# Harsh Environment Materials Testing: -100°C to 800°C

xSol® Combined with nanoDMA® III Testing of Tread Materials Used in Winter Tires

## Introduction

Modern automobile tires are composed of technologically advanced materials systems engineered to provide a combination of properties optimized for the tire's operating conditions. The polymer tread compound includes additives that help to balance such factors as wear rate, traction, strength, noise, ride quality, and rolling resistance. Optimizing the properties involves compromise because an adjustment to the mechanical properties that improves some characteristics often comes at the expense of others. The tread compound's mechanical properties also depend strongly on temperature, ensuring that the tire will perform properly only over a certain limited temperature range. Characterization of local mechanical properties is critical for understanding the distribution and impact of filler particles and additives across various areas of the tire's tread.

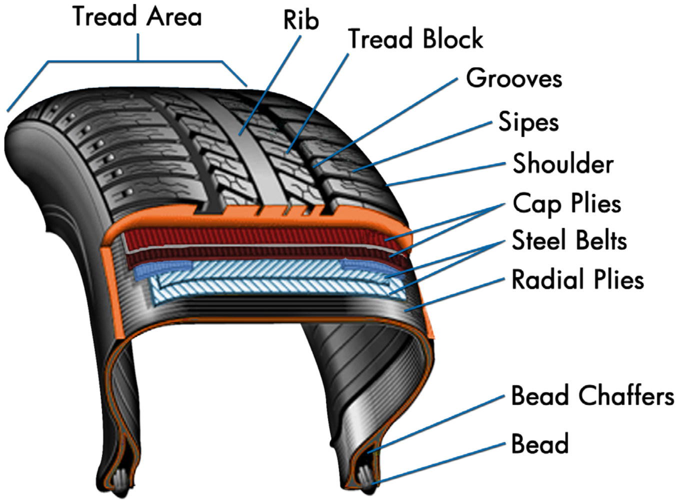


Figure 1: Tire structure and engineered layers.

## Nanoscale to Macroscale

Tire properties are engineered at the nanoscale by controlling the polymer's crosslink density and including varying amounts of such fillers as carbon black, clays, and silicates to further tune the properties. At intermediate scales, features such as micropores and compositional gradients may be employed to further enhance the tire's performance. At macro scale, the design of the tread pattern and the processing conditions used to form the tire finally determine the tire's overall behavior in service conditions.

Local mechanical properties strongly reflect the local particle distribution and are therefore of interest to the engineer. Volumes of several hundred cubic nanometers may be probed with a sharp indenter and an instrument with high force sensitivity. Microscale is accessed by using larger indenters with larger forces and penetration depths, allowing volumes of several hundred cubic microns to be measured. The stiffness and damping behavior of the tire at different scales will determine how it behaves under different conditions. Characteristics such as noise abatement, wear resistance, and friction are determined largely by small scale properties whereas the tire's ability to flex under load and dampen harshness from road imperfections is controlled by the material structure at larger scale.

## Experimental

The tread compound of a commercial winter tire was tested at temperatures between -60°C and 40ºC, reflecting the full range of temperatures a tire would likely face. The sample's properties were measured using a **TI 950 TriboIndenter®** nanomechanical test instrument equipped with an **xSol® Environmental Control Stage** and **nanoDMA® III** dynamic testing module. A cold, dry gas environment was

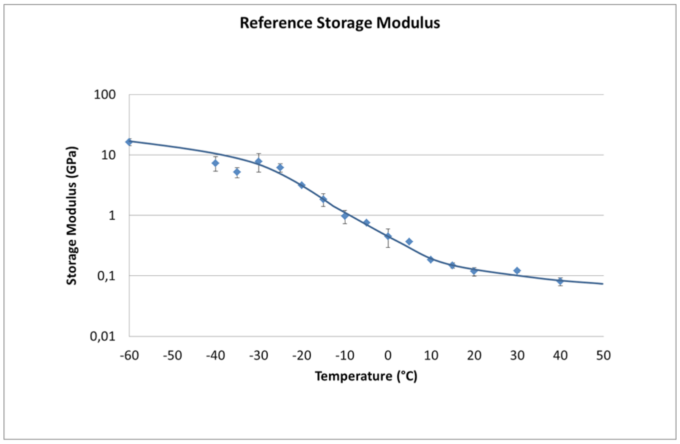


Figure 2: Storage Modulus of the tread compound of a winter tire measured at temperatures between -60°C and 40°C.

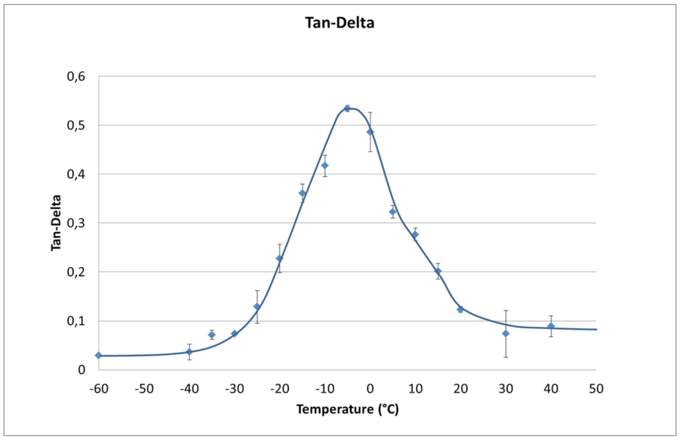


Figure 3: tan(δ) at a frequency of 75Hz of a tread compound of a winter tire measured at temperatures between -60°C and 40°C.

maintained around the sample by the controlled evaporation of liquid nitrogen in conjunction with PID controlled heaters on both sides of the sample. The evaporated nitrogen gas flowed over the sample surface continuously, creating a controlled microenvironment and preventing condensation or frost formation on the sample surface. The temperature was adjusted while dynamic indentation tests were performed using a Berkovich indenter probe at an oscillation frequency of 75Hz to measure the storage modulus (E') and loss tangent (tan δ) of the material.

## Results

Properties vary dramatically over the temperature range, with a storage modulus around 10GPa measured at -60ºC falling to less than 0.1GPa at 40ºC. The material displayed a strong tan δ peak at -5ºC, indicating a glass transition in the material that accounts for the sharp change in properties. The peak at -5ºC indicates a maximum in dissipation and accompanies a marked change in the material's stiffness. At temperatures much lower than -5ºC, noise and harshness will likely increase due to the high stiffness and low damping, while at temperatures well above -5ºC, wear life and mechanical stability will be compromised due to softening of the compound.

## Conclusions

Good control of sample environment is important to characterize advanced materials intended to operate inunder service conditions far from those found in the ambient atmosphere of a laboratory. The xSol Environmental Control Stage ensures a uniform sample temperature even for materials with low thermal conductivity and provides atmospheric control to prevent condensation even at very low temperatures. The xSol is engineered for nanoscale dimensional stability, allowing measurement of individual microstructural components and, at larger scales, overall composite response.

The combination of nanoDMA III with the xSol stage is a powerful DMTA characterization tool requiring a minimal quantity of material, little sample preparation, rapid temperature adjustment and equalization, and exceptional stability for fast and reliable measurement of properties across a range of environmental conditions and orders of length scale. In this experiment, the glass transition temperature and mechanical behavior of a winter tire tread compound were measured successfully over its entire operating range.



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