Biological Mechanical Properties Study of Oyster Shell

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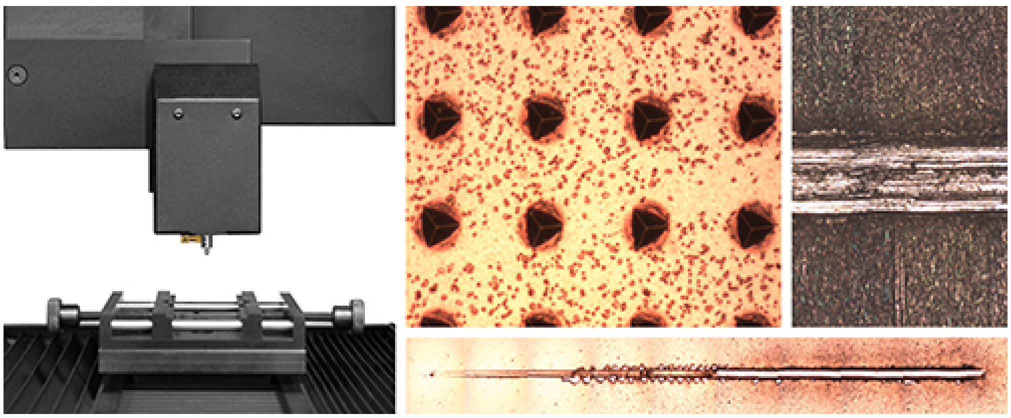
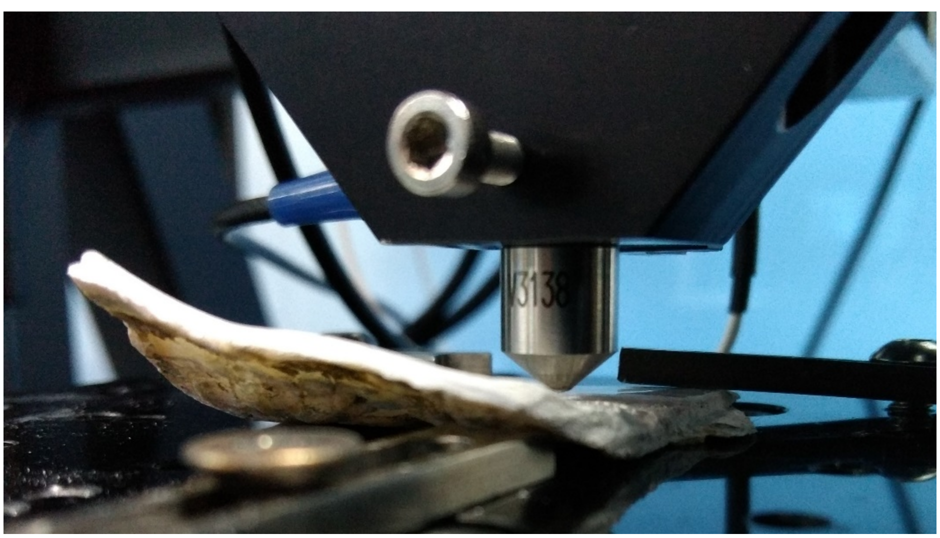
INTRO

As material science evolves, researchers have turned to biological materials for inspiration. The strong mechanical properties and unique structures of biological materials have been heavily researched in an attempt to replicate them. Desirable material property, such as hardness and flexibility, can be traced to how micro/nano structures are naturally formed. To demonstrate a quantifiable way of collecting data on material properties on biological material, the Nanovea Mechanical Tester's Micro module is used to perform indentation and coefficient of friction (COF) tests on different microstructures of an oyster shell.

