NANOINDENTATION OF
Polypropylene Film

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INTRO

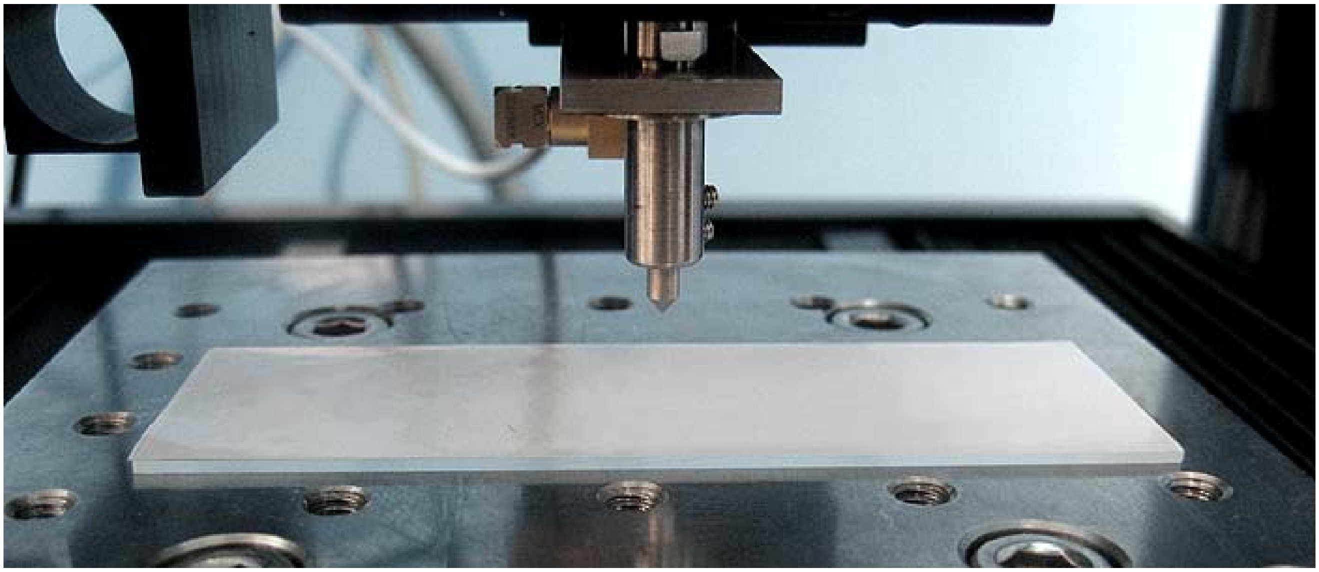
Polypropylene film is a thermoplastic polymer used in a wide variety of applications, including packaging materials, clothing, protective films, medical materials and many others. Polypropylene typically has an intermediate level of crystallinity and Young's modulus and it is much less brittle than competing polymers. The light weight and favorable characteristics of Polypropylene film has brought much attention to further investigate its capabilities and applications.

IMPORTANCE OF NANOINDENTATION FOR QUALITY CONTROL AND R&D APPLICATIONS

Standard Vickers hardness test fail to provide adequate hardness data because of the elastic response soft materials. Therefore Nanoindentation provides accurate hardness data as well as other studies such as elastic modulus and creep behavior of Polypropylene films. The intended hardness and Young’s Modulus of Polypropylene film can be linked to the success of the film when used in a specific application. Therefore the ability to understand and control the hardness and elastic modulus is crucial to the final product.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Nanoindentation Tester (seen below) is used to measure the Vickers hardness (Hv) and Young’s Modulus (E) of Polypropylene film. The Polypropylene film average value thickness is 20μm and the film was glued on a glass slide for stability. A Berkovitch tip was used to determine the property of the film itself. A spherical tip could have been used to analyze the compression property of the film which can be a different approach to this study. This would be very useful in cases where the material would have a distinct texture (such as honeycomb) on the surface. This was not the case with the samples tested in this study.



