Developments in the indentation modulus profile

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Executive summary

The modulus (indentation modulus) profiler was developed in 1987 by Gillen, Clough, and Quintana.[2]

The technique has a special resolution of about 100 microns. The modulus profiler has been used to study tire aging, durability in mechanical rubber goods, and treadwear by Terrill, Lewis and Pannikottu[1]. The technique has quantified modulus changes in small and internal components of rubber products.

Recently, a finer tip has been developed with instrument sensitivity enabling significantly improved resolution, to about 10-30 microns (almost 10 times better resolution).

This resolution enables new studies, including compound interfaces and properties close to steel cords and fabric cords.

Examples of these will be shown. At the same time, preliminary work has begun on another improvement to this characterization technique. Efforts to obtain dynamic mechanical properties (storage modulus, loss modulus and tangent delta) from the indentation modulus technique will be described.

In the past, a modulus profile of polymers was measured using an indenter tip that had a radius of 71 µm.[3] Modulus profiling provides a method to understand material properties and material property changes in new and used components (rubber and plastic components).

The current work has produced an instrument with a resolution of about 30 microns (33 points per millimeter).

The finer tip was produced by grinding technology. The new tip has a radius of 8.37 µm as compared to the pri-

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or art that used a tip with a radius of 71 µm. The 8.4 µm tip when used at similar strains as those produced with the 71 µm tip yielded similar modulus values.

This technique provides useful information about the modulus profile of a polymer component.

The higher resolution can provide more accurate measurements of the modulus at critical areas of a material such as the transitions between polymer layers in a tire.

Method

Two loads are applied to a parabolic tip with a radius of 8.37.m, **Fig. 1**. From the Hertz relationship, the modulus of the material can be calculated.

This process and calculation are described in Gillen's "Modulus Profiling of Polymers." This invention uses a smaller tip than the prior art, which provides greater positional resolution of the modulus along the profile of the component.

Results and discussion

A summary of the specifications of the two modulus profilers with the varying tip radii is shown in **Table I**.

Table I compares the two tips and their effect on stress, strain and modulus.

Although the loads and tip radius are about one-tenth, the stress and strains are about twice. As a result, the contact radius is about one-fourth. The modulus results are similar.

Fig. 8 and **Fig. 9** show how a similar modulus is calculated for Sample 1.

Figs. 10, 11 and **12** examines the innerliner and plycoat region of a tire. Higher positional resolution was obtained with the new tip (about three times higher).

The modulus profile for Sample A is shown in **Fig. 2** and **3**, with 71 µm tip and 8.37 µm tip respectively.

The modulus profile for Sample D is shown in **Figs. 4, 5**, and **6** with each profiler tip and major load as specified.

Conclusion

This technique provides useful information about the modulus profile of a polymer component.

The smaller tip allows for higher special resolution of the modulus within a polymer component (or laminate).

This can provide more detailed information about the modulus in critical areas of a material such as the transitions between polymer layers in a tire.

Introduction

In the past modulus profiles of polymers have been measured using an indenter tip that had a radius of 71 µm and with the sample only experiencing static conditions.[3]

The modulus profiling technique used with the dynamic modulus profiler provides useful information about a material's properties under dynamic conditions.

From these measurements the modulus of the material can be calculated. Because the indenter tip has a small radius (1.4mm), this allows for many measurements along the material that shows the dynamic mechanical properties as a function of position.

Method

The new technique uses a Metravib DMA +150. Dynamic conditions were applied to the material using the Mertavib DMA +150. The indenter was a round tip with a known radius (shown in **Fig. 7**) making indentations in one spot at a high frequency.

The instrument includes x-direction and y-direction positioning devices, an alignment device, and sample holder attached on the lower clamp of the Metravib. The indenter tip was attached to the upper jaw of the Metravib.

See **Modulus,** *page 18*

Fig. 1. Finer tip produced by ARDL.

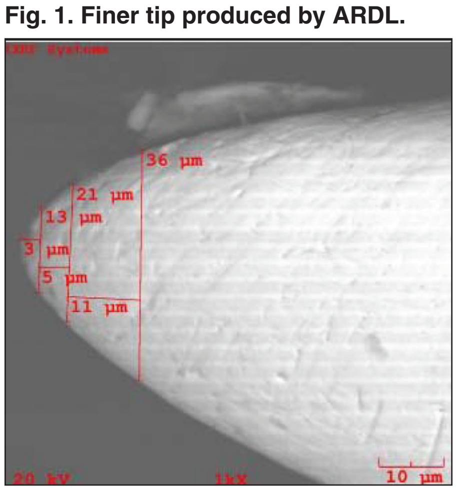


Table I. Summary of modulus profilers with varying tip radii.