IMPORTANCE OF MECHANICAL TESTING IN DETERMINING HARDNESS AND COF OF BIOLOGICAL MATERIALS

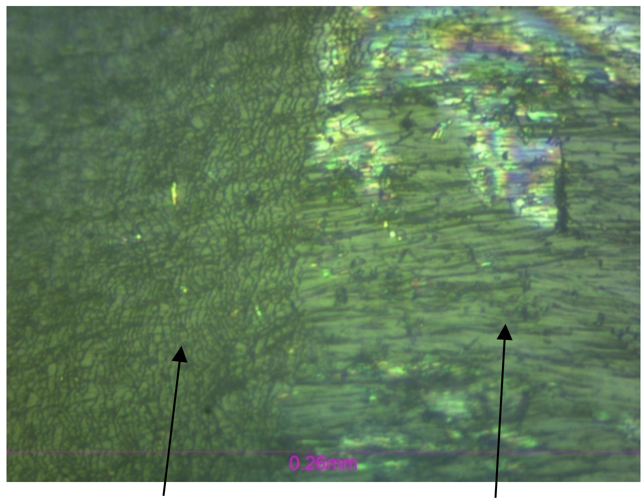


Nanovea Mechanical Tester's Micro Module is ideal for collecting local material properties on the surface of the sample. Insights on the relationships between microstructure to mechanical performance can be formed from the material properties each microstructure possess. This can provide information on the multifunctionality of the biological material and its benefits for the organism's survival in nature. These insights can also be used to help synthetically replicating biological materials.



MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester's Micro module is used to measure the hardness (GPa), Young's Modulus (E), and COF at different microstructures on the interior of an oyster shell. The different microstructures were observed and located under a microscope before indentation and COF testing was performed. The hardness, Young's Modulus, and COF were compared between the two observable microstructures.



Microstructure #1 Microstructure #2

TEST CONDITIONS AND PROCEDURE

The following indentation parameters were used:

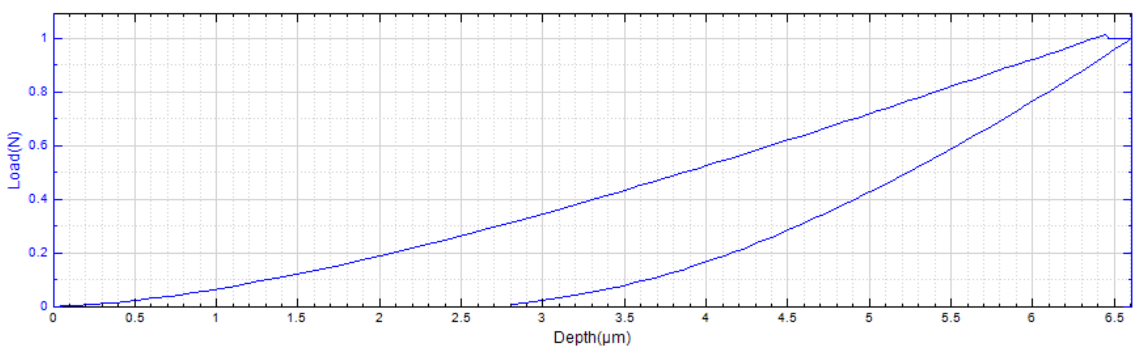
Test Parameters	All Tests
Maximum Force (N)	1
Loading Rate (N/min)	4
Unloading Rate (N/min)	4
Creep (s)	10
Computation Method	ASTM E-2546 & Oliver & Pharr
Indenter Type	Vickers

