MECHANICAL PROPERTIES OF HYDROGEL USING NANOINDENTATION

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INTRO

Hydrogel is known for its super absorbency of water allowing for a close resemblance in flexibility as natural tissues. This resemblance has made Hydrogel a common choice not only in biomaterials, but also in electronics, environment and consumer good applications such as contact lens. Its usage in different applications has their own unique needs and requirements. One of the essential requirements is known and controlled mechanical properties.

USING NANOINDENTATION FOR HYDROGEL

Hydrogels create unique challenges to Nanoindentation including test parameters and sample preparation. Many nanoindentation systems have major limitations since they were not originally designed for such soft materials. For example, some of the nanoindentation systems use a coil/magnet assembly to apply force on the sample. There is no actual force measurement, leading to inaccurate and non-linear loading when testing soft materials. Determining the point of contact is extremely difficult since the depth is the only parameter actually being measured. It is almost impossible to observe the change of slope in the depth vs time plot during the approach of the tip to soft materials. In order to overcome the limitations of these systems, the nano module of the Nanovea Mechanical Tester measures the force feedback with an individual load cell to ensure high accuracy on all types of materials, soft or hard. The piezo-controlled displacement is extremely precise and fast. This allows unmatched measurement of viscoelastic properties by eliminating many theoretical assumptions that systems with a coil/magnet assembly and no force feedback must account for.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Nanoindentation mode, is used to study the hardness, elastic modulus and creep of a Hydrogel sample.

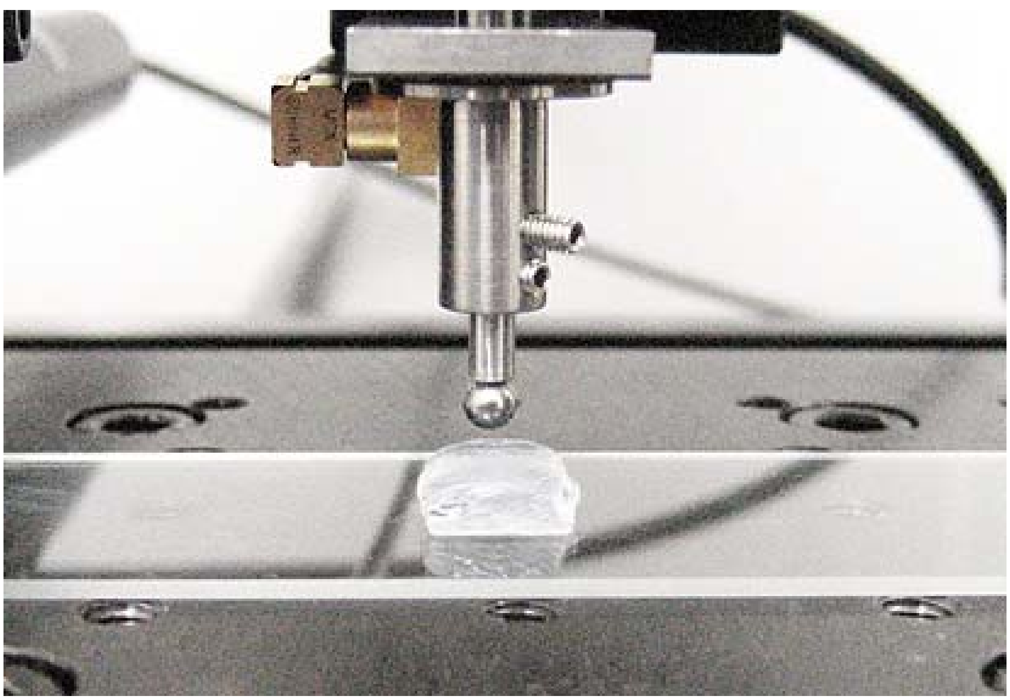


Fig. 1: Ball indenter on the hydrogel sample.

