NANOVEA

DMA FREQUENCY SWEEP

ON POLYMER USING NANOINDENTATION

Prepared by

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INTRODUCTION

Viscoelastic materials exhibit both viscous and elastic characteristics when undergoing deformation. Long molecular chains in polymer materials contribute to their unique viscoelastic properties, i.e. a combination of the characteristics of both elastic solids and Newtonian fluids. Stress, temperature, frequency and other factors all play roles in the viscoelastic properties. Dynamic Mechanical Analysis, also known as DMA, studies the viscoelastic behavior and complex modulus of the material by applying a sinusoidal stress and measuring the change of strain.

IMPORTANCE OF DMA FREQUENCY SWEEP TEST

The changing frequency of the stress often leads to variations in the complex modulus, which is a critical mechanical property of polymers. For example, tires are subjected to cyclical high deformations when vehicles are running on the road. The frequency of the pressure and deformation changes as the car accelerates to higher speeds. Such a change can result in variation in the viscoelastic properties of the tire, which are important factors in the car performance. A reliable and repeatable test of the viscoelastic behavior of polymers at different frequencies is in need. The Nano module of the ***NANOVEA*** Mechanical Tester generates sinusoidal load by a high precision piezo actuator and directly measures the evolution of force and displacement using ultrasensitive load cell and capacitor. The combination of easy setup and high accuracy makes it an ideal tool for DMA frequency sweep.

MEASUREMENT OBJECTIVE

In this application, we study viscoelastic properties of a polished tire sample at different DMA frequencies using the Most Powerful Mechanical Tester, ***NANOVEA*** *PB1000, in Nanoindentation mode.*

