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Dynamic Viscoelastic Measurements of Vulcanized Rubber

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1.   Introduction

Vulcanized rubber, rubber treated with sulfur and organic polysulphides, is used widely as an industrial material. Creating crosslinks in the molecular structure of raw rubber through vulcanization decreases the rubber's thermoplasticity and improves elasticity, tensile strength, and abrasiveness, allowing the rubber to be used for industrial purposes. Vulcanization further improves the rubber's solvent resistance, heat resistance, and cold resistance. The crosslink density and crosslink distribution of vulcanized rubber varies with the amount of vulcanizing agent, vulcanizing catalyst, heat, time, etc. involved in the vulcanizing process.

The differences in the rate of vulcanization of vulcanized rubber, in other words the differences in the degree of crosslinking through vulcanization, can be investigated through dynamic viscoelasticity measurement. This Application Brief demonstrates the measurement of vulcanized rubber with differing crosslink densities using dynamic viscoelasticity measurement methods.

2.   Experiment Conditions

Samples for the experiment consisted of three types of fluoro-rubber (A, B and C) whose crosslink densities were modified through changing the amount of vulcanizing agent introduced. Sample A contains the least amount of vulcanizing agent, while samples B and C contain respectively greater amounts of agent.

The measurement devices used were a SDM5600H Rheol. Station connected to a DMS200 Dynamic Mechanical Spectrometer (Tension Module). The measurement frequency used was 1Hz. The measurement temperature ranged from -100   to 200   and the rate of temperature increase was 2 /min.

3.   Measurement results

Figures 1 through 3 show each of the dynamic viscoelasticity spectrums of vulcanized rubber A, B, and C. These figures show the E', E'', and tan   curves at a 1Hz measurement frequency. While seeing peaks on the tan   curves representing main dispersion (glass transition) around -15   to -10 , the E' curve shows a decrease in elasticity accompanying glass transition.
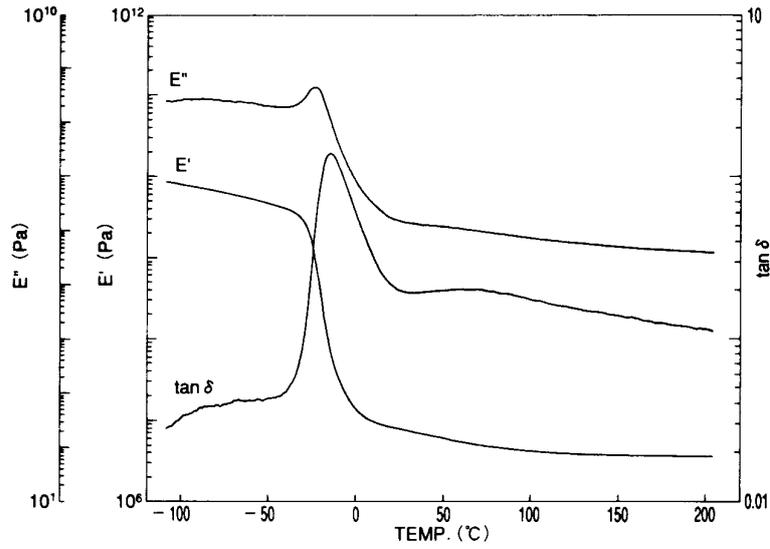
Figure 1   Dynamic viscoelasticity spectrum of vulcanized rubber A
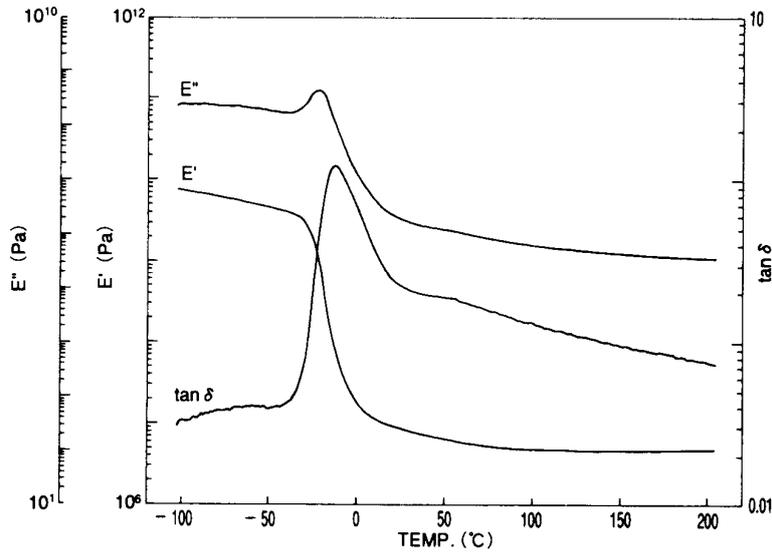