TEST CONDITIONS

A hydrogel sample placed on a glass slide was tested by nanoindentation technique using Nanovea Mechanical Tester. For this soft material a 3 mm diameter spherical tip was used. The load linearly increased from 0.06 to 10 mN during the loading period. The creep was then measured by the change of indentation depth at the maximum load of 10 mN for 70 s. The test conditions are summarized in Table 1.

Approach Speed	100 µm/min
Contact Load	0.06 mN
Max Load	10 mN
Loading Rate	20 mN/min
Creep	70 s
Indenter Type	Spherical, 3 mm diameter

Table 1: Test conditions of the nanoindentation on hydrogel.

RESULTS AND DISCUSSION

The evolution of the load and depth as a function of time is shown in Fig. 2. It can be observed that on the plot of the depth vs time (Fig. 2b), it is very difficult to determine the point of the change of slope at the beginning of the loading period, which usually works as an indication where the indenter starts to contact the soft material. However, the plot of the load vs time (Fig. 2a) shows the peculiar behavior of the Hydrogel under an applied load. As the hydrogel begins to get in touch with the ball indenter, the Hydrogel pulls the ball indenter due to its surface tension, which tends to decrease the surface area. This behavior leads to the negative measured load at the beginning of the loading stage. The load progressively increases as the indenter sinks into the Hydrogel, and it is then controlled to be constant at the maximum load of 10 mN for 70 s to study the creep behavior of the Hydrogel.

(a) Load vs. time (b) Depth vs. time

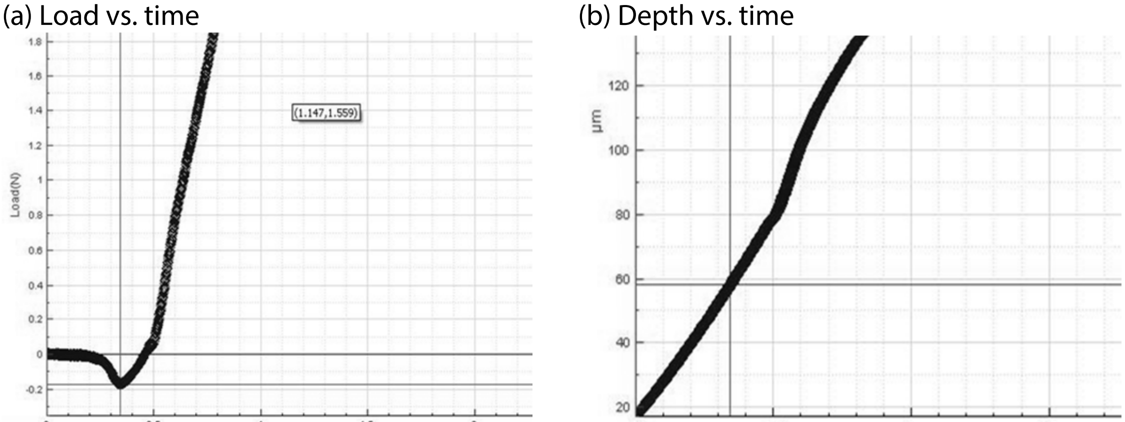


Fig. 2: Evolution of the load and depth as a function of Time.

The load vs. displacement plot of the nanoindenation test on the Hydrogel sample is shown in Fig. 3 and the plot of the creep depth vs time is shown in Fig. 4. The hydrogel in this study possesses a hardness of 16.9 KPa and a Young's modulus of 160.2 KPa, as calculated based on the load-displacement curve using the Oliver-Pharr method.

Creep is a very important factor for the mechanical properties of the Hydrogel. The close-loop feedback control between piezo and ultrasensitive load cell ensures a true constant loading during the creep time at the maximum load. As shown in Fig. 4, the Hydrogel subsides ~ 42 μm as a result of creep in 70 s under the 10 mN maximum load by the 3mm ball tip.

