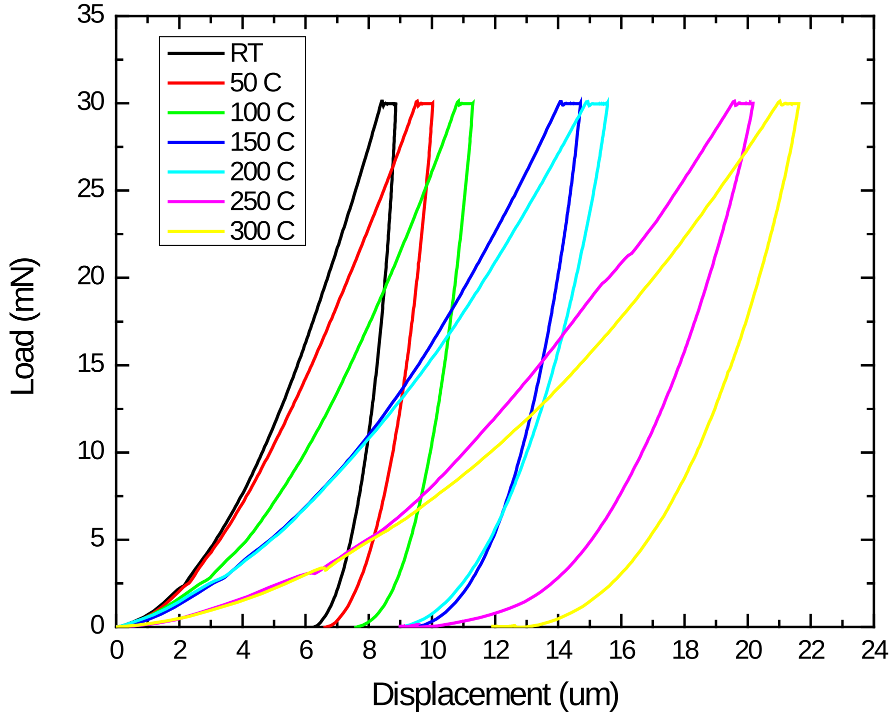
# TEFLON MECHANICAL PROPERTIES AT HIGH TEMPERATURE BY NANOINDENTATION





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## INTRODUCTION

Temperature stability is a critical property of polymers for high temperature applications, spanning the electronic, automotive, aerospace, nuclear technology and others. Polymers are more susceptible to creep under a high stress as they soften and degrade at elevated temperatures. In order to ensure performance reliability, a better understanding of the thermomechanical behavior of polymers is vital for high temperature service.

### IMPORTANCE OF NANOINDENTATION TEST AT HIGH TEMPERATURE

At elevated temperatures, heat changes the mechanical properties of the polymers such as the hardness and viscoelasticity, which may result in mechanical failures of the polymeric part. A reliable measurement of the thermo-mechanical behavior of polymeric materials is in need to quantitatively evaluate the candidate materials for high temperature applications. The Nano module of the Nanovea Mechanical Tester studies the Hardness, Young's Modulus and Creep by applying the load with a high-precision piezo and measuring the evolution of force and displacement. An advanced oven creates a uniform temperature surrounding the indentation tip and the sample surface throughout the nanoindentation test so as to minimize the effect of thermal drift.

## MEASUREMENT OBJECTIVE

In this application, we showcased that the Nanovea Mechanical Tester in Nanoindentation mode measures the mechanical properties such as hardness, Young's modulus and creep of a Teflon sample at elevated temperatures up to 300 °C, making it an ideal tool for analyzing and selecting the best high-performance thermoplastics for targeted high temperature applications.

