ADVANCED DYNAMIC MECHANICAL ANALYSIS OF A TIRE SAMPLE BY NANOINDENTATION

Duanjie Li and Pierre Leroux, Nanovea, Irvine, CA

Abstract

The viscoelastic properties of a tire sample are comprehensively studied by dynamical mechanical analysis (DMA) using nanoindentation technique with the Nanovea Mechanical Tester. We found that the tire possesses different viscoelastic mechanical properties across the tire layers. Such strategical distribution of the hardness and complex modulus at different layers meets the functionality requirements of the tire. The tire shows increased storage and loss modulus as the loading frequency increases from 0.1 to 20 Hz. The DMA frequency sweep test 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.

Introduction

An elastic material strains immediately when stressed and it returns to its original shape once the stress is removed. In comparison, a viscous material under the influence of external forces strains linearly with time. Viscoelastic materials such as polymers exhibit both viscous and elastic characteristics as they undergo deformation, and they are therefore characterized by the complex modulus:

\[ E^* = E' + iE'' \]

where the storage modulus \( E' \) characterizes the elastic behavior, and the loss modulus \( E'' \) characterizes the viscous behavior (loss of energy due to internal friction between molecules). Dynamical mechanical analysis (DMA) is developed to investigate this behavior by applying a sinusoidal oscillatory stress in tension, compression, torsion or bending to a sample of known geometry. From the response of the sample subjected to the controlled stress or strain the complex modulus can be calculated.

Nanovea Mechanical Tester performs the DMA measurement using nanoindentation technique. Compared to conventional macroscale DMA testers, nanoindentation does not require fabricating test samples into stringent geometry, thus cutting significant cost and time on sample preparation. More importantly, the small indentation size makes it possible to perform multiple measurements on one single sample, allowing mapping the viscoelastic properties of the sample as a function of location.

Many nanoindentation systems on the market use a coil/magnet assembly as the actuator to apply sinusoidal oscillation to the indenter without measuring the load with a separate sensor. Because of mechanical and electrical delay from the order to move to the actual position, complex algorithms are needed to account for the delays in the calculation of the phase shift between the load and displacement. In comparison, the Nanovea Mechanical Tester generates the sinusoidal load by a high-precision piezo actuator and directly measures the evolution of force and displacement using ultra-sensitive load cell and capacitor. The feedback load sensing not only eliminates complex algorithms, but also significantly improves the precision and accuracy of the data. The combination of easy setup and high accuracy makes the Nanovea Mechanical Tester an ideal tool for DMA tests.

Tires are subjected to cyclical high deformations when vehicles are running on the road. When exposed to harsh road conditions, the service lifetime of the tires is jeopardized by many factors, such as the wear of the tread, the heat generated by friction, rubber aging, and others. As a result, tires usually have composite layer structures made of carbon-filled rubber, nylon cords, and steel wires, etc. In particular, the composition of rubber at different layers of the tire architecture is optimized to provide different functional properties, including but not limited to wear resistant tread, cushion rubber layer and hard rubber base layer. A reliable and repeatable test of the viscoelastic behavior of rubber is critical in R&D and quality control of new tires, as well as evaluation of life span of old tires. In addition, frequencies of the tire rotation and deformation change as the car accelerates to higher speeds. Such a change in cyclic stress and strain can result in variation in the viscoelastic properties of the tire and in turn influence the car performance.

In this study, we evaluate the viscoelastic properties of different layers of the tire by DMA tests using nanoindentation technique. The complex modulus and mechanical behavior of the tire are mapped across the cross section of the tire. The DMA frequency sweep test is performed on the tire sample to investigate the effect of the cyclic stress/strain frequency on the complex modulus of the tire, which represents the viscoelastic properties of the tire at different car speed.
Test Conditions

The Nanovea Mechanical Tester is used to measure the viscoelastic properties of the polished cross section of a Continental tire sample by means of DMA tests. The sinusoidal oscillation was applied to a 100-μm spherical diamond tip. The Nanovea Mechanical Tester has true close feedback control on the load applied. The load applied by the fast piezo is independent from the load measured 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 sensors. The calculation of phase shift is direct and does not need complex mathematical modeling that adds inaccuracy to the resulting loss and storage modulus.

A series (40 points) of nanoindentation DMA was performed along the thickness of the tire sample and the distribution of the indentations is illustrated in Fig. 1. The 40 indentations along the tire thickness were spaced 0.38 mm apart for a total distance of ~15.5 mm. The test conditions are summarized in Table 1.

DMA frequency sweep test by nanoindentation was performed on the tire sample. The test conditions are summarized in Table 2.

Table 2: Test conditions of the DMA frequency sweep by nanoindentation.