	Sample #1	
Tip Radius:	71um	8.37um
$M_{contact}$	0.300g	0.040g
M_{major}	1.913g	0.280g
Avg. $r_{c,contact}$ (contact radius at $M_{contact}$):	25.0um	5.98um
Avg. $r_{c,major}$ (contact radius at M_{major}):	46.3um	11.4um
$A_{contact}$ (stress)	1.5MPa	3.50MPa
A_{major} (stress)	2.8MPa	6.69MPa
Avg. Strain at $M_{contact}$	0.20	0.40
Avg. Strain at M_{major}	0.37	0.77
Average Modulus	7.6MPa	8.6MPa

Fig. 2. Modulus profile of Sample A using tip with a 71-micron tip radius.

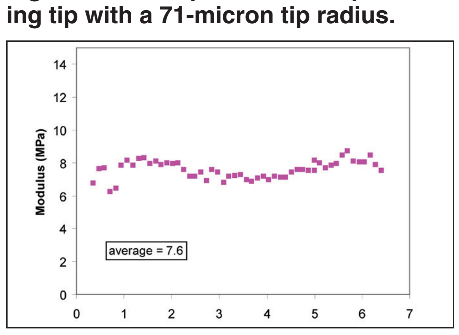


Fig. 3. Modulus profile of Sample A using tip with an 8-micron tip radius.

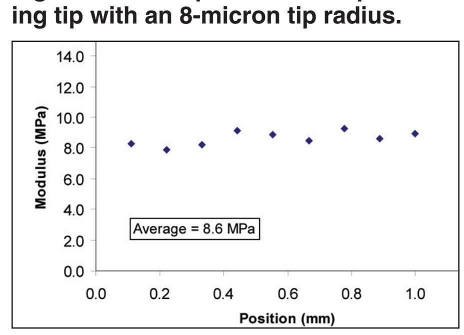
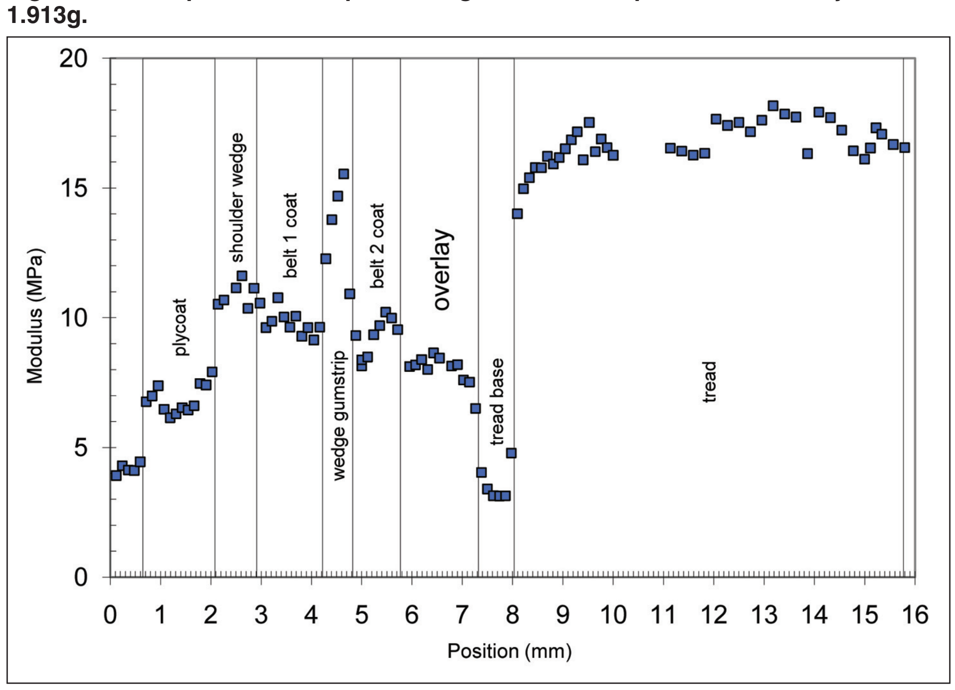


Fig. 4. Modulus profile of Sample D using a 71-micron tip radius and a major load of 1.913g.



Modulus

Continued from page 17

Results

The modulus profiles of two samples were collected under static conditions and under dynamic conditions.

The static conditions used a tip with a radius of 71 µm.

The dynamic stiffness, tangent delta, and the static modulus results are shown as functions of position for Sample 1 in **Figs. 8, 9** and **10**, respectively.

