AMMONITE FOSSIL MECHANICAL PROPERTIES USING NANOINDENTATION

Prepared by
Duanjie Li, PhD

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

INTRODUCTION

As remained traces of plants, animals and other organisms once lived on the ancient earth, fossils preserve information of the evolution of life. The oldest fossils can be traced back as far as the Archaean Eon, about 3.48 billion years old[i ii]. Paleontology studies the formation mechanism and evolutionary relationships of fossils across geological time. The fossilization process includes several major mechanisms depending on the tissue type and external conditions, such as permineralization, casts and molds, authigenic mineralization, replacement and recrystallization.

IMPORTANCE OF NANOINDENTATION FOR FOSSIL STUDY

Fossils possess different mechanical properties determined by the stage and mechanism of the fossilization process. For instance, shells and bones can be replaced with different types of minerals when they are exposed to various environment. At different stages of the recrystallization process, the hardness of the fossil changes as well.

Nanoindentation precisely measures the mechanical behaviors of materials at small scales[iii iv]. The high-resolution load-displacement curves from the nanoindentation measurement can provide a variety of physicomechanical properties, including hardness, Young's modulus, creeping, fracture toughness and many others. A mechanical property mapping of the fossil sample can play a key role in determining the mechanism and stages of the fossilization process. The small indentation force applied using the nanoindentation technique provides an ideal non-destructive mechanical testing solution to prevent damage to fossils under the test.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Nanoindentation mode is used to map the mechanical properties of different locations of an ammonite fossil sample. We would like to showcase the capacity of Nanovea Mechanical Tester in performing nanoindentation mapping on a fossil sample with high precision and reproducibility.

Fig. 1: Nanoindentation tip on the ammonite fossil sample.

TEST CONDITIONS

The Nano Module of the Nanovea Mechanical Tester was applied perform a series (3×3, spaced 0.08 mm apart, 9 indents in total) of nanoindentation on the top of an ammonite fossil sample using a Berkovich indenter. All indentations were performed to a maximum load of 50 mN. The distribution of the indentations is illustrated in Fig. 2 and the test conditions are summarized in Table 1.