RESULTS: Microindentation

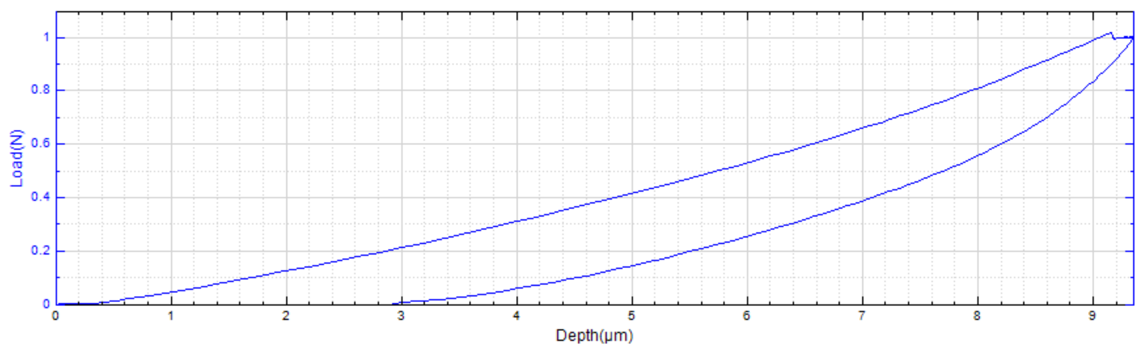
It should be noted that the layers beneath the microstructure can influence the hardness and Young's modulus. As a result, the values observed should not be taken as an absolute value, but comparative amongst the other microstructures. From the data gathered, Microstructure #2 shows a higher hardness than microstructure #1.

While the standard deviation for hardness and Young's modulus is large microstructure #2, the average values between the two microstructures are distinctly different from each other. oyster shell.

	Microstructure #1			
Test	Hardness [Vickers]	Hardness [MPa]	Young's Modulus [MPa]	Max Depth [um]
1	186.5	1973	15700	6.252
2	182.9	1935	15490	6.314
3	176.3	1866	15340	6.387
4	193.5	2047	14620	6.338
5	190.8	2020	15010	6.310
Average	**186.0**	**1968**	**15230**	**6.320**
Standard Deviation	**6.8**	**72**	**420**	**0.049**



Microstructure #2				
Test	Hardness [Vickers]	Hardness [MPa]	Young's Modulus [MPa]	Max Depth [um]
1	74.70	790.6	9184	9.041
2	73.91	782.2	8335	9.269
3	77.03	815.2	8584	9.101
4	68.63	726.3	10800	9.008
5	70.69	748.1	10040	8.985
Average	**72.99**	**772.5**	**9389**	**9.081**
Standard Deviation	**3.33**	**35.3**	**1027**	**0.114**



CONCLUSION: Microindentation

The Nanovea Mechanical Tester's Micro Module displays its ability to obtain hardness and Young's modulus on two different microstructures of an oyster shell. The two microstructures have distinct hardness and Young's modulus values, giving insight into the strength from the arrangement or composition of the microstructures themselves.

While only indentation tests were conducted in this study, these additional functionalities in the Nanovea Mechanical Tester may prove useful for other biological materials: Viscoelastic creep, martens hardness, ultimate yield strength, multi-cycle test, and fracture toughness. High sensitivity, low load tests can also be conducted with Nanovea Mechanical Tester's Nano Module.

TEST CONDITIONS AND PROCEDURE

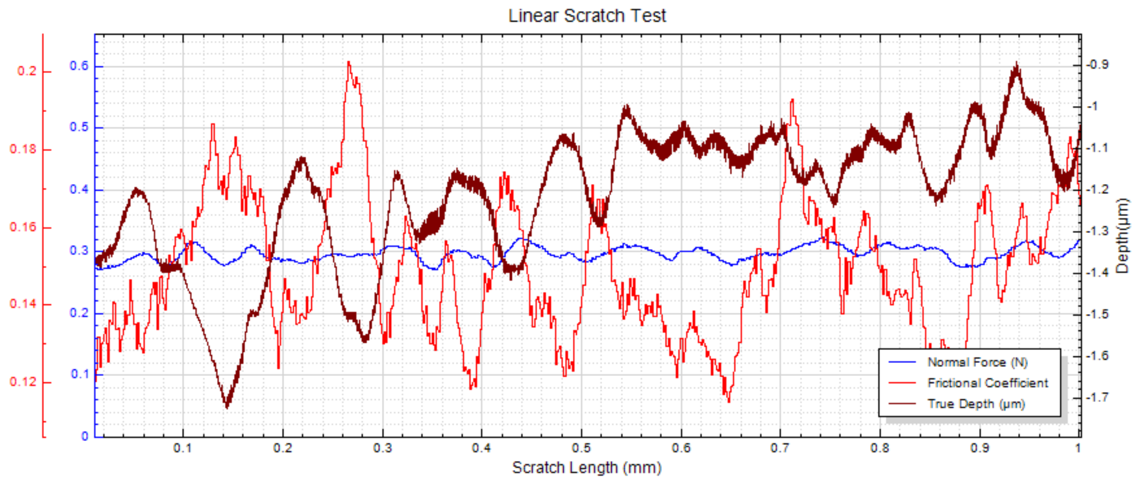
Test Parameters	All Tests
Load type	Constant
Applied Load (N)	0.3
Scratch Length (mm)	1
Scratching speed (mm/min)	1
Indenter geometry	Ball
Indenter material	52100 Steel
Ball diameter (mm)	3

