Characterization of Cured Rubber by DMA

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INTRODUCTION

DMA measures the modulus (stiffness) and damping (energy dissipation) properties of a material as it is deformed under a periodic stress. These measurements provide quantitative and qualitative information about the performance of materials. DMA is particularly useful for evaluating elastomeric materials that exhibit time, frequency, and temperature effects on mechanical properties because of their viscoelastic nature.

The determination of the storage modulus and glass transition temperature is often used to characterize the cure profile of rubber materials. Differences in these properties correspond to differences in cure. DMA is used to examine the cure properties of two rubber samples each cured for different lengths of time.

EXPERIMENTAL

The following experimental conditions were used to characterize the two samples:

Clamping Geometry:	Film-Tension Clamp
Temperature Range:	-70 to 50 °C
Heating Rate:	5 °C/min
Amplitude:	15 µm
Frequency:	1 Hz

Four elastomer samples were tested. The "old" sample was an aged formulation of the fresh, "new" sample. Both samples were treated to two cure times of 1.0 and 1.5 hours each before DMA characterization.

The film-tension clamps are calibrated for compliance prior to running of the samples. Should the clamp compliance not be considered, then the measured storage modulus results will be lower than actual values.

RESULTS AND DISCUSSION

Because of its inherent sensitivity, DMA is an ideal technique for the determination of the glass transition of elastomeric materials. In addition, absolute modulus values may be determined both below and above the glass transition. The storage modulus above the glass transition is related to the degree of cure (cross-link density) of the material; the higher the storage modulus, the higher the degree of cure.

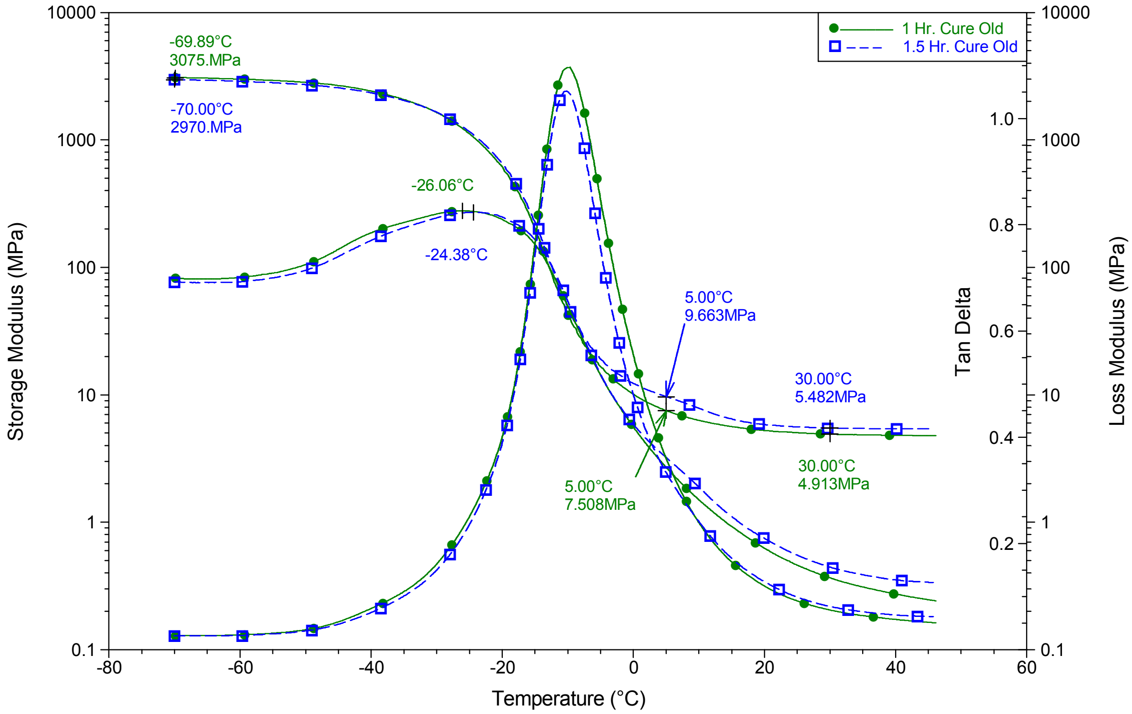

Figure 1 - "OLD" Rubber Samples, Different Cure Times

The glass transition temperature (Tg) is also an indication of degree of cure; the higher the Tg, the higher the degree of cure.