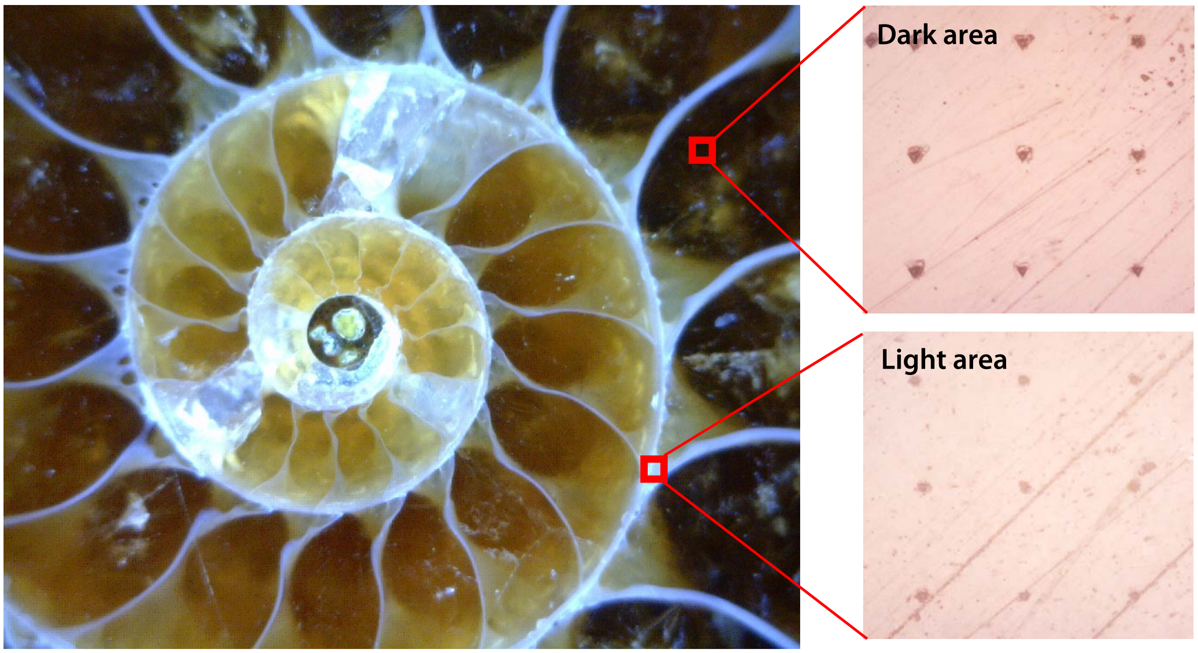

Fig. 2: Location of indentations on the ammonite fossil sample.

Maximum force (mN)	50
Loading rate (mN /min)	100
Unloading rate (mN /min)	100
Creep (s)	5
Indenter type	Berkovich

Table 1: Test conditions of the nanoindentation mapping.

RESULTS AND DISCUSSION

The images of the 3×3 indentation matrix in the Dark and Light areas are displayed in Fig. 2. Two representative load-displacement curves of the indentations on the dark and light areas are shown in Fig. 3, and the corresponding hardness and Young's Modulus calculated using Oliver and Pharr Method [v] are summarized and compared in Fig. 4. The light area possesses an average hardness of ~4.9 GPa and Young's modulus of ~90.4 GPa, compared to ~4.1 GPa and ~87.2 GPa, respectively, for the dark area.

Such a 3×3 indentation matrix was carried out with superior accuracy and repeatability, thanks to the high-precision piezo load control and the direct load/displacement measurement by the

Nanovea Mechanical Tester. The excellent position control of the sample stage allows users to precisely pinpoint the target area for mechanical properties mapping. This makes mapping of the mechanical properties substantially less time-consuming and user friendly. It provides a solution of detecting different materials, phases and fossilization stages on different locations of the fossil sample.

We welcome further discussion and comments on the correlation of the hardness/Young's modulus and the stage of fossilization and recrystallization, which will be a useful tool that provides insight into the mechanism of fossil formation and the fossil's age.

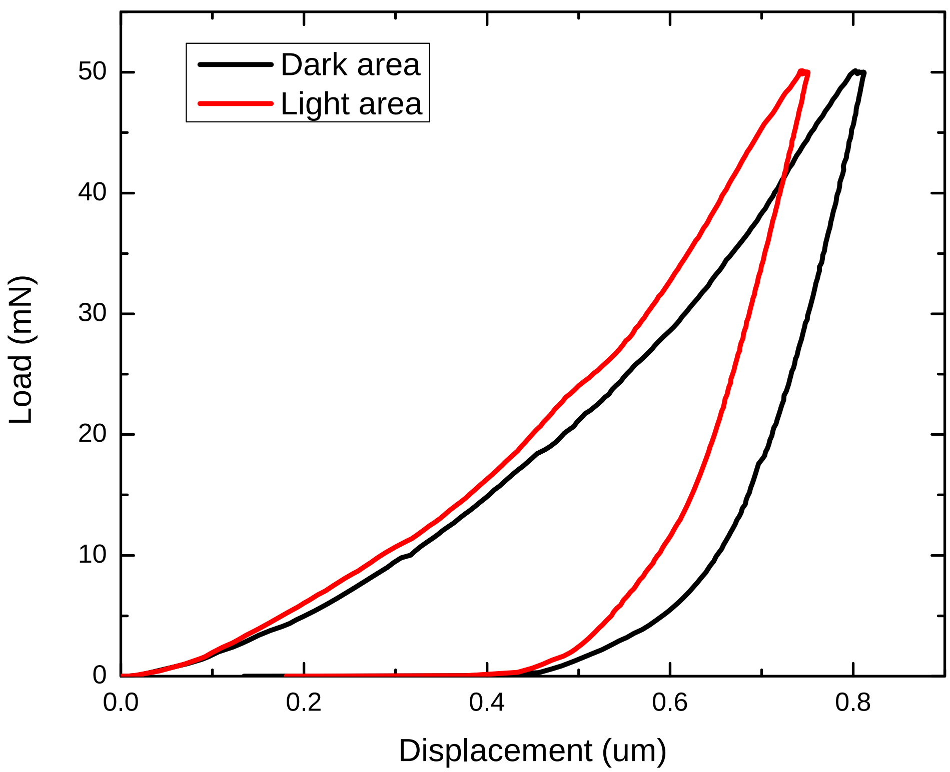

Fig. 3: Load-displacement curves of the indentations.