MEASUREMENT PRINCIPLE

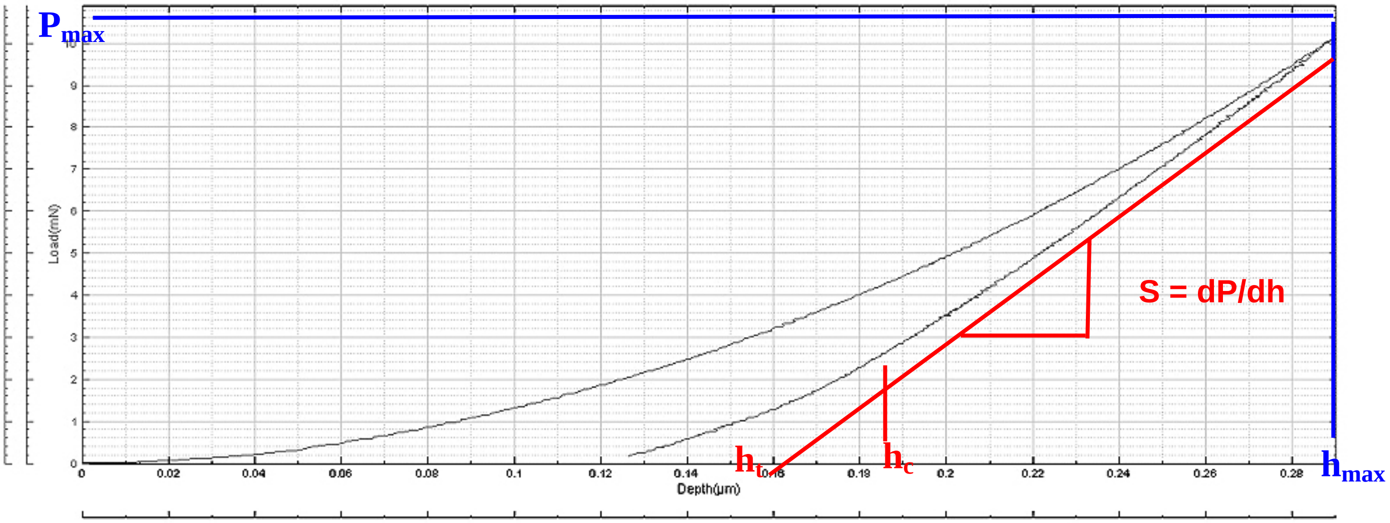
Nanoindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an already established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set 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 $A_c=2\pi Rh_c$ 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:

Work of Indentation, Volume of indent, Plastic & Elastic Work, Visco Elastic , Creep, Compression and others.

Elastic indentation Behavior (indenter geometry)	**m (power law exponent)**	**ε**
Flat	1	1
Paraboloid	1.5	0.75
Conical	2	0.72

Calculation of Young's Modulus and Hardness

Young's Modulus

The reduced modulus, E_r, is given by:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}} = \frac{\sqrt{\pi}}{2}\frac{1}{C}\frac{1}{\sqrt{A_c}}$$

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.

Hardness

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

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

Test conditions and procedure

The following indentation parameters were used:

Maximum force (mN)	20
Loading rate (mN/min)	40
Unloading rate (mN/min)	40

Creep (s)	20
Computation Method	ASTM E-2546 & Martens Hardness
Indenter type	Berkovich Diamond KM

Results

These full results present the measured values of Hardness and Young’s modulus as the penetration depth (d) 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.

	Polypropylene			
	Hv [Vickers]	**H [GPa]**	**E [GPa]**	**Δ d [µm]**
1	4.24	0.04	0.77	5014.86
2	3.95	0.04	0.78	5131.69
3	4.18	0.04	0.85	4968.30
4	4.28	0.05	0.77	4998.50
5	4.16	0.04	0.88	4955.42
Average	**4.16**	**0.04**	**0.81**	**5013.75**
Standard deviation	**0.13**	**0.00**	**0.05**	**70.02**