Figures 1 and 2 show DMA data for the two rubber samples. A longer cure time should lead to a higher degree of cure. This should result in a higher value of storage modulus (E') in the rubbery plateau (*1*). Figure 1 shows the values of E' for the old 1 hour and old 1.5 hour cured materials, respectively. There is a noticeable difference between the values with the E' of the 1.5 hour cured sample being 11 % higher than that of the 1 hour cured sample.

The new samples show similar results. Figure 2 shows the values of E' in the rubbery plateau. The 1.5 hour cured sample shows a storage modulus that is higher than the value of the 1 hour cured sample by 17 %.

REFERENCES

1. L.E. Nielsen, R.F. Landel, *Mechanical Properties of Polymers and Composites*, **1994,** Marcel Dekker, p 49.

KEYWORDS

cure, dynamic mechanical analysis, glass transition, modulus

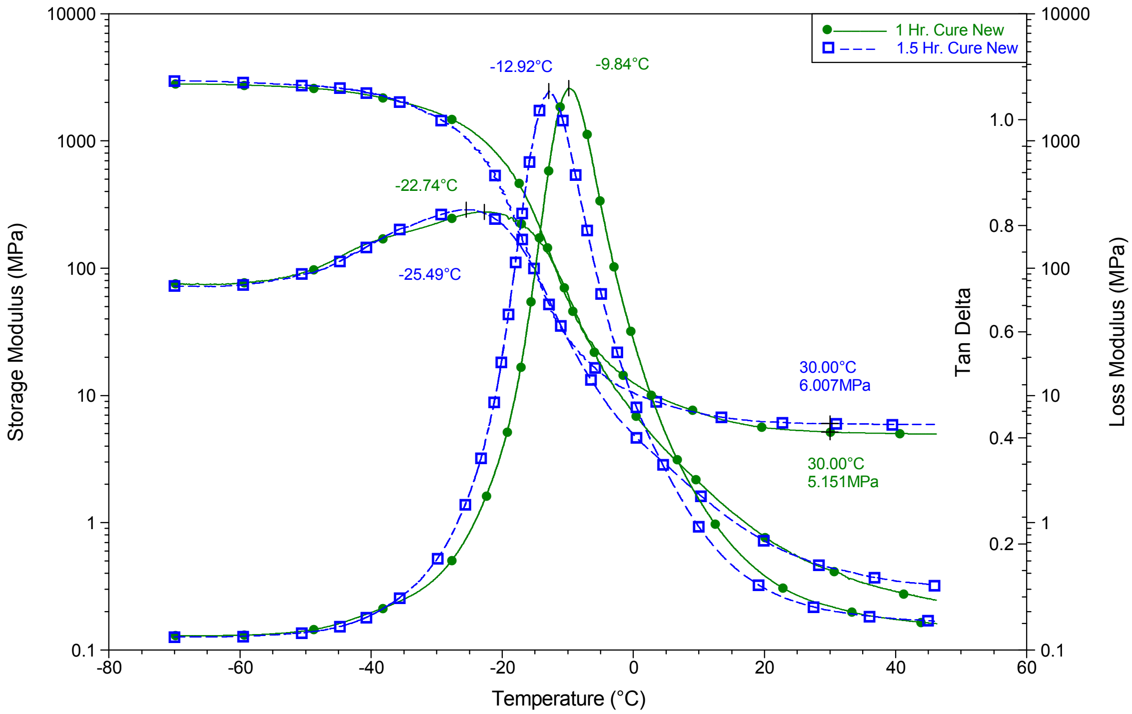

Figure 2 - “NEW” Rubber Samples, Different Cure Times

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