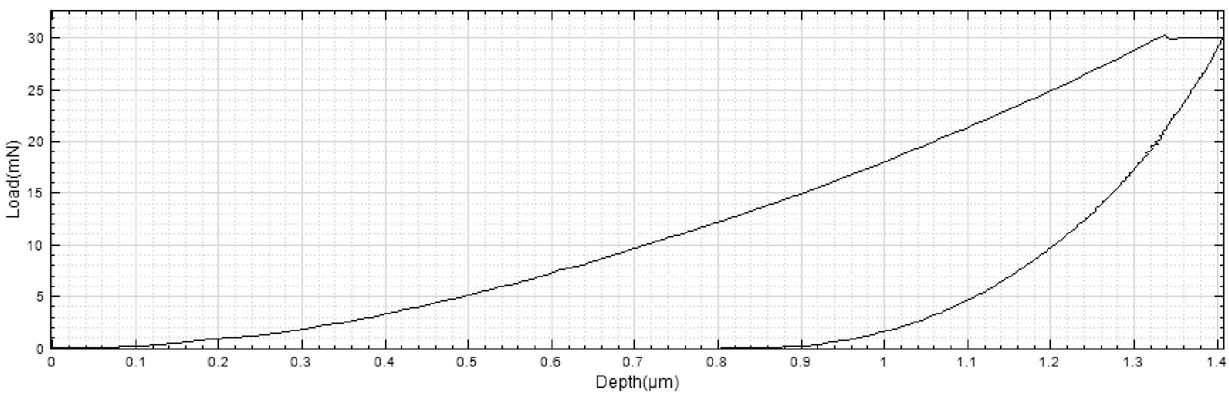
Summary table of main numerical results

Sample	Hardness [Vickers]	Hardness [GPa]	Young's Modulus [GPa]	Depth [nm]
Dentin	83.0 ± 3.0	0.878 ± 0.032	17.9 ± 0.5	1360 ± 19
Decay	24.3 ± 5.1	0.257 ± 0.054	4.58 ± 0.47	3329 ± 310
Pulp	20.5 ± 0.3	0.217± 0.004	3.49 ± 0.07	3643 ± 32

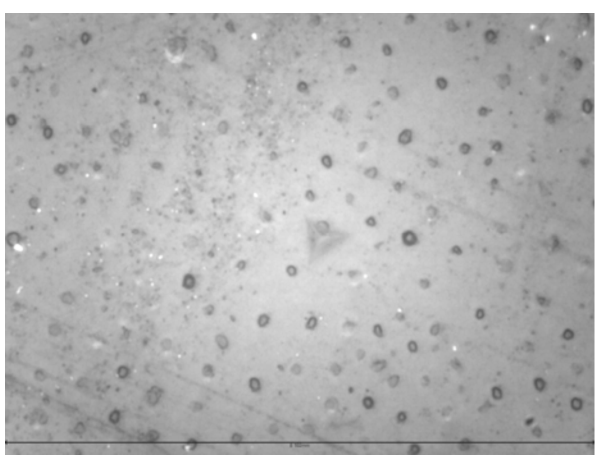


Detailed results- Dentin

	Dentin			
	Hardness [Vickers]	Hardness [GPa]	Young's Modulus [GPa]	Max Depth [nm]
1	87.5	0.926	18.3	1332
2	83.3	0.881	17.5	1361
3	79.4	0.841	18.2	1380
4	83.5	0.884	18.2	1353
5	81.3	0.861	17.2	1376
Average	**83.0**	**0.878**	**17.9**	**1360**
Standard Deviation	**3.0**	**0.032**	**0.5**	**19**



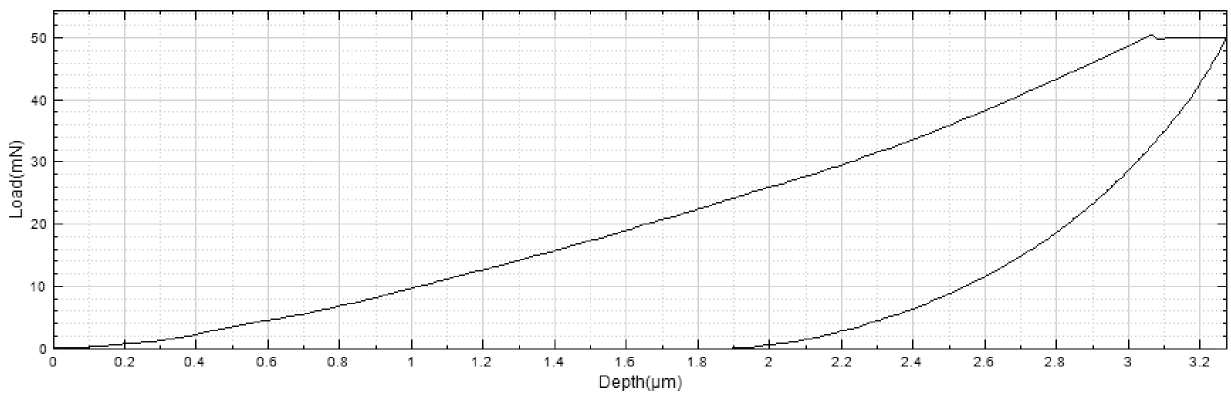
Loading Curve –Dentin, Indent 1



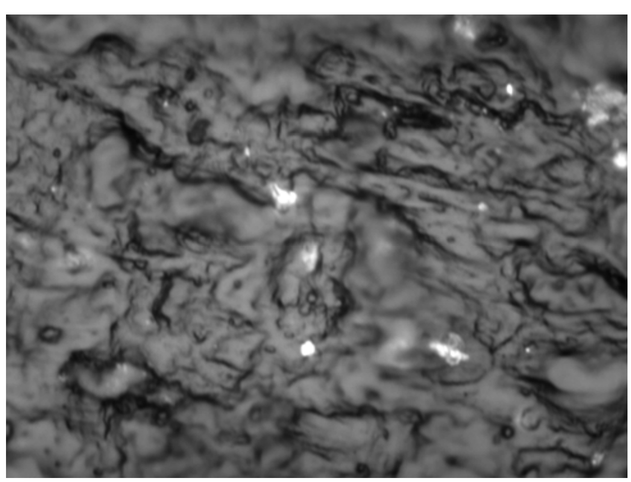
Micrograph of Indent 500X –Dentin, Indent 1

