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Acoustic Spectrometer – Innovative Tool in Rubber Characterization

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I. Introduction

Reinforcing properties of carbon black are very well documented in the literature [1], however, the effects of carbon black on specific tire properties *(i.e.* tire traction) are not always fully predicted by existing tire material characterizations. The existing approaches to characterize traction properties of tread compounds, may be able to differentiate between polymers but do not predict the carbon black influence on tire traction behavior. Current tire traction testing suggests that high frequency viscoelastic properties of tread compounds may be used to predict this important characteristic. It can be shown that when tire is locked, at 100 km/h (GM trailer test), the deformation of the rubber at the tread-road interface occurs around the MHz frequency range. Ultrasonic technique seems to be the right choice to assess the information at high frequency strain input. Ultrasonic methods as non-destructive testing of materials are used in many areas of material science [2,3]. The propagation characteristics (attenuation coefficient α, and sound velocity υ) of ultrasonic waves are known to depend upon the elastic and viscous properties of the material in which they propagate. Therefore, they may be used to study the viscoelasticity of rubber compounds. In addition to predict tire traction, ultrasonic technique could directly probe the polymer-filler interactions otherwise difficult to determine by other experimental techniques.

In the present paper, the experimental and theoretical aspects evaluated in order to build the high frequency automatic spectrometer, will be presented.

II. Experimental

The high frequency automatic acoustic spectrometer was built using the principle of ultrasonic signal propagation in condensed media. It allows measurements of the longitudinal attenuation coefficient in the temperature range from -90°C to +60°C. This system is fully automatic and no attention of an operator is required during the entire measurement process. The Beer's law of absorption [2] is applied in transmission mode. Such technique requires that the number of samples of the same material with different thickness be measured under given conditions and the attenuation coefficient will be calculated from the following equation: $I = I_0 e^{(-\alpha d)}$, where α is the longitudinal attenuation coefficient. The transmission mode of measurements requires two identical transducers. One piezoelectric transducer is used as a transmitter that generates ultrasonic waves. When a high frequency electrical signal from the pulser/receiver (see Fig. 1) is applied to the piezoelectric crystal, it vibrates according to its characteristic frequency. These mechanical vibrations are in turn detected by the second transducer which functions as a receiver. In the receiver, the mechanical vibrations are converted into an electrical signal, the magnitude of which is proportional to the intensity of the ultrasonic signal. Ethanol is used as a coupling fluid, its low freezing point provides a media able to cover a wide range of temperatures. Ethanol also wets well the rubber surface, which maximizes the ultrasonic wave propagation. The reflection at the interface between the immersion fluid and the rubber sample are minimized by the fact that the acoustic impedance of the ethanol is very close to that of the rubber sample.

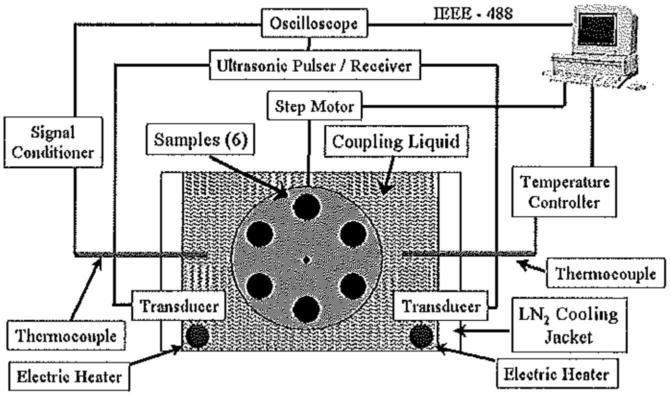

Fig. 1 Experimental setup.

All signals are monitored on a digital signal-processing oscilloscope, equipped with Fast Fourier Transform (FFT) capability. The FFT is performed and the area under the FFT is calculated "live" on the oscilloscope and consequently stored in the computer via a GPIB port.