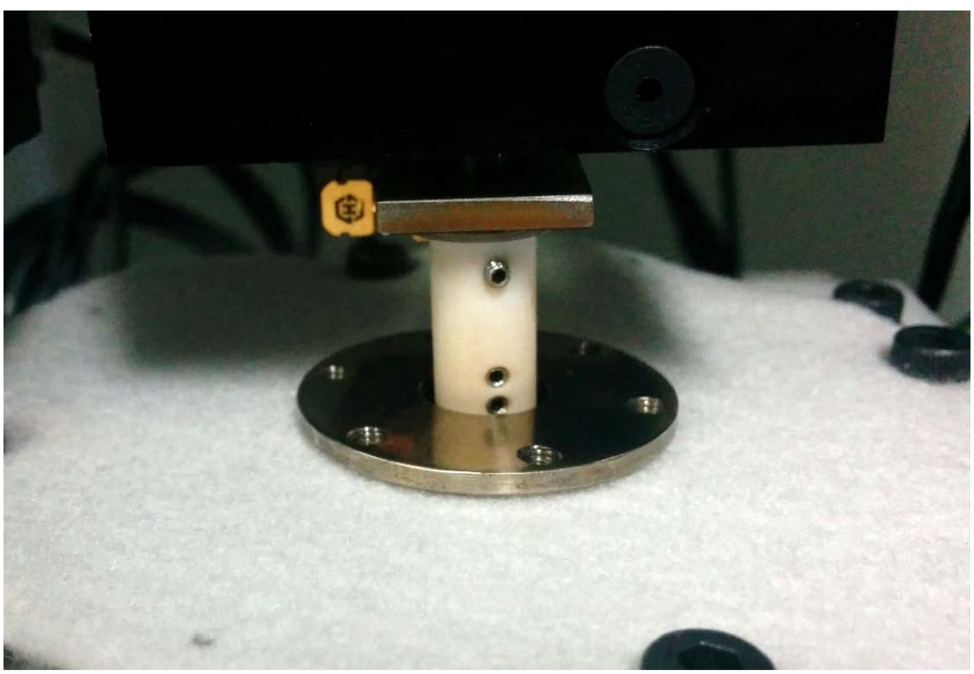

**Fig. 1: High temperature oven for nanoindentation.**

## TEST CONDITIONS

The Nanovea Mechanical Tester in nanoindentation mode is employed to measure the hardness, Young's modulus and creep of a Teflon sample at different elevated temperature ranging from the room temperature, RT, to 300 °C. The melting point of Teflon is 326.8 °C. The sample surface and the indenter are enclosed in an oven providing uniform temperature during the indentation test to minimize the thermal drift and guarantee measurement accuracy. The creep is measured by the change of indentation depth at the maximum load of 30 mN for 10 s at each temperature. A 10 s holding period was taken at 90% unloading load to perform further thermal drift correction in all experiments. The test conditions are summarized in Table 1.

| | |
|---|---|
| **Temperature (°C)** | RT, 50, 100, 150, 200, 250, 300 |
| **Maximum load (mN)** | 30 |
| **Loading rate (mN/min)** | 60 |
| **Unloading rate (mN/min)** | 60 |
| **Creep time (s)** | 10 |
| **Computation Method** | ASTM E-2546 & Oliver & Pharr |
| **Indenter type** | Berkovich Diamond |

**Table 1: Test conditions of the nanoindentation at different temperature.**

## RESULTS AND DISCUSSION

The load vs. displacement plot of the nanoindenation tests at different temperature is shown in Fig. 2. The evolution of hardness and Young's modulus as a function of the temperature is plotted in Fig. 3. As the temperature increases from RT to 300 °C, the load-displacement curve shifts progressively towards higher penetration depth, leading to decreased hardness from ~0.021 to ~0.003 GPa. Meanwhile, the Young's modulus decreases from 0.47 to 0.05 GPa.

The time-dependent visco-elastic deformation of the Teflon sample is also affected by the temperature. Nanovea Mechanical Tester provides excellent accuracy of the creep measurement by applying the close loop control of the load with ultra-sensitive load cell and piezo. Polymer materials usually exhibits higher creep rate upon exposure to a high temperature environment. As shown in Fig. 4, higher creep rate is observed when the sample is subjected to higher temperature in this study, leading to higher creep depth at the end of the 10 s maximum load period.

