

# Cross-Sectional DMA Testing on Automotive Tires Using Continuous Stiffness Measurement on Nanoindentation.

## Introduction

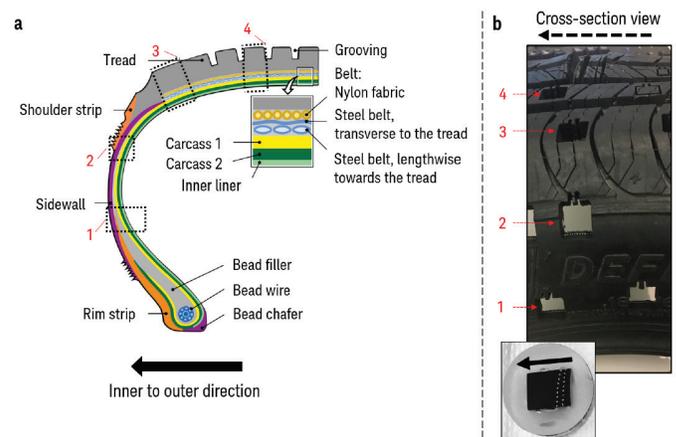
Automotive tires have complex laminated structures made up of layers packed on top of each other from the inner to the outer side of the tire (Figure 1a). The individual components of tires include multiple rubber compounds, textiles, steel belts, beads, etc.<sup>1</sup> Modern tires are designed and manufactured based on complicated engineering simulations and extensive real-world testing for achieving the highest performance in terms of steering, stability, wearing, etc.,<sup>2</sup> which comes from the best mechanical response of all components functioning together.

Dynamic mechanical analysis (DMA) has been widely used to measure the storage and loss modulus of tires. In conventional DMA, the sample is oscillated by factoring in tension, compression, or bending to determine the complex modulus reflecting the viscoelastic materials' response to the oscillation.<sup>3</sup> KLA Nano Indenter systems with continuous stiffness measurement (CSM), and the ability to oscillate samples offers an optional method of measurement of the complex modulus of tires spatially, as well as for a wide range of frequencies due to the lesser mass involved in this technique.<sup>4</sup> The cross-sectional analysis of different parts of a tire consisting of different materials was performed using a flat punch indenter tip with the CSM technique, available on the KLA Nano Indenter systems and is presented in this application note. Profiles of storage and loss modulus variations were generated from the inner side to the outer side of the tire.

## Test Methodology

Structures and materials commonly used in tires vary along the sidewalls as well as the treads and other parts, depending on their functions in tire performance. Samples from different parts of the tire were cut with an industrial waterjet and mounted in epoxy, and then the cross-sectional surfaces were polished and prepared for indentation. The locations of the tested samples on a piece of tire are shown in Figure 1b. Dynamic indentation tests were performed by a diamond flat punch ( $D = 100\mu\text{m}$ ) using the CSM Flat Punch method with frequency sweep varying from 10Hz to 150Hz along the lines from the inner side to the outer side of the tire (Figure 1a). Distances between the indents were kept as 0.2mm and 0.3mm, depending on the locations on the

tire. The areas containing steel belts or fabrics were not included in the test. At each test point, the indenter was initially pressed  $5\mu\text{m}$  into the sample. After achieving full contact, the storage and loss modulus were measured by oscillating the indenter at the above mentioned frequencies.



**Figure 1. (a) Schematic of cross-sectional view of the tire structure containing layers of different materials. Locations are marked as 1-4 from different regions of tire. (b) Samples cut from locations 1-4 (top) and a mounted and polished sample in epoxy for nanoindentation test (bottom) with an arrow showing the direction of indents performed on the cross section of the tire.**

## Measurement Results

Storage modulus is a measure of viscoelastic materials' stiffness—it is proportional to the stored energy during loading. Under single low loading and reversible deformation, it is equivalent to Young's modulus of the material. On the other hand, loss modulus is proportional to the energy dissipated during the loading cycle—it is an indication energy lost in the form of heat, which is the type of energy that cannot be recovered during unloading. The ratio of loss modulus to storage modulus is called loss factor, which is a dimensionless parameter that measures energy lost and indicates the mechanical damping or internal friction of viscoelastic material. Figure 2 shows the distribution of the storage modulus and loss factor at cross sections of the tire at different locations. Results corresponding to the indents performed along the lines from the inner layer to the outer layer of the tire are shown in Figure 1b.