III. Results

In order to evaluate the high frequency spectrometer as a tool to characterize the high frequency viscoelasticity of rubber compounds, the following variables have been studied: polymer type, carbon black loading, carbon black grade, filler dispersion. These studies were performed over a wide range of temperatures.

Carbon Black Loading Study

The loading data indicate that both, the attenuation coefficient and the sound velocity increase significantly with increased carbon black loading. As it can be seen in Fig. 2, where room temperature data were extracted from temperature sweep experiments, the attenuation coefficient α, or the sound velocity υ, obtained for different grades of carbon black could not be distinguished at low carbon black loadings. However, above 40 phr one could observe distinctive differences between different carbon blacks. The higher attenuation coefficient obtained for some blacks (XLH81) suggests that the dispersion may be responsible for the higher sound attenuation phenomena. The sound velocity could be correlated with the modulus of the compound. These data also suggest that the XLH81 black have a lower modulus, which can be related to better micro-dispersion.

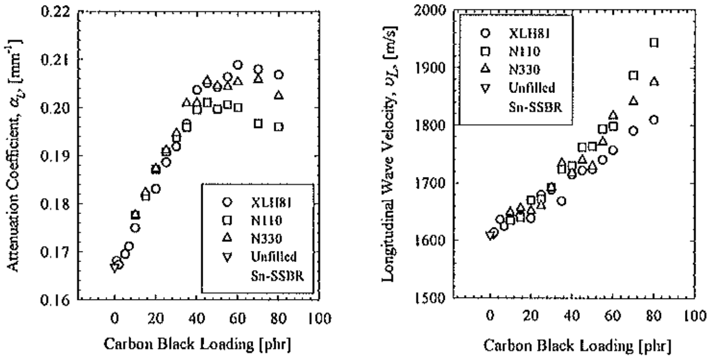

Fig. 2 Attenuation Coefficient and Sound Velocity vs. Carbon Black Loading.

Role of Carbon Black

There is an interesting dependence of the sound velocity, υ, with an increasing N_2SA (50 phr loading). This can be explained by the increase of the modulus of the rubber compound [2].

Carbon Black Micro-Dispersion Study

The level of micro-dispersion was monitored by changing mixing time. It is assumed that the longer mixing time the better the micro-dispersion. The evaluation of micro-roughness (Mechanical Scanning Microscopy - MSM) corroborate these assumption and correlate well with the attenuation coefficient (Fig.3). The better the microdispersion the more polymer chains are immobilized.

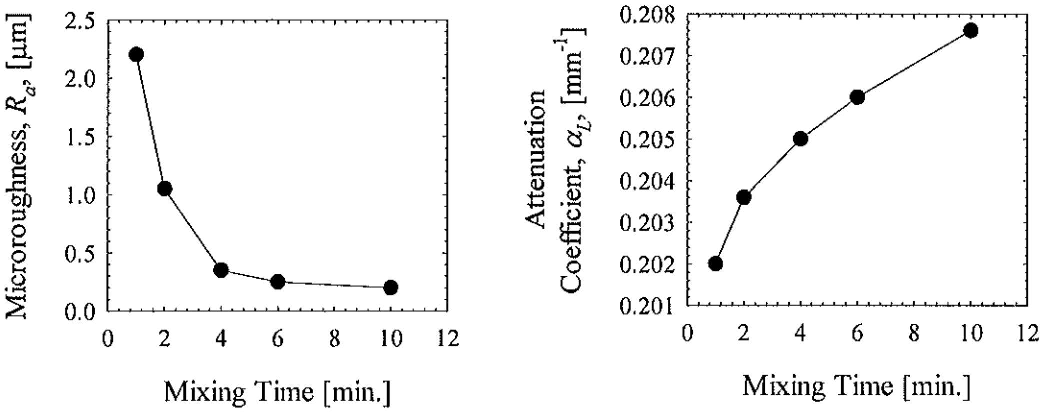

Fig. 3. Micro-roughness and Attenuation Coefficient obtained for the compounds prepared with Sn-SSBR.