The dynamic stiffness, tangent delta, and static modulus results are shown as functions of position for Sample 2 in **Figs. 11, 12** and **13**, respectively.

Discussion

The stiffness results (shown in **Figs. 8** and **11**) correlated with the static modulus results (shown in **Figs. 10** and **13**).

This shows that the dynamic modulus profiler is able to measure compound stiffness correctly.

Conclusion

This technique provides useful information about a material's dynamic properties.

The measurements potentially provide useful information into how materials behave in a dynamic application.

The use of the small tip taking measurements along the material's profile can also give information about the modulus as it may vary with position on the material.

This can provide information particularly useful in materials that the material properties change through it such as a rubber article (laminate).

Example articles include golf balls and the rubber layers that make up a tire.

References

1. Rubber World, 231, No. 1 [2004] 40.
2. Polymer Degradation and Stability 17 [1987] 31-47.
3. Gillen, Kenneth, et. al. "Modulus Profiling of Polymers," Polymer Degradation and Stability. 17 (1987): 31-47.

Fig. 5. Modulus profile of innerliner and plycoat of Sample D using a 71-micron tip radius and a major load of 1.913g.

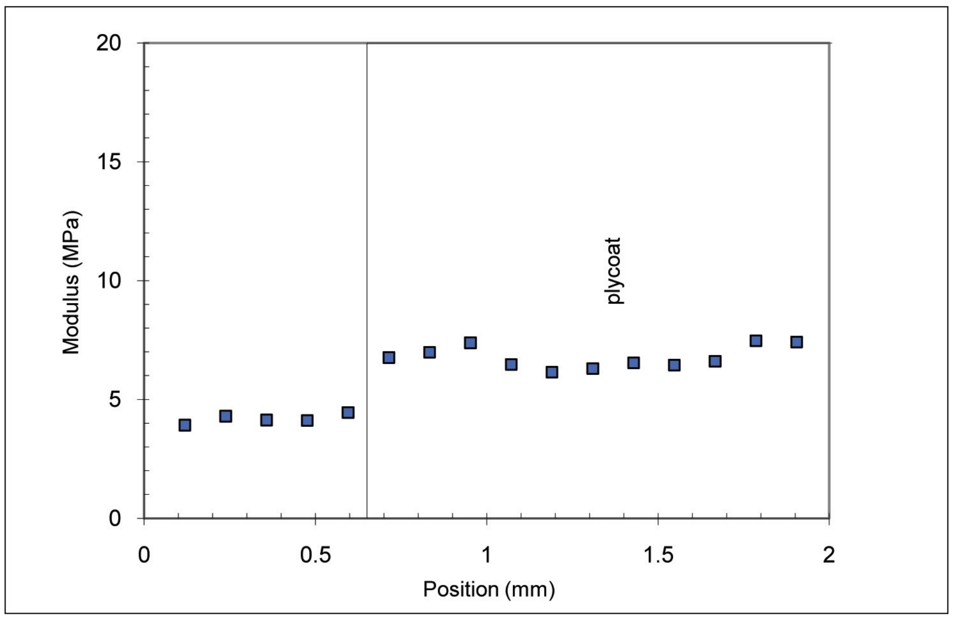


Fig. 6. Modulus profile of innerliner and plycoat of Sample D using an 8-micron tip radius and a major load of 0.26g.

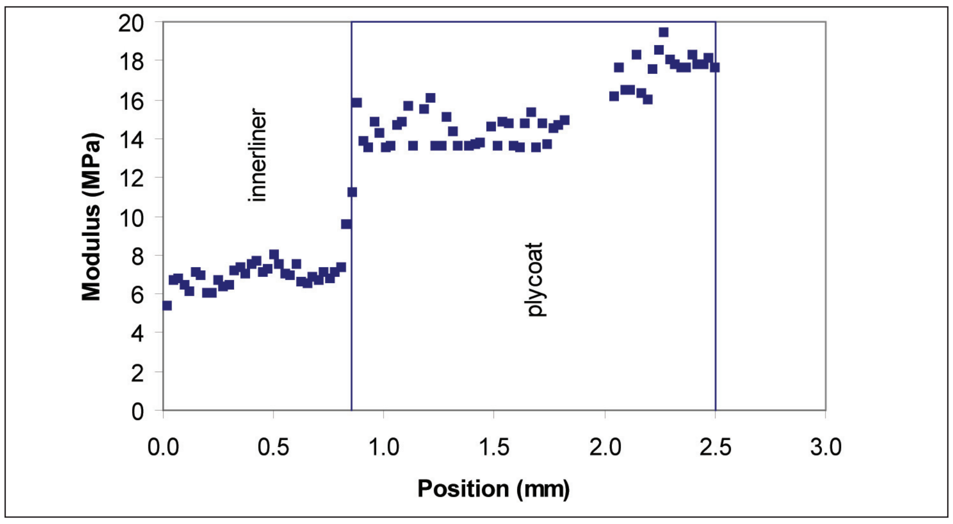


Fig. 7. Metra vib modulus profiler tip (tip radius = 1.4 mm). In the shown scale, each line with a number represents one millimeter.