RESULTS: Coefficient of Friction

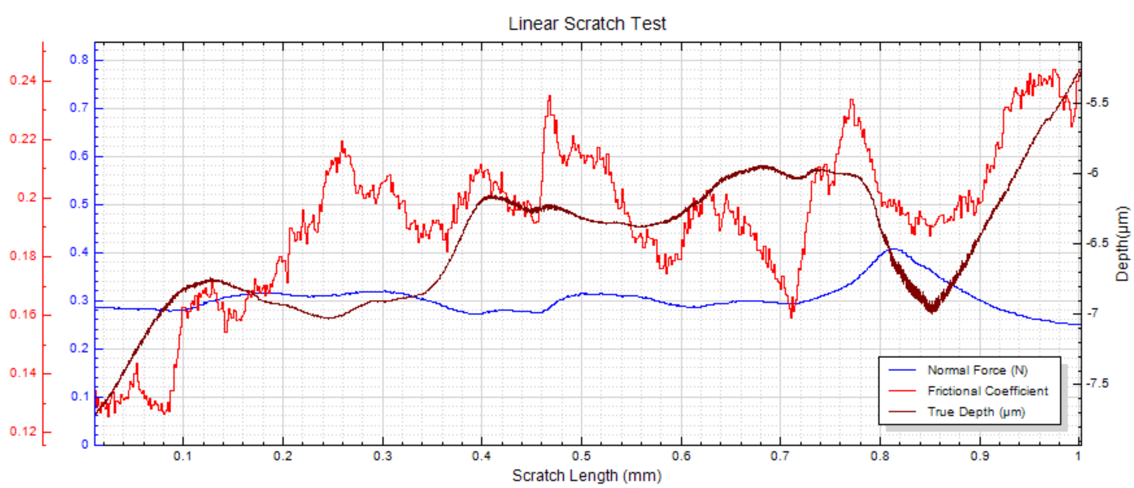
A coefficient of friction test was conducted by running a linear constant load scratch test. When targeting COF properties, minimal damage and penetration to the sample is desired to minimize influence beyond the surface. As a result, a low load of 300mN and an indenter that will give a large contact area was used.

The results show microstructure #1 has a larger average COF than microstructure #3. Standard deviation for each microstructure is low, showing high consistency for the data collected.

Microstructure #1			
Test	Max COF	Min COF	Average COF
1	0.2439	0.1254	0.1920
2	0.2558	0.1270	0.1971
3	0.2731	0.1523	0.2241
Average	**0.2576**	**0.1349**	**0.2044**
Standard Deviation	**0.0120**	**0.0123**	**0.0141**



Microstructure #2			
Test	**Max COF**	**Min COF**	**Average COF**
1	0.1903	0.0806	0.1466
2	0.2028	0.1133	0.1492
3	0.2098	0.0948	0.1394
Average	**0.2010**	**0.0962**	**0.1451**
Standard Deviation	**0.0081**	**0.0134**	**0.0042**



Conclusion: Coefficient of Friction

The study on the COF of different microstructures yielded values of 0.2044 ± 0.0141 and 0.1451 ± 0.0042 for microstructure #1 and #2 respectively. From the tests conducted, the Nanovea Mechanical Tester's Micro Module has successfully characterized the COF on the two microstructures of the oyster shell.