Polymer Type

Temperature sweep experiments are ideal to investigate the polymer response to the ultrasonic signal. The results obtained for unfilled polymers, as well as for carbon black filled compounds, indicate the temperature shift of the glass transition temperature, T_g, toward higher temperatures.

The values of the attenuation coefficient for the unfilled polymers coincide with the values obtained for the filled compounds only in the glassy stage. Above T_g the ultrasonic energy in the unfilled compounds is dissipated at a slower rate than in the filled compounds.

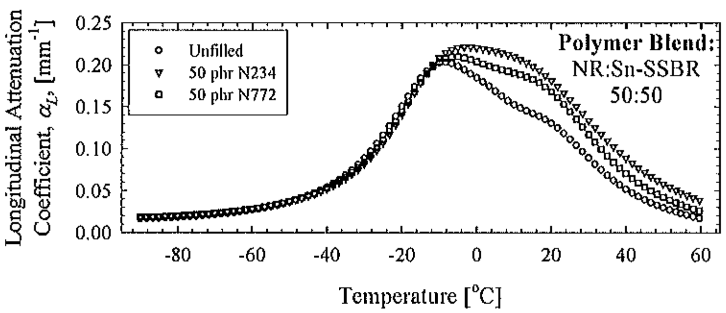

Fig. 4. Attenuation Coefficient obtained for unfilled and carbon black filled compounds.

Polymer Blend System

The morphology of the polymer blends system depends on composition, processing conditions, rheological properties, and thermodynamic miscibility of the components. Depending on the blend composition, either one phase forms the continuous phase and the other the discrete phase or co-continuous phases may exist. For the filled polymer blends, the distribution of the filler between polymer phase and the dispersion of the filler in individual phase plays a role in determining the physico-mechanical properties.

The high frequency measurements allow one to see the degree of compatibility between the polymer blends systems, as well to elaborate on the preferable filler distribution [3].

Filler-Polymer Interactions

Temperature sweep experiments are also used to evaluate the filler-polymer interactions. This could be done, by comparing the data obtained for unfilled and filled compounds. More polymer-filler interactions will lead to the following behavior: the difference in the value of the attenuation coefficient at the maximum between filled and unfilled compounds will increase, the difference in the position of maximum on the temperature scale will increase and the width of the high frequency spectrum will increase for the filled compounds as compared to the unfilled counterpart.

Tire Traction Prediction

The correlation obtained for the predictive tire traction testing depends on the actual tire testing methods. Good correlations were established between the wet traction as tested by the GM trailer and the attenuation coefficient measured at room temperature and frequency 1MHz. The obtained correlation coefficient was in the order of 0.95 as compared to less than 0.5 obtained by testing tanδ at 0°C.

IV. Conclusions

As it can be seen from this paper and the previously published results the high frequency acoustic technique can provide valuable informations where other experimental methods fail. Even if the polymer type has the greatest influence on the attenuation coefficient value as well as on the longitudinal wave velocity the filler seems to play a secondary role. Nevertheless, the effect of carbon black can be clearly observed.

All experiments suggests that carbon black dispersion has a large influence on the obtained results, suggesting the better filler micro-dispersion allows better energy dissipation (more filler-polymer interactions).

This technique could be easily adopted to cover wide frequency range by utilizing transducers with different characteristic frequency.

References

[1] J. B. Donnet, R. C. Bansal, and M. J. Wang, *"Carbon Black, Science and Technology"*, Mercel Dekker, Inc., New York, 1993.

[2] M. Gerspacher, C. P. O'Farrell, L. Nikiel, H. H. Yang, and F. LeMehaute, *Rubber Chem. Technol.*, **69** (1996) 786.

[3] M. Gerspacher, C. P. O'Farrell, L. Nikiel, and H. H. Yang, *Rubber & Plastic News,* **August 26**, (1996) 39.