NANOVEA
PB1000

TEST CONDITIONS

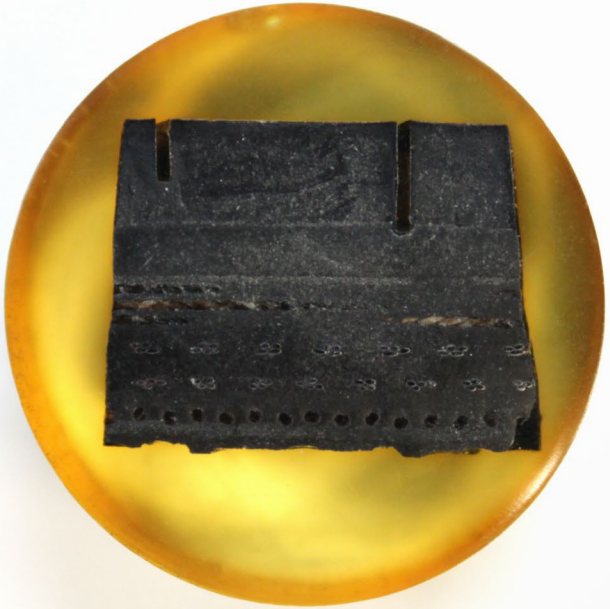
FREQUENCIES (HZ) 0.1, 1.5, 10, 20

CREEP TIME AT EACH FREQ.	OSCILLATION VOLTAGE	LOADING VOLTAGE
50 sec	0.1 V	1 V

INDENTER TYPE

Spherical

Diamond | 100 µm



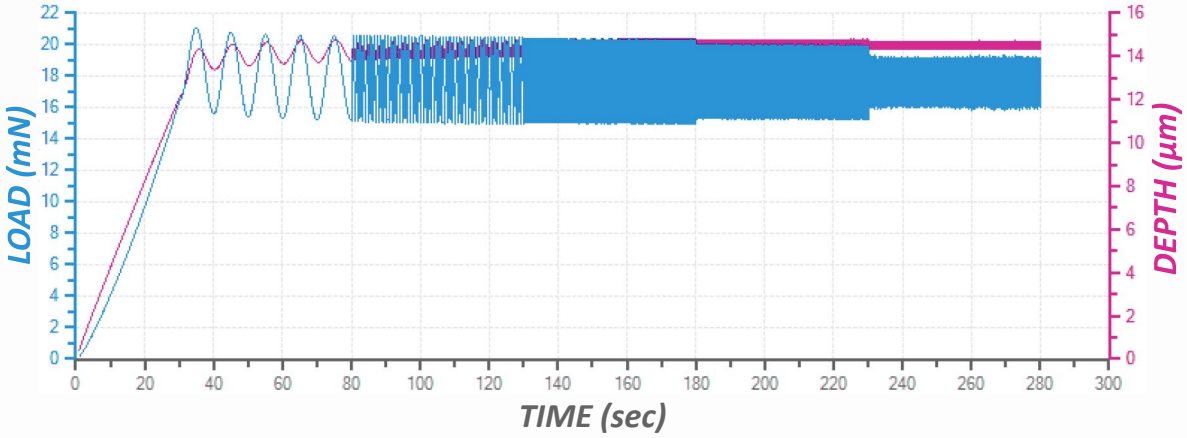
* the tested sample

RESULTS & DISCUSSION

The DMA frequency sweep at the maximum load allows a fast and simple measurement on the viscoelastic characteristics of the sample at different loading frequencies in one test. The phase shift and the amplitudes of the load and displacement waves at different frequencies can be used to calculate a variety of fundamental material viscoelastic properties, including ***Storage Modulus***, ***Loss Modulus*** and ***Tan (δ)*** as summarized in the following graphs.

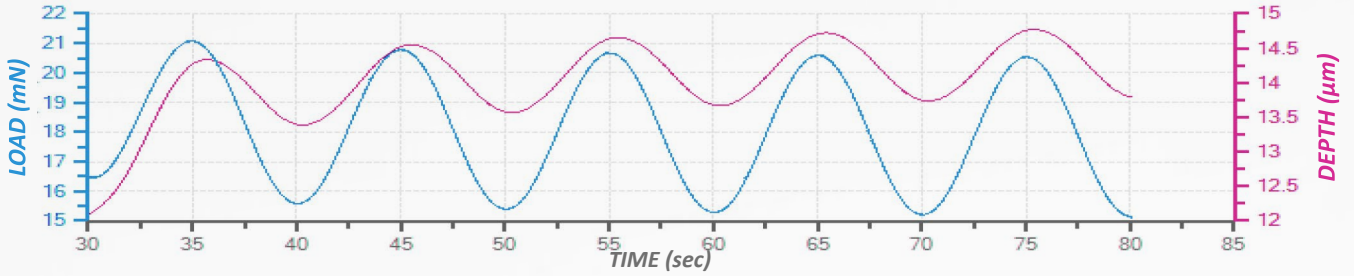
Frequencies of 1, 5, 10 and 20 Hz in this study, correspond to speeds of about 7, 33, 67 and 134 km per hour. As the test frequency increases from 0.1 to 20 Hz, it can be observed that both Storage Modulus and Loss Modulus progressively increase. Tan (δ) decreases from ~0.27 to 0.18 as the frequency increases from 0.1 to 1 Hz, and then it gradually increases to ~0.55 when the frequency of 20 Hz is reached. DMA frequency sweep allows measuring the trends of Storage Modulus, Loss Modulus and Tan (δ), which provide information on the movement of the monomers and cross-linking as well as the glass transition of polymers. By raising the temperature using a heating plate during the frequency sweep, a more complete picture of the nature of the molecular motion under different test conditions can be obtained.