A variety of industrial applications require polymeric materials which possess reliable mechanical behaviors when exposed to high temperature environment. Nanovea Mechanical Tester provides accurate nanoindentation measurement of the mechanical properties such as the hardness, Young's modulus and creep at high temperatures up to 400 °C, making it an ideal tool for assessing and selecting the best materials for intended high temperature applications.

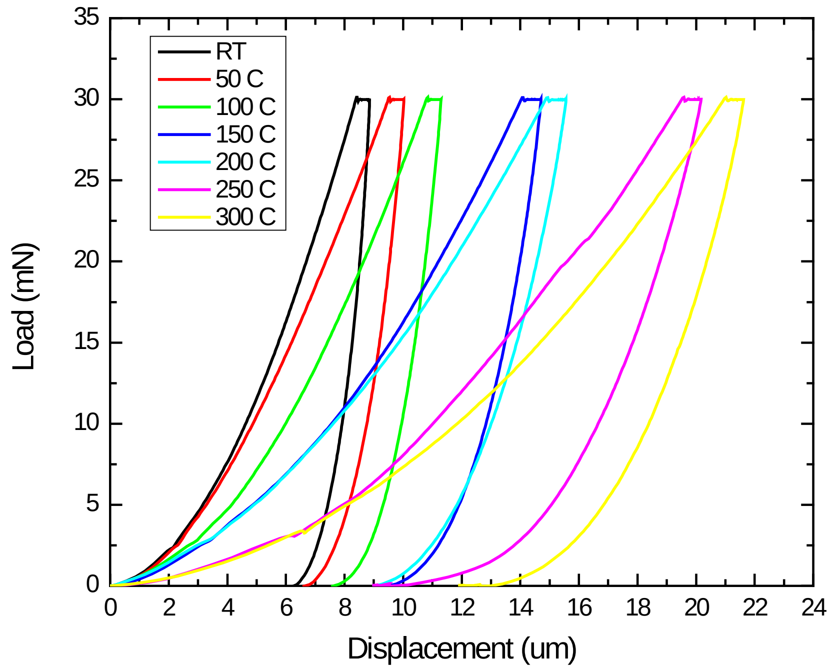


**Fig. 2: The load vs. displacement curves at different temperatures.**

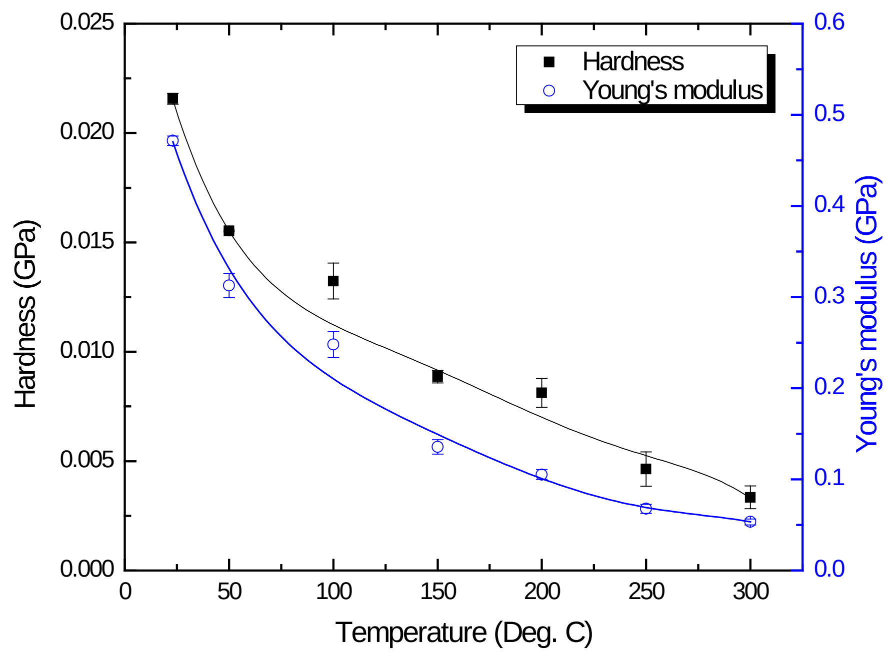


**Fig. 3: The hardness and Young's modulus as a function of the temperature.**

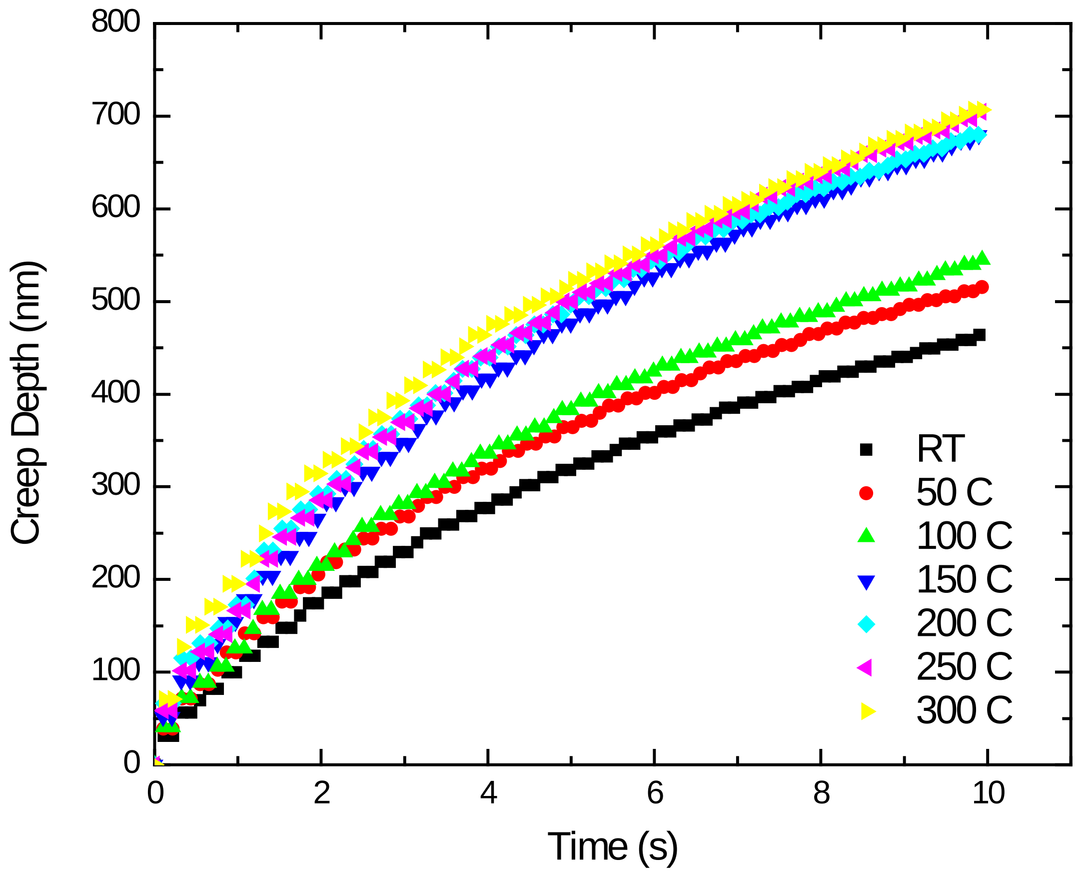


**Fig. 4: Evolution of the creep depth at the maximum load at different temperature.**

## CONCLUSION

The Nanovea Mechanical Tester performs the nanoindentation test on a Teflon sample at different elevated temperatures up to 300 °C. Both the Teflon sample and the indenter are enclosed in an oven with uniform temperature to minimize the impact of thermal drift. The mechanical properties, such as hardness, Young's modulus and creep are affected by elevated temperatures. The creep at the maximum load provides a quantitative and reliable measurement on the time-dependent visco-elastic behavior of the polymer materials for high temperature applications. The Teflon sample in this study shows progressively decreased hardness and Young' modulus as the temperature elevates. Creep contributes to significant plastic deformation during the indentation at high temperatures.