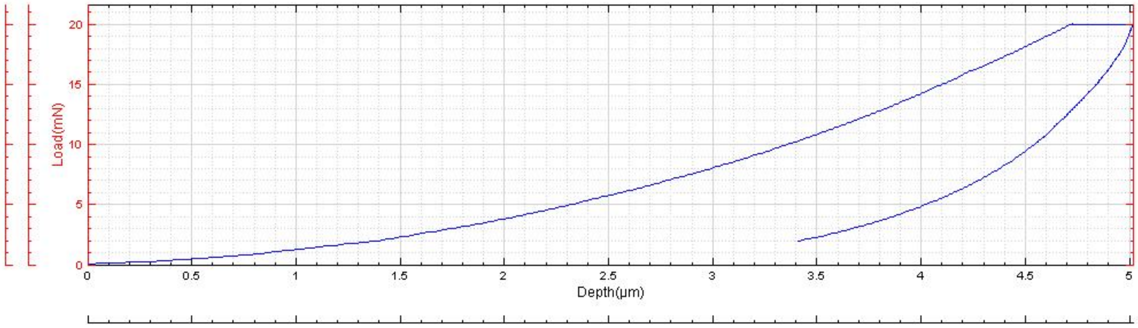


Figure 1: Loading Curve - Polypropylene, Indent 1

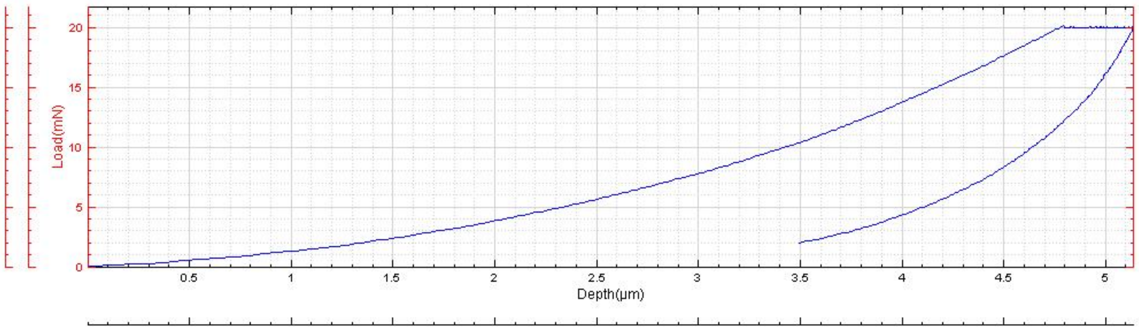


Figure 2: Loading Curve - Polypropylene, Indent 2

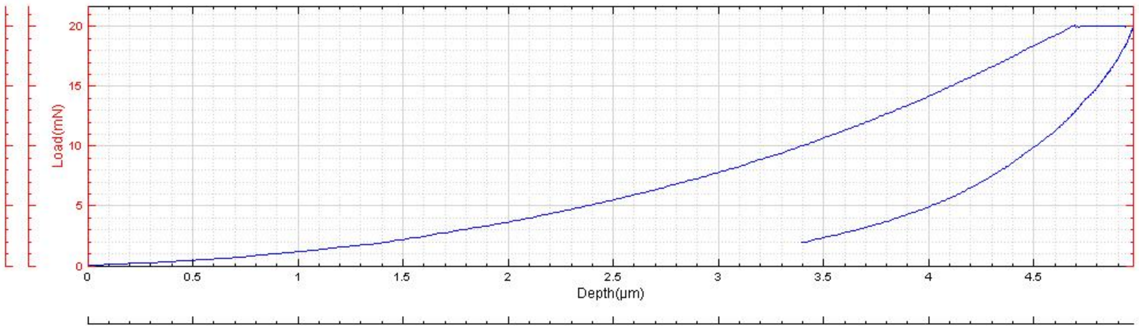


Figure 3: Loading Curve - Polypropylene, Indent 3

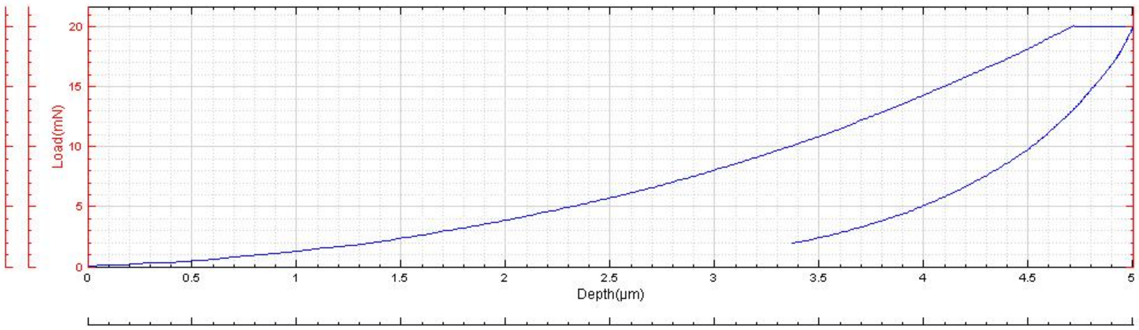


Figure 4: Loading Curve - Polypropylene, Indent 4

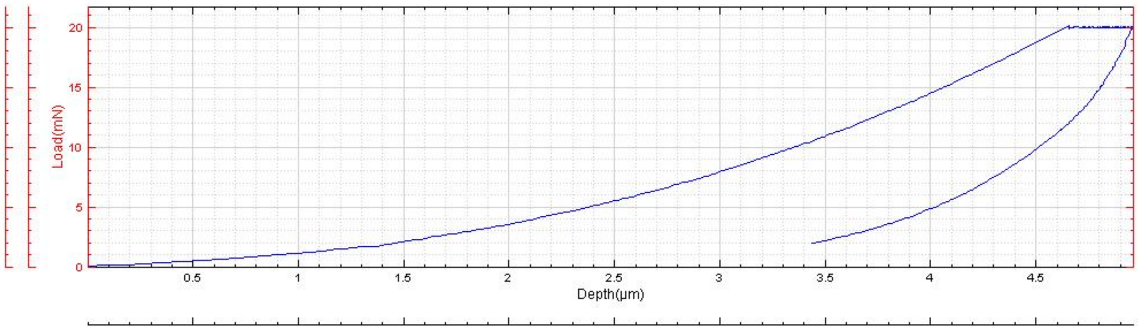
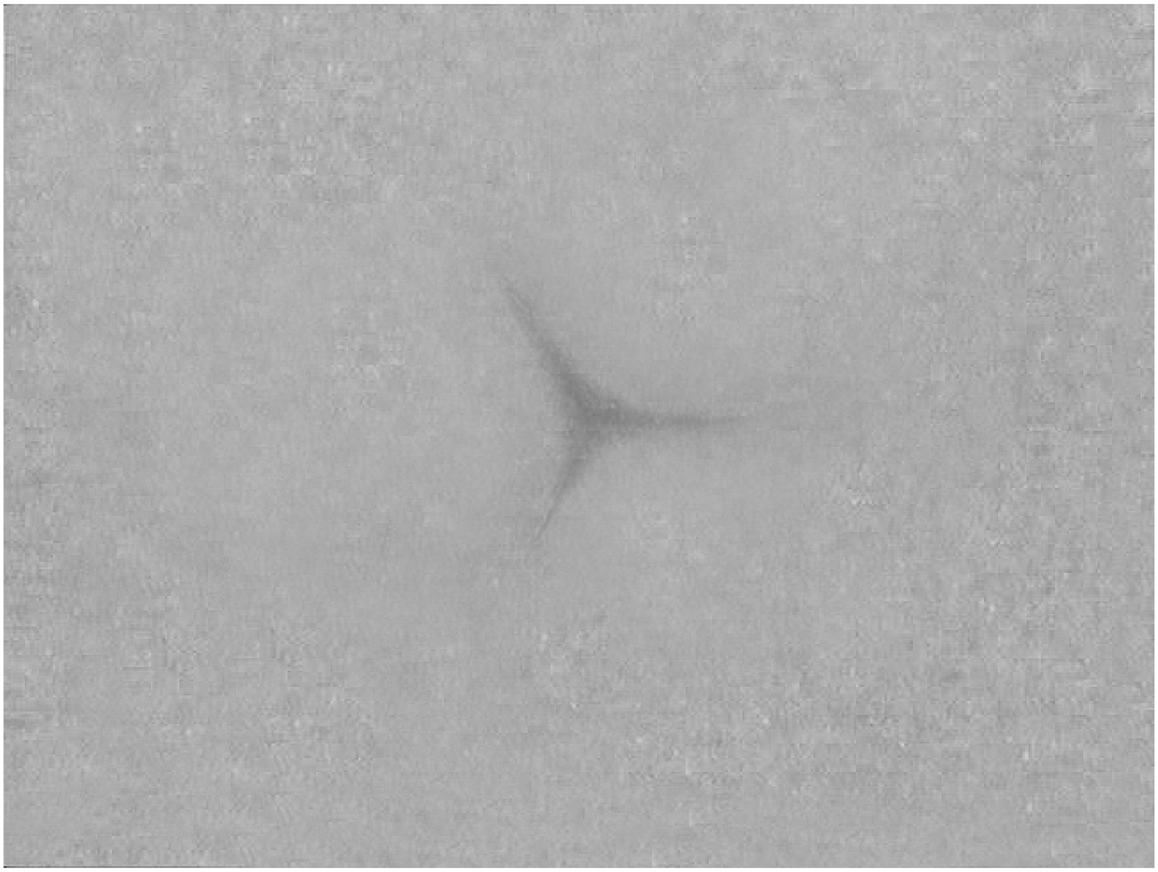


Figure 5: Loading Curve - Polypropylene, Indent 5



CONCLUSION:

In conclusion, we have shown that the Nanovea Nanondentation Tester can provide reliable and reproducible hardness and elastic modulus data on soft polymer thin films such as a 25micron Polypropylene film. The films properties were measured in its manufactured state and not as a bulk material which might or might not be representative of the real behavior of the film. Nanoindentation could have been used also to study the creep behavior in more details, fatigue tests and dynamic mechanical analysis could have been used to study the visco elastic properties of the film. Using a larger spherical tip, one could study (all these parameters) in terms of the full behavior of the film in a compressive mode. Other tests that could have be done on the same instrument include nano wear/friction study, marring of the surface during nano scratch testing, plastic and elastic deformation during scratch testing and surface measurement using AFM imaging. Learn more about Nanoindentation