Figure 2   Dynamic viscoelasticity spectrum of vulcanized rubber B
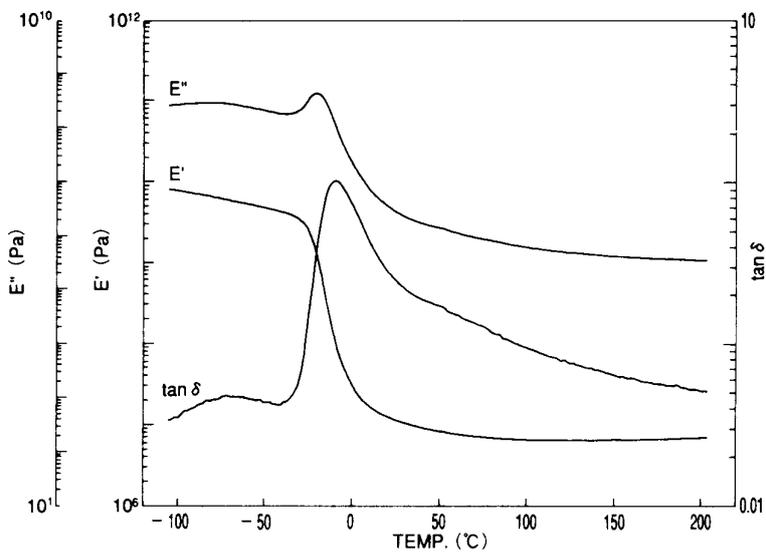

Figure 3   Dynamic viscoelasticity spectrum of vulcanized rubber C

Figure 4 shows the results of comparing the *E'* curve measurements for each sample in Figures 1 through 3. Sample C, which contains the highest amount of vulcanizing agent among the three types of vulcanized rubber, and therefore the highest crosslink density, has the highest *E'* curve from the transfer area through the section of the curve where the rubber is in a viscous state depicted by the flat area of the respective curves. Sample B and sample A have respectively lower curves over the same range. Previous evidence has shown that the storage elasticity rates along the flat section of the respective sample curves will be detected higher in elasticity the higher the sample's crosslink density. This report as well confirms that the sample with the highest crosslink density has the highest stored elasticity. Using this characteristic, other experiments have shown how to calculate crosslink density and inter-crosslink molecular weight from the storage elasticity rates along the flat section of the sample curves.
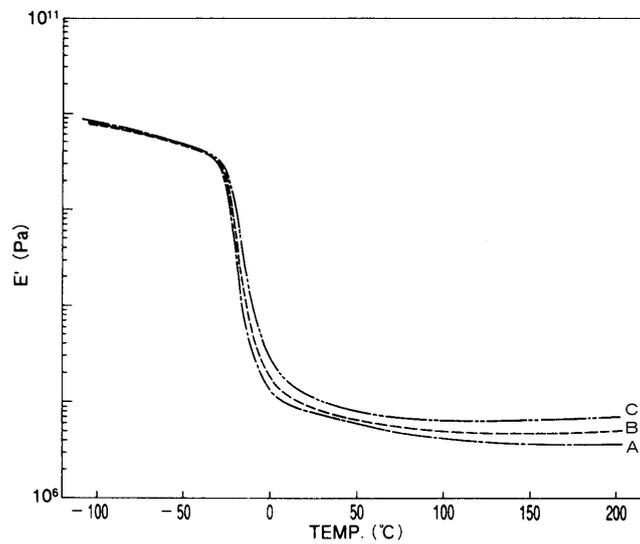

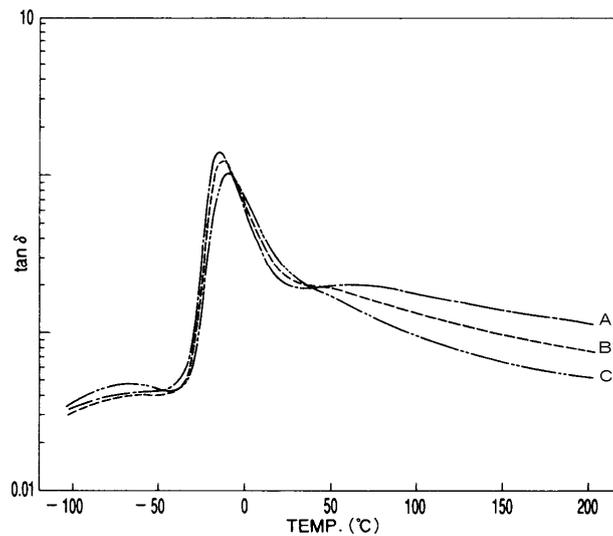Figure 4   *E'* curves of vulcanized rubber A, B, & C

Figure 5   tan δ curves of vulcanized rubber A, B, & C

Figure 5 shows the result of comparing the measurement results of the tan curve for each sample shown in Figures 1 through 3. Sample A, the sample among the three with the least vulcanizing agent and therefore the lowest crosslink density has the highest values over the bottom flat section of the curve, with B and C following in order with lower amounts. Furthermore, the higher the crosslink density, the more the peak of the main dispersion (glass transition) shifts toward higher temperature ranges and the more the peak range widens. The tan curve is lower the higher the crosslink densities. Other sources have indicated that the tan peak range widens at the same time it shifts toward higher temperatures. This trend is confirmed in the Figure 5 measurement results as well.

4. Conclusion

This Application Brief has investigated the influences of crosslink density on the dynamic viscoelectricity of vulcanized rubber. In a wider sense, this useful procedure can be applied to other cross-link polymers in determinomg their physical properties. Dynamic viscoelectricity measurement is used widely as an effective method to understand the influence of the crosslink rate of various crosslink polymers.

Supplementary Note