<table>
<thead>
<tr>
<th>Loading voltage (V)</th>
<th>1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Loading rate (V /min)</td>
<td>2</td>
</tr>
<tr>
<td>Unloading rate (V /min)</td>
<td>2</td>
</tr>
<tr>
<td>Oscillation amplitude voltage (V)</td>
<td>0.1</td>
</tr>
<tr>
<td>Creep time at each frequency (s)</td>
<td>50</td>
</tr>
<tr>
<td>Frequency (Hz)</td>
<td>0.1, 1, 5, 10, 20</td>
</tr>
</tbody>
</table>

Results and Discussion

1. DMA mapping across the tire sample

The Tan(δ), storage modulus, loss modulus and hardness measured from the DMA mapping along the thickness of the tire sample are summarized in Fig. 2 to Fig. 5. The distribution of the hardness and complex modulus meets the functionality requirements of the rubber at different layers of the tire. The rubber at the positions from 0.38 to 2.66 mm and from 8.22 and 11.64 mm exhibits a relatively low hardness below 2 MPa and low complex modulus. Such a relative soft feature allows the rubber in these regions to serve as a cushion layer and absorb the shocks and vibrations. In comparison, the rubber at the positions from 3.04 to 7.84 mm shows higher hardness and complex modulus, due to the composite structure consisting of high tensile-strength reinforcing fabric encased in the rubber compound. The enhanced mechanical properties of this layer provide strength and toughness to the tire structure. The positions from 12.02 to 15.82 mm reside in the tire tread and exhibit higher hardness and complex modulus compared with the “cushion layer”. The high carbon black concentration in this layer provides reinforced abrasion resistance, cut resistance as well as better traction.

Table 1: Test conditions of the nanoindentation DMA along the thickness of the tire sample.

| Maximum force (mN) | 75 |
| Loading rate (mN /min) | 150 |
| Unloading rate (mN /min) | 150 |
| Creep (s) | 20 |
| Amplitude (mN) | 15 |
| Frequency (Hz) | 10 |
The desired functionality of the different tire layers is achieved by the strategical design of specific viscoelastic properties in these layers. Zones of high loss modulus will absorb differently than zones of low loss modulus. The DMA mapping tests allow one to develop tires for smoother and safer rides in different weather conditions. The Nanovea Mechanical Tester can perform the DMA tests at various temperatures and even in liquids. It can also be conducted at various frequencies to mimic the behavior of tires as the car runs at different speeds. In the following section, we further investigate the influence of the oscillation frequency during the DMA tests on the viscoelastic properties of the tire rubber.

2. DMA frequency sweep

Long molecular chains in polymer materials contribute to their unique viscoelastic properties. Such a combination of the characteristics of both elastic solids and Newtonian fluid can be influenced by the oscillation frequency. The DMA frequency sweep at the maximum load measures the viscoelastic characteristics of the sample at different loading frequencies in one test. The load & depth curve of the full frequency scan is plotted in Fig. 6 and the load and depth as a function of time at each frequency is shown in Fig. 7. The phase shift and the amplitudes of the load and displacement waves at different frequencies, i.e. 0.1, 1, 5, 10 and 20 Hz in this DMA sweep test can be used to calculate a variety of fundamental material viscoelastic properties, including storage modulus, loss modulus and Tan (δ) as summarized in Fig. 8 and Fig. 9. Frequencies of 0.1, 1, 5, 10 and 20 Hz in this study, correspond to speeds of about 0.7, 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 the Storage Modulus and the 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. The variation of Storage Modulus, Loss Modulus and Tan (δ) at different frequencies provides information on the movement of the monomers and cross-linking in polymers. The DMA frequency sweep test can also be performed at various temperatures and in different media to mimic the realistic working environment of the tire under different weather, providing a more complete picture of the nature of the molecular motion under different service conditions.

**Fig. 6**: Evolution of load & depth of the full DMA frequency sweep.

**Fig. 7**: Load and depth as a function of time at different frequencies.

**Fig. 8**: Storage and Loss Modulus at different frequencies.

**Fig. 9**: Tan (δ) at different frequencies.

**Conclusion**

In this study, we comprehensively evaluated the viscoelastic properties of a tire sample by the DMA mapping and frequency sweep tests using the Nanovea Mechanical Tester. Unlike the nanoindentation systems that use coil actuator to generate oscillation, the Nanovea Mechanical Tester performs the nanoindentation by applying the load with a fast piezo that 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, which significantly improves the accuracy of the calculated loss and storage modulus.

The DMA mapping across the depth of the tire shows the change of the complex modulus at different tire layers,
which meets the functionality requirements of the tire design. In the DMA frequency sweep test, the tire shows increased storage and loss modulus as the loading frequency increases from 0.1 to 20 Hz. This test 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.

In conclusion, the DMA measures the 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. In addition, the Nanovea Mechanical Tester provides unmatched multi-functional Nano and Micro/Macro modules, including scratch tester, hardness tester and wear tester modes.

Bio

Duanjie Li is the lead materials scientist at Nanovea, 6 Morgan, Ste. 156, Irvine CA 92618, email: duanjie@nanovea.com. He has a B.S. degree from Tsinghua University in China and a Ph.D. from McGill University in Montreal, Quebec, Canada. He specializes in advanced mechanical, tribological, and surface profile characterization techniques. He develops novel functions and algorithms to improve accuracy and versatility of the precision materials characterization instruments.

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