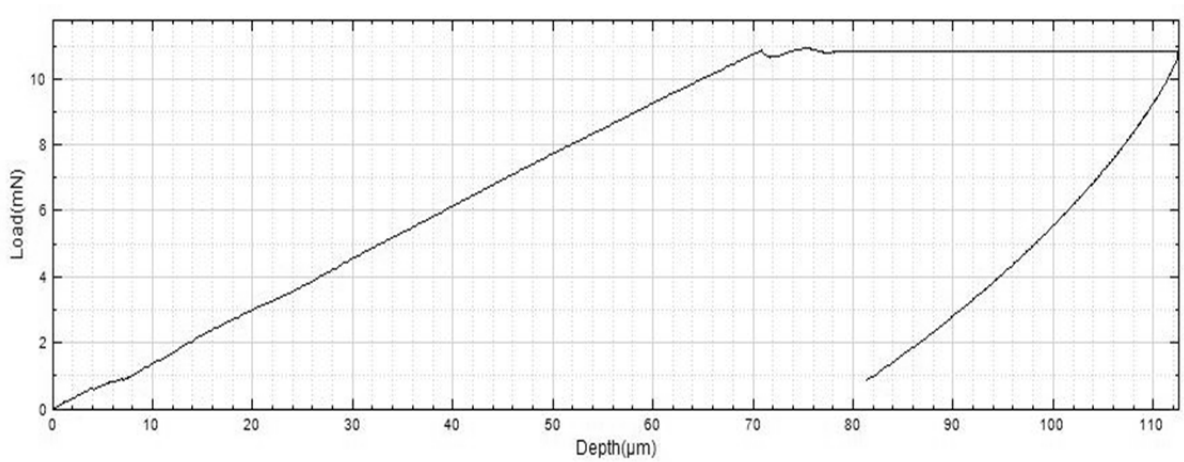


Fig. 3: The load vs. displacement plot of the Hydrogel.