In this study, the COF was gathered under normal, dry conditions. The Nanovea Mechanical Tester can also include other add-ons to conduct testing under different environmental conditions, including lubrication, humidity, and temperature. These testing conditions can also be applied to indentation testing.

Learn more about the Nanovea Mechanical Testers or Lab Services

APPENDIX: MEASUREMENT PRINCIPLE

The micro-Hardness test is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an 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 partial or complete relaxation occurs. This procedure is performed repetitively; at each stage of the experiment the position of the indenter relative to the sample surface is precisely monitored with an optical non-contact depth sensor.

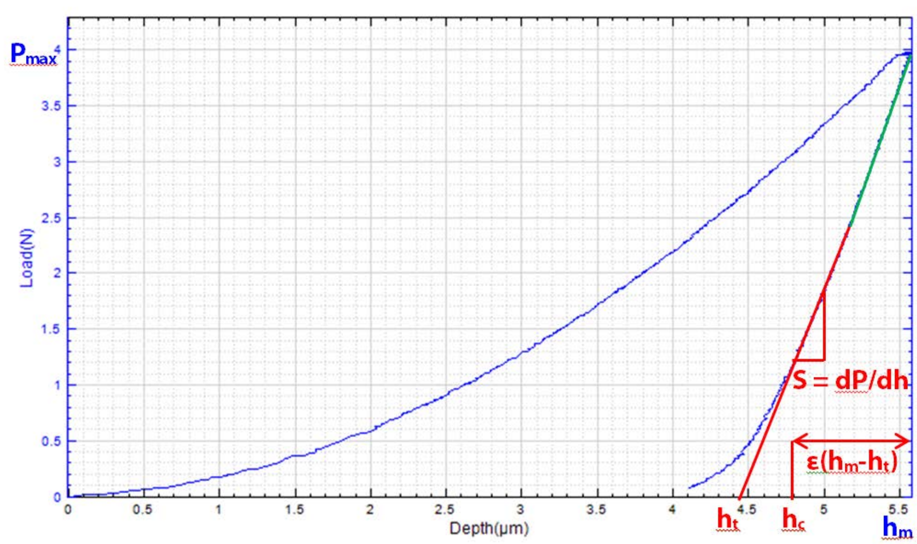
For each loading/unloading cycle, the applied load value is plotted with respect to the corresponding position of the indenter. The resulting load/displacement curves provide data specific to the mechanical nature of the material under examination. Established models are used to calculate quantitative hardness and modulus values for such data.

The MHT is especially suited to perform tests of penetration depths in the micrometer scale and has the following specifications:

Displacement measurement:	White Light Chromatic Confocal
XY Lateral Accuracy:	0.25 μm
Maximum indenter range:	300 μm
Load application:	Z motor controlled with force feedback
loop	
Load range	0 – 40 N | 0 – 400 N
Minimum Contact Load	3 mN | 30 mN
Minimum load:	30 mN | 300 mN
Maximum load:	40 N | 400N
Contact force hold time:	unlimited

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:

$$h_c = h_{\max} - {}^{3P_{\max}}\!/_{4S}$$

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= - (π)(h_c)^2 + 2(π)R(h_c) where R is the radius of the indenter

Other parameters calculated during standard instrumented indentation include:
Work of Indentation; Volume of indent; Maximum Stress and Maximum Strain; Plastic Work and Elastic Work; % Plastic work in relation to the total work of indentation