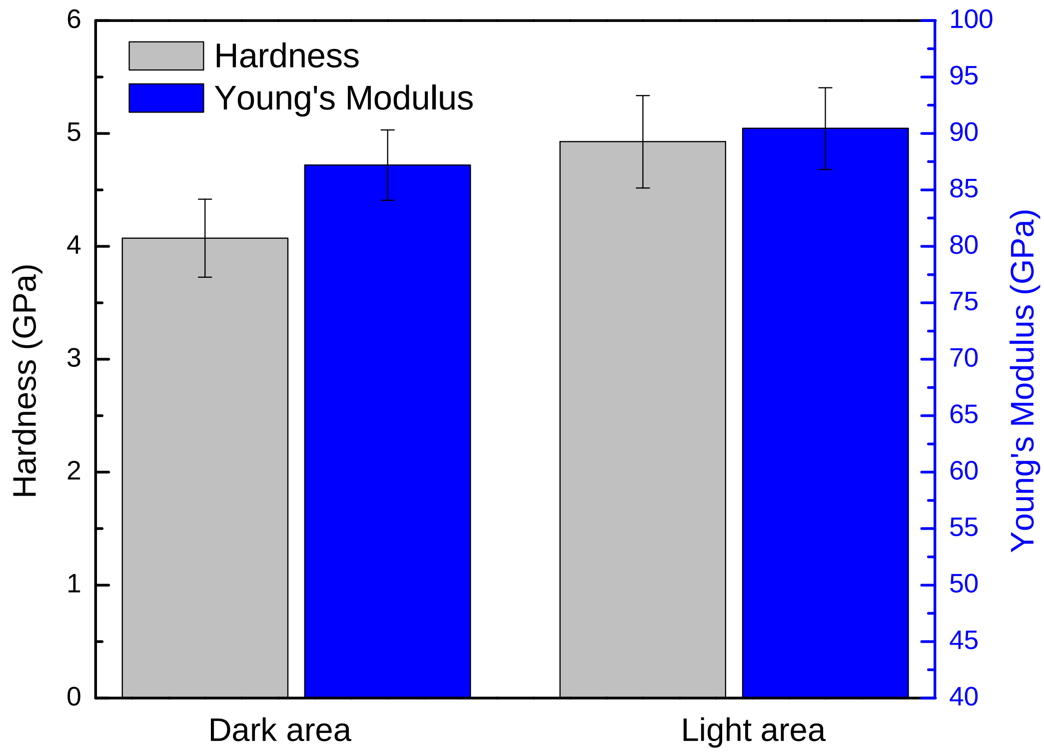

Fig. 4: Hardness and Young's Modulus at the Dark and Light areas of the fossil sample.

CONCLUSION

In this study, we showcased the capacity of Nanovea Mechanical Tester in performing nanoindentation mapping on a fossil sample. The precise position control and accurate measurement of hardness and Young's modulus enable identification of the mechanical properties of the materials at different locations on the fossil surface, which may be related to the stage of fossilization and recrystallization, as well as the fossil's age. The tested ammonite fossil sample possesses varied hardness from 4.1 to 4.9 GPa at different locations.

The Nano, Micro or Macro modules of the Nanovea Mechanical Tester all include ISO and ASTM compliant indentation, scratch and wear tester modes, providing the widest and most user friendly range of testing available in a single system. Nanovea's unmatched range is an ideal solution for determining the full range of mechanical properties of thin or thick, soft or hard coatings, films and substrates, including hardness, Young's modulus, fracture toughness, adhesion, wear resistance and many others.