Detailed results – Decay

	Decay			
	Hardness [Vickers]	Hardness [GPa]	Young's Modulus [GPa]	Max Depth [nm]
1	29.0	0.307	5.07	3054
2	25.0	0.264	4.53	3268
3	18.9	0.200	4.13	3665
Average	**24.3**	**0.257**	**4.58**	**3329**
Standard Deviation	**5.1**	**0.054**	**0.47**	**310**



Loading Curve –Decay, Indent 1



Decay Area

Detailed results – Pulp

	Pulp			
	Hardness [Vickers]	Hardness [GPa]	Young's Modulus [GPa]	Max Depth [nm]
1	20.9	0.221	3.57	3606
2	20.3	0.215	3.44	3668
3	20.4	0.216	3.47	3656
Average	**20.5**	**0.217**	**3.49**	**3643**
Standard Deviation	**0.3**	**0.004**	**0.07**	**32**

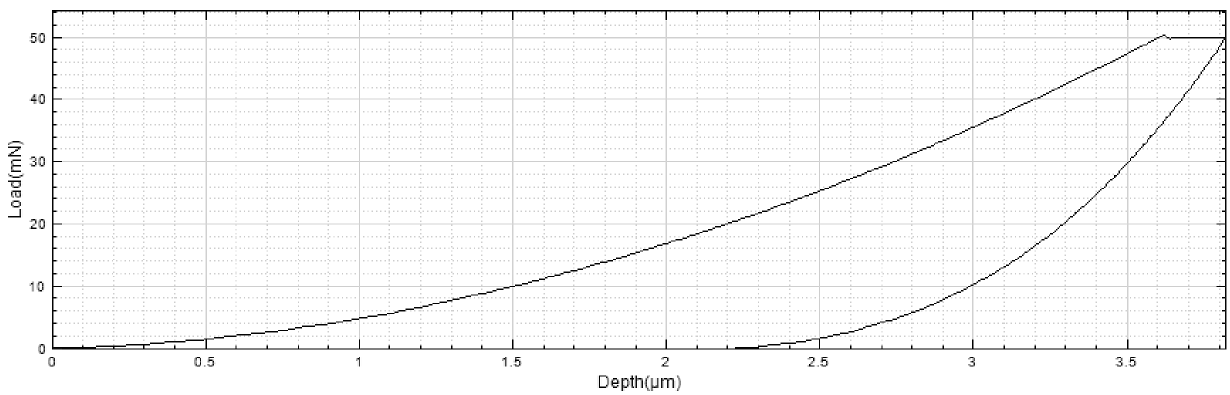
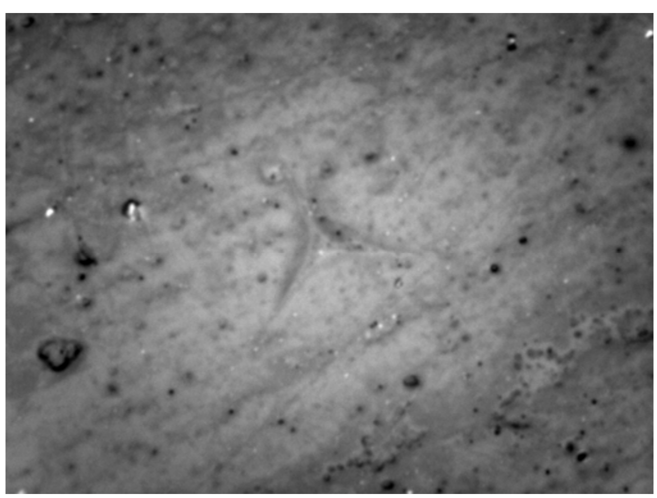


Figure 1: Loading Curve – Pulp, Indent 1



Pulp Indent

CONCLUSION

In conclusion, we have shown how the Nanovea Mechanical Tester, in Nanoindentation mode, provides precise measurement of the mechanical properties of a tooth. The data can be used in the development of fillings that will better match the mechanical characteristics of a real tooth. The positioning capability of the Nanovea Mechanical Tester allows full mapping of the hardness of the teeth across the various zones.

Using the same system, it is possible to test teeth material fracture toughness at higher loads up to 200N. Multi-cycle loading test can be used on more porous materials as to evaluate the remaining level of elasticity. Using a flat cylindrical diamond tip can give yield strength information in each zone. In addition, with DMA "Dynamic Mechanical Analysis", the viscoelastic properties including loss and storage moduli can be evaluated.

The Nanovea nano module is ideal for these tests because it uses a unique feedback response to control precisely the load applied. Because of this, the nano module can also be used to do accurate nano scratch testing. The study of scratch and wear resistance of tooth material and filling materials adds to the overall usefulness of the Mechanical tester. Using a sharp 2 micron tip to quantitatively compare marring on filling materials will allow better prediction of the behavior in real applications. Multi-pass wear or direct rotative wear testing are also common tests providing important information on the long term viability. To learn more about Nanovea Nanoindentation.