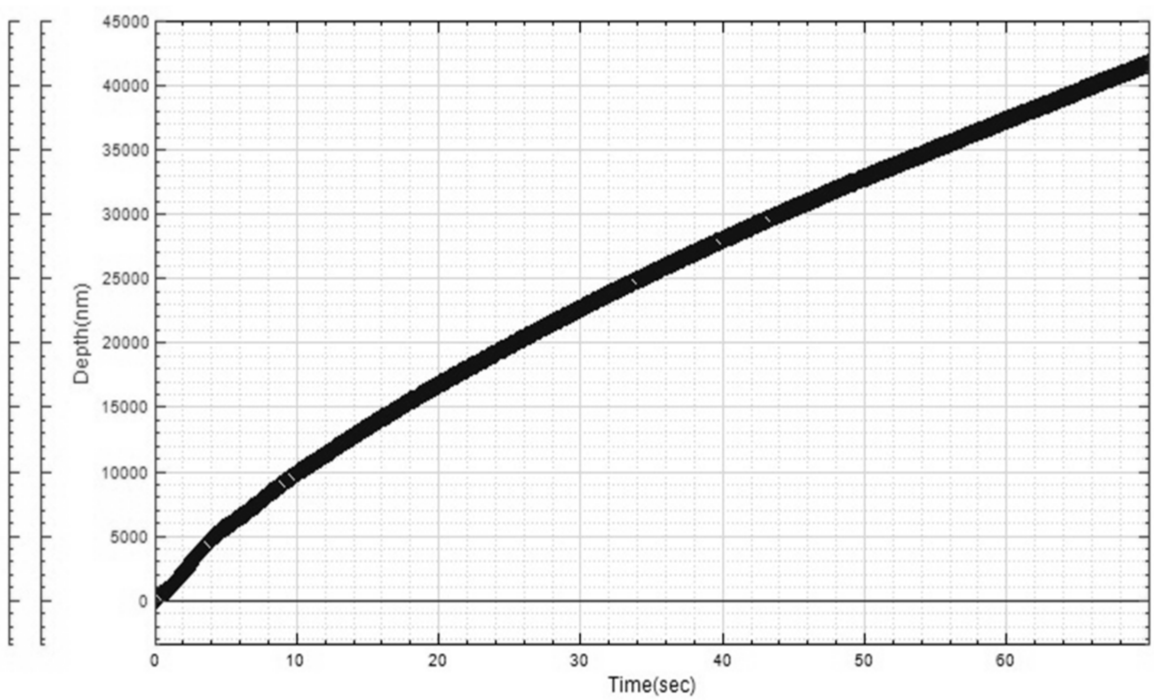


Fig. 4: Creeping at a maximum load of 10 mN for 70 s.

CONCLUSION

In this study, we showcased that Nanovea Mechanical Tester, in Nanoindentation mode, provides precise and repeatable measurement of the mechanical properties of Hydrogel including hardness, Young's modulus and creep. The large 3mm ball tip ensures proper contact against the Hydrogel surface. High precision motorized sample stage allows accurate positioning of the flat face of the Hydrogel sample under the ball tip. The Hydrogel in this study exhibits a hardness of 16.9 KPa and a Young's modulus of 160.2 KPa. The creep depth is ~42 µm under a 10 mN load for 70 s.

The Nanovea Mechanical Testers provide unmatched multi-function Nano and Micro/Macro modules on a single platform. Both the Nano and Micro/Macro modules include scratch tester, hardness tester and wear tester modes, providing the widest and most user friendly range of testing available on a single module.

To learn more about Nanovea Mechanical Tester or Lab Services.

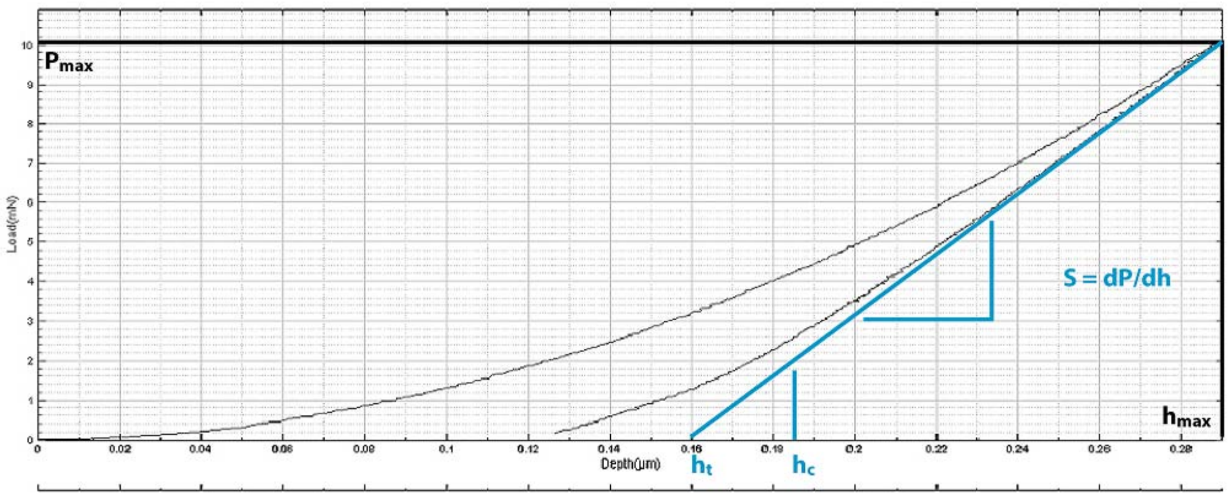
APPENDIX: MEASUREMENT PRINCIPLE

Nanoindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an 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 or 250µm
Depth Resolution (Theoretical)	: 0.003 nm
Depth Resolution (Noise Level)	: 0.05 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:

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

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=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 might be a better choice.