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.

## 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.

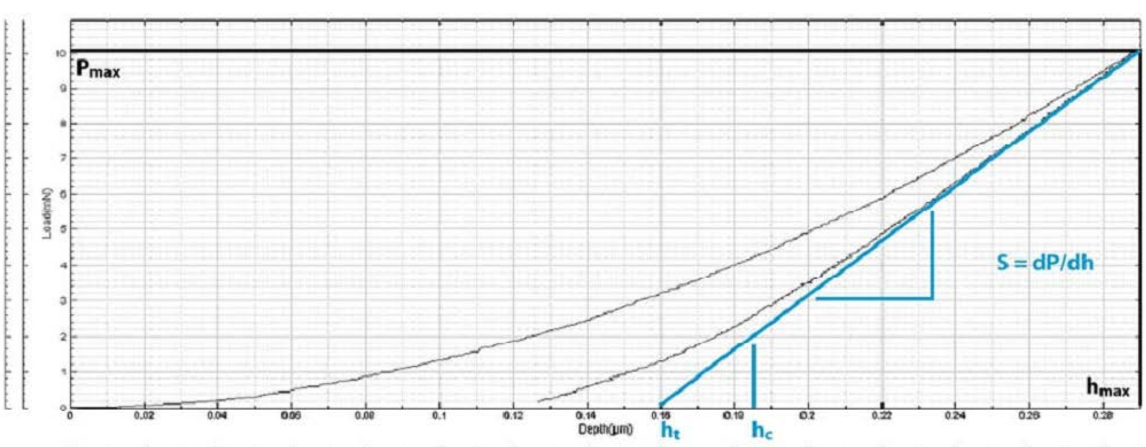


**Fig. 5: Load-displacement curve of nanoindentation.**

### 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 $\nu_i$ are the Young's modulus and Poisson's ratio of the indenter and $\nu$ 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 $A_c$=24.5$h_c^2$. For Cube Corner indenter, the area function is $A_c$=2.60$h_c^2$. For Spherical indenter, the area function is $A_c$=2π$Rh_c$ , 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 $\sigma$ is the stress, $\eta$ is the viscosity of the material, and dε/d$t$ 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.

## DMA SINUS MODE PRINCIPLE

**Sinus Mode (Ranging from 0.1 Hz to 100 Hz):** A sinusoidal stress is applied and the strain in the material is measured. This allows plotting hardness and elastic modulus versus depth and can be used to study viscoelastic materials such as polymers, varnishes, plastics.

**Storage modulus *E'*** characterizes the elastic behavior.
**Loss Modulus *E''*** characterizes the viscous behavior (loss of energy due to internal friction).

$E^* = E' + iE''$, $\quad E' = \frac{\sqrt{\pi}}{2\sqrt{A_{co}}} \frac{\Delta P}{\Delta h} Cos\phi \ (1-\nu^2)$, $\quad E'' = \frac{\sqrt{\pi}}{2\sqrt{A_{co}}} \frac{\Delta P_o}{\Delta h_o} Sin\phi \ (1-\nu^2)$

Where $\phi$, the phase shift between depth and load curves, $\frac{\Delta P_o}{\Delta h_o}$, the variation of load and depth respectively for one oscillation. $A_{co}$, the projected contact area for the oscillation. The viscosity factor λ can be calculated from $\lambda = \frac{1}{2\pi f} \frac{\Delta P_o}{\Delta h_o} Sin\phi$ where *f* is the frequency at which the test was performed.