In addition, optional 3D non-contact profiler and AFM Module are available for high resolution 3D imaging of indentation, scratch and wear track in addition to other surface measurements such as roughness.

To learn more about Nanovea Mechanical Tester or Lab Services.

APPENDIX: MEASUREMENT PRINCIPLE

Nanoindentation 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 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 or 250 μm
Depth Resolution (Theoretical)	: 0.003 nm
Depth Resolution (Noise Level)	: 0.15 nm
Maximum force	: 400 mN
Load Resolution (Theoretical)	: 0.03 μN
Load Resolution (Noise Floor)	: 0.3 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.

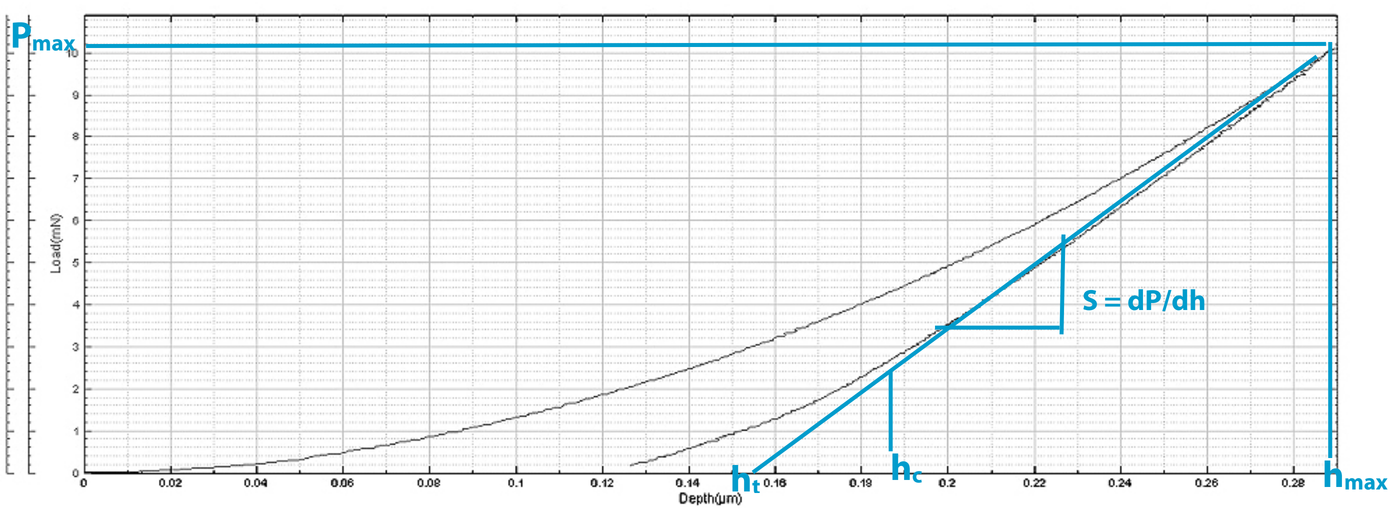

Fig. 5: Load-displacement curve of nanoindentation.

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's ratio of the indenter and ν the Poisson's ratio 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 A_c 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=2πRh_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 is a better choice.

Other possible measurements by Nanovea Mechanical Tester:

Stress-Strain & Yield Stress, Fracture Toughness, Compression strength, Fatigue testing and many others.

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[i] **Noffke, Nora; Christian, Christian; Wacey, David; Hazen, Robert M., Astrobiology, 13 (12) pp, 1103–24**

[ii] **Borenstein, Seth. Oldest fossil found: Meet your microbial mom, Associated Press. 15 November 2013.**

[iii] **Oliver, W. C.; Pharr, G. M., Journal of Materials Research., Volume 19, Issue 1, Jan 2004, pp.3-20**

[iv] **Schuh, C.A., Materials Today, Volume 9, Issue 5, May 2006, pp. 32–40**

[v] **Oliver, W. C.; Pharr, G. M., Journal of Materials Research, Volume 7, Issue 6, June 1992, pp.1564-1583**