EVOLUTION OF LOAD & DEPTH
OF THE FULL DMA FREQUENCY SWEEP

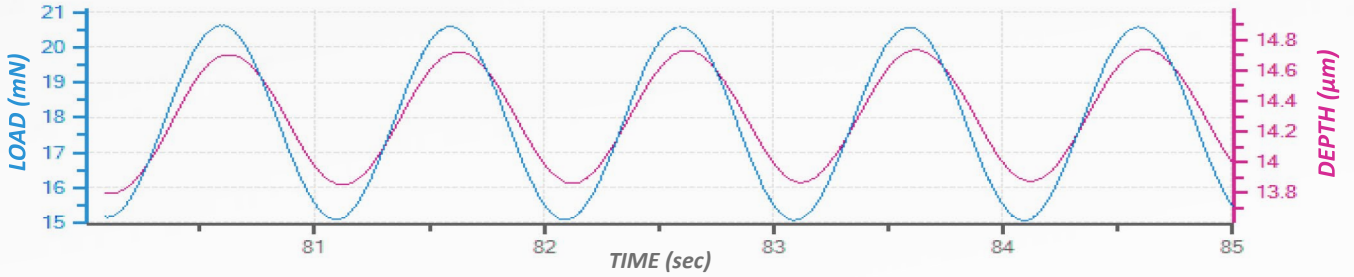


LOAD & DEPTH vs TIME AT DIFFERENT FREQUENCIES

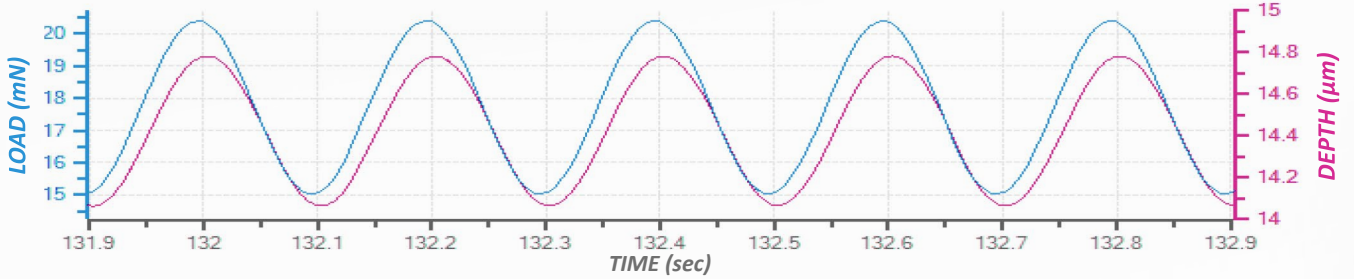
0.1 Hz



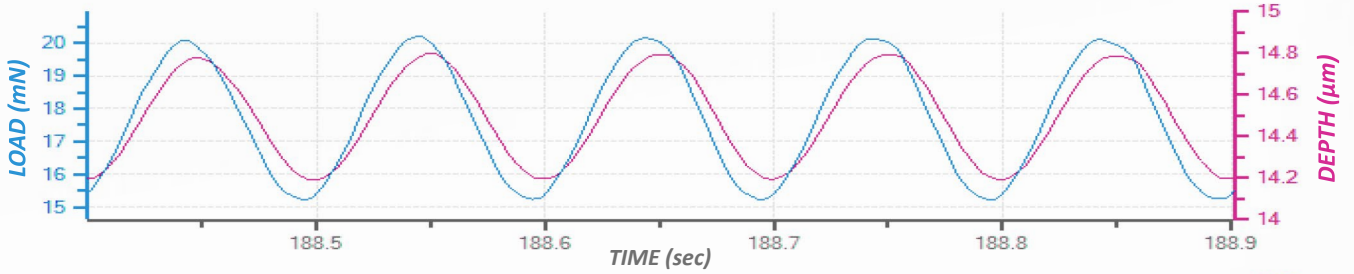
1 Hz



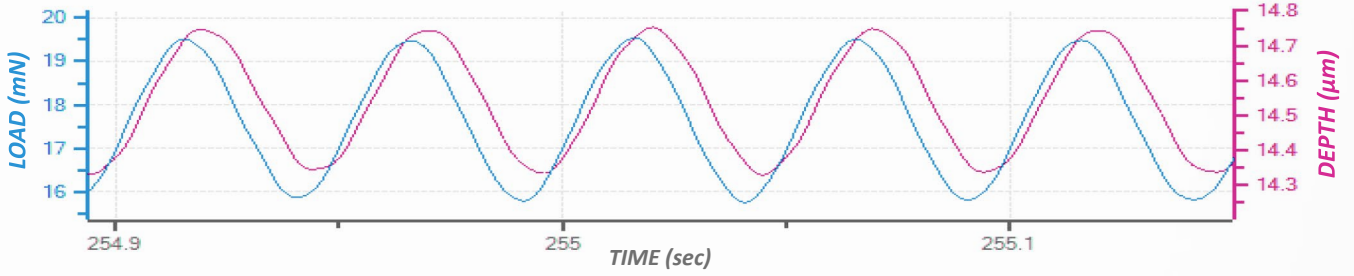
5 Hz



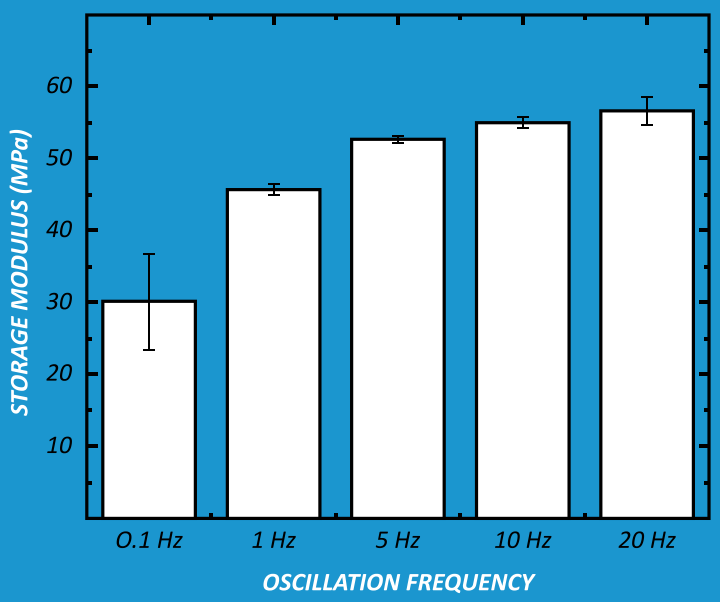
10 Hz



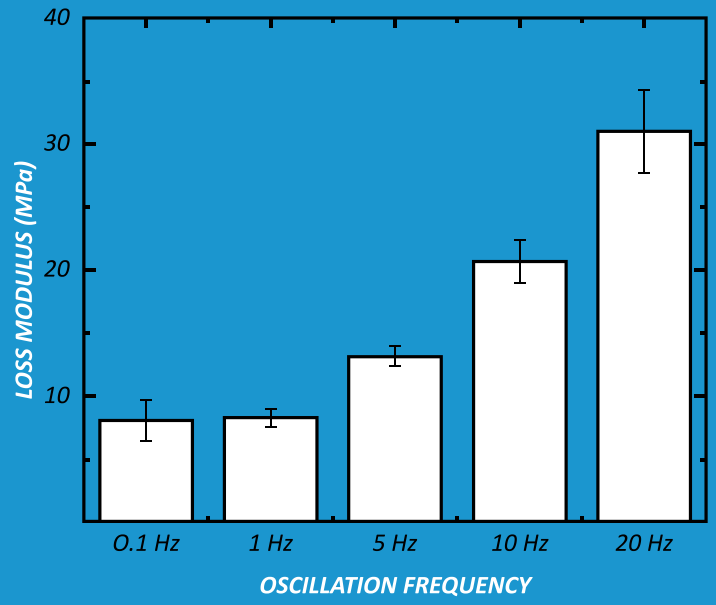
20 Hz



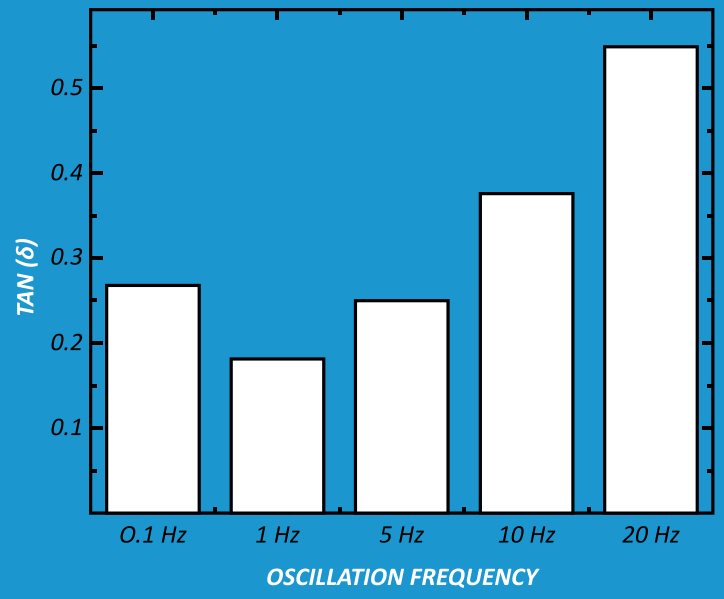
STORAGE MODULUS
AT DIFFERENT FREQUENCIES



LOSS MODULUS
AT DIFFERENT FREQUENCIES



TAN (δ)
AT DIFFERENT FREQUENCIES



CONCLUSION

In this study, we showcased the capacity of the ***NANOVEA*** Mechanical Tester in performing the DMA frequency sweep test on a tire sample. This test measures the viscoelastic properties of the tire at different frequencies of stress. The tire shows increased storage and loss modulus as the loading frequency increases from 0.1 to 20 Hz. It provides useful information on the viscoelastic behaviors of the tire running at different speeds, which is essential in improving the performance of tires for smoother and safer rides. The DMA frequency sweep test can be performed at various temperatures to mimic the realistic working environment of the tire under different weather.

In the Nano Module of the ***NANOVEA*** Mechanical Tester, the load application with the fast piezo is independent from the load measurement done by a separate high sensitivity strain gage. This gives a distinct advantage during DMA since the phase between depth and load is measured directly from the data collected from the sensor. The calculation of phase is direct and does not need mathematical modeling that adds inaccuracy to the resulting loss and storage modulus. This is not the case for a coil-based system.

In conclusion, DMA measures loss and storage modulus, complex modulus and Tan (δ) as a function of contact depth, time and frequency. Optional heating stage allows determination of materials phase transition temperature during DMA. The ***NANOVEA*** Mechanical Testers provide unmatched multi-function Nano and Micro modules on a single platform. Both the Nano and Micro 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.