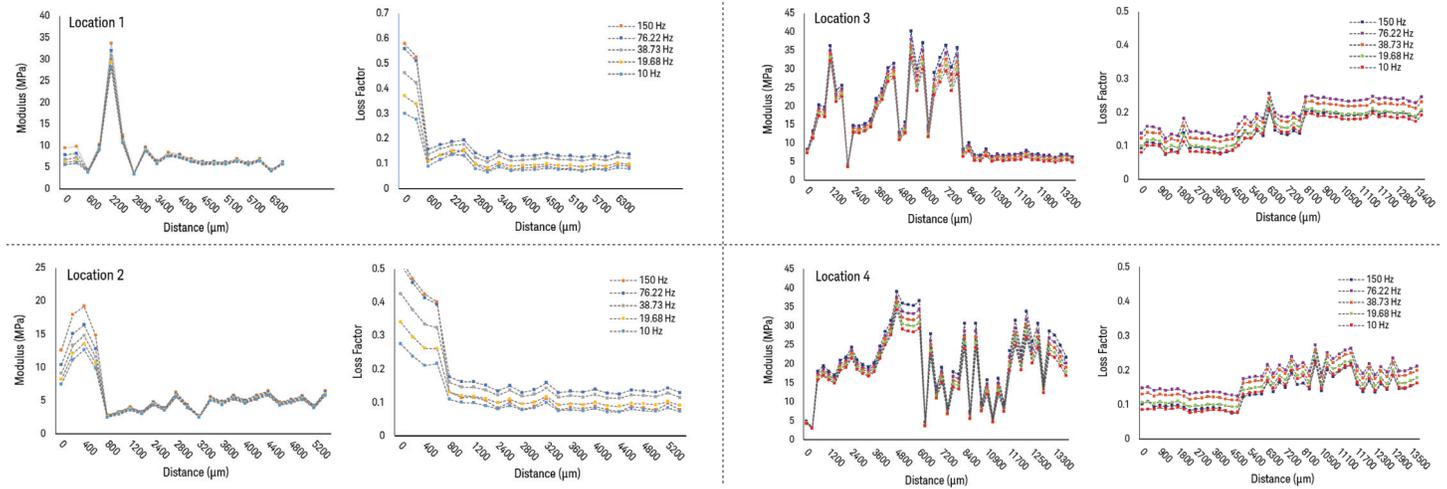


Figure 2. Distribution of the storage modulus and loss factor in cross sections of the tire at different locations (1-4) from the inner to the outer side.

It can be seen that tire storage modulus varies in different layers of the tire as well as at different locations depending on the mechanical requirements for that part of the tire. In Figure 2, location 1 is closest to the beads on the sidewall of the tire which is not in contact with the road surface. This location exhibits consistent storage modulus from the inside to the outside of the tire, but it peaks where a reinforcing fabric exists. A similar behavior is seen in location 2 which is still part of the sidewall of the tire, but closer to the shoulder with a peak near the inner side of the tire, as a result of the existing reinforcing materials. In both locations, loss factor is quite different from that on the inner sides. A high loss factor in these regions is necessary for providing a better damping condition and absorption of shocks and vibration caused by high internal air pressure. Comparing the sidewalls to the midsection of the tire (tread), properties of rubber structures differ across the tire. Several layers of rubber as well as reinforcing steel belts and fabrics are included in the structure of these locations (Figure 1a). The enhanced mechanical properties of rubber in the outer layer are visible in both locations 3 and 4, but particularly at location 4 where the tire is in full contact with the road surface and the tread must provide the best wearing properties as well as greater strength and durability. Figure 3 shows the structure of rubber in various locations of the tire, providing a visual of the differences in mechanical properties on the tire designed to perform specific functions.

In the design of tires, it is essential to have zones with high loss modulus for absorbing energy during vibration. This becomes even more important at higher speeds. To mimic this scenario, nanoindentation tests were conducted across various frequencies. Higher frequencies coincide with higher speeds.

While both storage modulus and loss modulus increase with frequency, loss modulus increases more. As a result, loss factor net increases, indicating that the tire will dampen more energy at higher speeds. This is more evident for inner layer of locations 1 and 2, where higher energy absorption is required. Tires also show time-temperature dependency in storage and loss modulus. In real-world environments, tires experience a wide range of temperatures from tens of degrees below 0°C to temperatures that can reach up to 100°C. Therefore, it is worthwhile to study the mechanical response of tires at various temperatures.

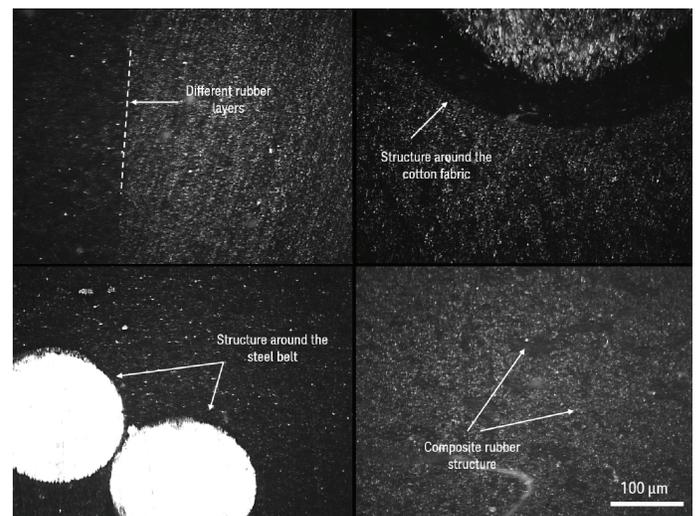


Figure 3. Variation in rubber structure in different regions of the tire.

### Conclusions

Cross sections of the tire were tested using CSM technique by a diamond flat punch tip. The local storage and loss modulus were measured at various locations of the tire that had different rubber structures. The CSM technique equipped with the KLA Nano Indenter systems is capable of dynamically measuring the complex modulus of viscoelastic materials, offering a unique option to measure the variations in properties spatially and temporally. It is a very simple, fast, and automated way of measuring viscoelasticity in materials compared to the traditional DMA testing methods that require a specific sample geometry. The application of CSM technique using a flat punch tip on tires can reveal spatial variations in the mechanical properties of tires at different layers, which leads to better understanding of tire functionality under different mechanical and environmental conditions.

### References

1. B. Lebreton, A. Tum, A quantitative approach to assessing the profitability of car and truck tire remanufacturing, *International Journal of Production Economics*, 104 (2014), 639-652.
2. G. Heinrich, M. Klüppel, Rubber friction, tread deformation and tire traction, *Wear*, 265 (2008), 1052-1060.
3. KP Menard, *Dynamic mechanical analysis: a practical introduction*, Taylor Francis group, Second edition (2008).
4. G. M. Odegard, T. S. Gates, H. M. Herring, characterization of viscoelastic properties of polymeric materials through nanoindentation, *Journal of Experimental Mechanics*, 45 (2005), 130-136.

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KLA Corporation  
One Technology Drive  
Milpitas, CA 95035  
[www.kla.com](http://www.kla.com)

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