Fig. 8. Dynamic stiffness of Sample 1 as a function of position.

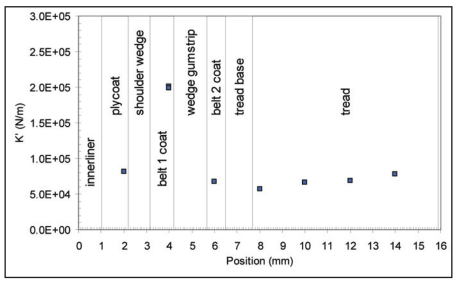


Fig. 9. Tangent Delta of Sample 1 as a function of position.

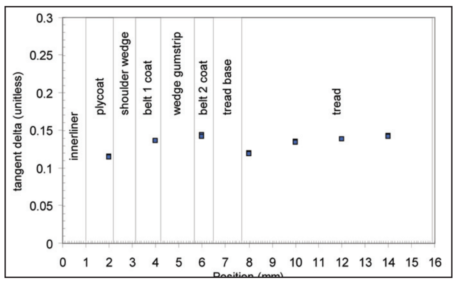


Fig. 10. Static modulus of Sample 1 as a function of position.

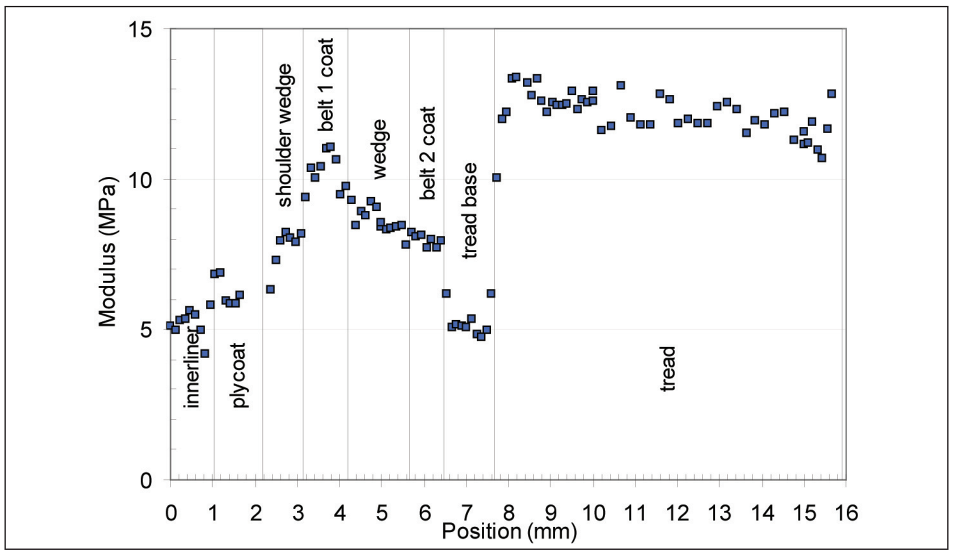


Fig. 11. Dynamic stiffness of Sample 2 as a function of position.

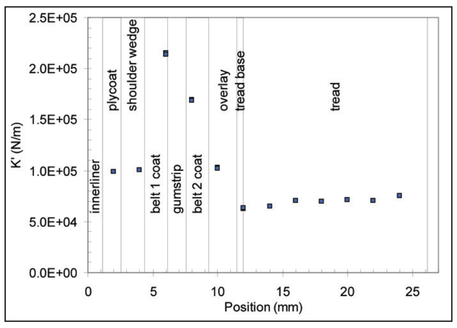


Fig. 12. Tangent Delta of Sample 2 as a function of position.

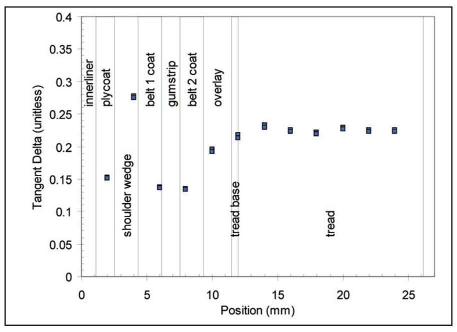


Fig. 13. Static modulus of Sample 2 as a function of position.