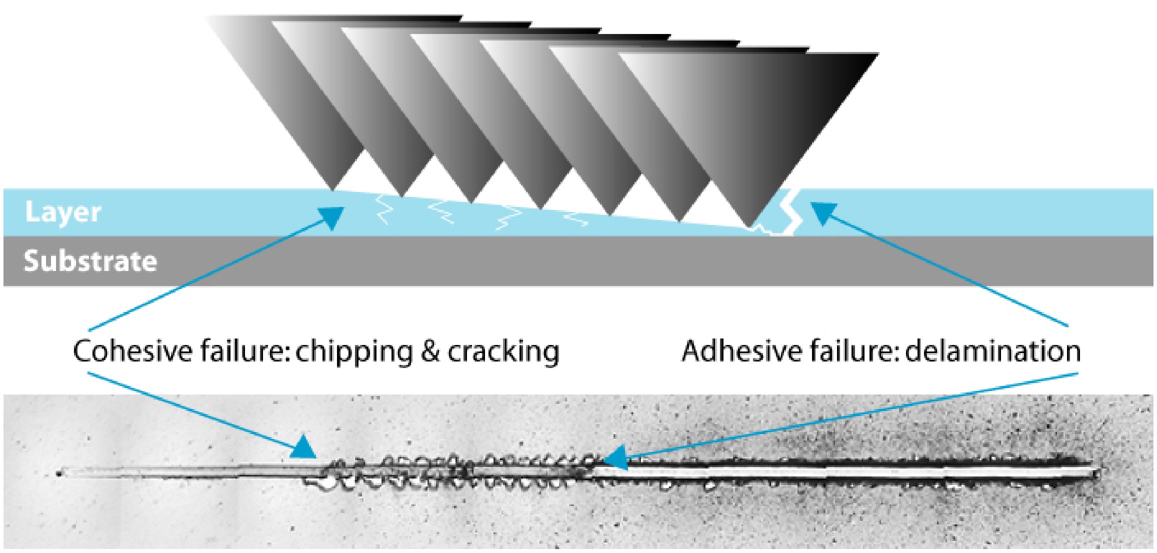
Theory of Scratch Testing

Principle

The scratch testing method is a quantitative test in which critical loads at which failures appear in the samples are used to evaluate the relative cohesive or adhesive properties of a coating or the scratch resistance of a bulk material. During the test, scratches are made on the sample with a sphero-conical stylus which is drawn at a constant speed across the sample, under a constant load, or, more commonly, a progressive load with a fixed loading rate. Sphero-conical styluses are available with different radii (which describes the “sharpness” of the stylus). Common radii are from 20 to 200µm for micro/macro scratch tests, and 1 to 20µm for nano scratch tests.

When performing a progressive load test, the critical load is defined as the smallest load at which a recognizable failure occurs. In the case of a constant load test, the critical load corresponds to the load at which a regular occurrence of such failure along the track is observed.

Progressive load measuring depth, friction & acoustic emission



Comments on the critical load

The scratch test is a quantitative test with high repeatability. The critical load depends on the mechanical strength (adhesion, cohesion) of a combined coating-substrate system but also on several other parameters. Some of them are directly related to the test itself, while others are related to the coating-substrate system.

Test parameters affecting critical load:

- Loading rate
- Scratching speed

- Indenter tip radius
- Indenter material (and also indenter tip wear)

Sample specific parameters affecting critical load:

- Friction coefficient between surface and indenter
- Internal stresses in the material
- Substrate hardness and roughness
- Coating hardness and roughness
- Coating thickness

By keeping the test parameters constant one can obtain very repeatable data to quantifiably compare samples.

Means for critical load determination

Microscopic observation is the most reliable method to detect surface damage. This technique is able to differentiate between cohesive failure within the coating and adhesive failure at the interface of the coating-substrate system.
The friction force recording enables the force fluctuations along the scratch to be studied and correlated to the failures observed under the microscope. Typically, a failure in the sample will result in a change (a step, or a change in slope) in coefficient of friction. Frictional responses to failures are very specific to the coating-substrate system in study.

The depth sensor recording can also sometimes indicate where a failure occurs. Typically, a significant fall in the depth will indicate that the indenter has broken through one layer of a sample down to the next. The depth recording can also be used to study deformation of a sample surface. Plastic and elastic deformation can be studied by performing pre- and post-scans of the scratch.