



Alternate Instrumental Methods of Measuring Scorch and Cure Characteristics*

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ABSTRACT

After the development of the oscillating disk curemeter over 30 years ago, standard conditions for measuring scorch times (time till the onset of vulcanization) were developed. ISO 3417 and ASTM D2084 describe t_{s1} and t_{s2} test parameters which are accepted by the rubber industry as a method for measuring scorch times. t_{s1} is the time until one torque unit rise above minimum torque and t_{s2} is the time until two torque units rise above minimum torque. These parameters are arbitrary and do not measure the 'true' scorch time. Also t_{s1} and t_{s2} may not be an accurate comparison for compounds which differ greatly in their state of cure. Experience has shown that some rotorless curemeter designs are far more sensitive to physicochemical changes occurring during the cure process than the conventional oscillating curemeters. The MDR 2000[®] rotorless curemeter (MDR) and the RPA 2000[™] Rubber Process Analyzer (RPA) measure subtle changes in viscoelastic properties

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during the curing process. This paper discusses the feasibility of using the time until the viscous torque (S'') peaks ($t_{\max S''}$) to more accurately measure the 'true' scorch time. Other techniques for measuring cure characteristics are also reviewed for comparison.

INTRODUCTION

Scorch time is defined by ASTM as the time to incipient cure crosslinking under specified isothermal conditions.¹ Scorch time is also referred to as 'time to scorch', 'scorch delay' or 'induction time'. At scorch, the mixed stock becomes partially vulcanized (the onset of vulcanization). At this point the stock also loses its plastic quality thus preventing the stock from being processed further.^{2,3}

The scorch time of a batch of stock depends on the stock's heat history. Higher process temperatures and/or longer process time reduce the scorch time. Likewise, higher bin storage temperatures and/or longer bin storage times reduce the scorch time of a mixed stock.⁴

Effective measurement of the scorch time of a mixed stock predicts any premature vulcanization in 'downstream' processes (such as extrusion and calendaring) prior to the final cure stage. If scorch frequently occurs before the stock passes through these 'downstream' processes, high scrap rates result. Therefore, a stock's scorch time is very important for factory quality assurance. The following are test methods which can be used to measure scorch time.

MOONEY: TIME-TO-SCORCH (t_5)

The Mooney viscometer has been used to measure time to scorch since the 1940s.⁵ The Mooney viscometer measures the viscosity of a mixed rubber stock from the torque required to rotate a disk at 2 rpm while embedded in a rubber specimen within a sealed cavity at a constant temperature. It was the first instrumental method used to measure scorch safety of a mixed stock. ASTM D 1646 describes the manner in which Mooney time-to-scorch is to be reported. Mooney scorch is generally reported for a large rotor at the time required for the viscosity to rise 5 Mooney units above the minimum. This is generally denoted as t_5 . Figure 1 shows how this property is measured.

CUREMETER: SCORCH TIME (t_{s1})

The oscillating disk curemeter was developed in the early 1960s.⁶ This instrument measures the torque required to oscillate (not rotate) a biconi-

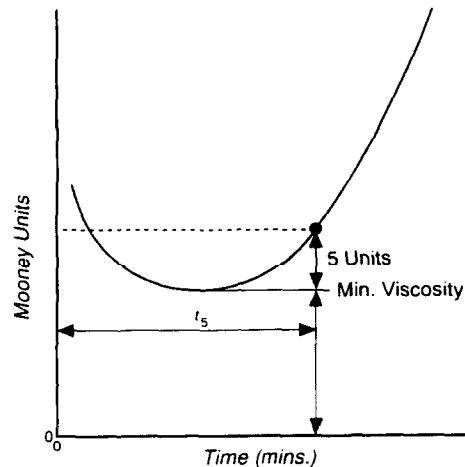


Fig. 1. Typical measurement of t_5 Mooney scorch time in accordance with ASTM D 1646.

cal disk while embedded in a rubber specimen. This disk oscillates through a preset shear strain (rotational amplitude) at a constant frequency and temperature. Not only can the oscillating disk curemeter measure scorch and cure characteristics, it also measures the stiffness of a fully cured compound which a Mooney viscometer cannot. ASTM D 2084 describes this test method.¹

Curemeter scorch time is commonly reported at the one degree arc setting as the time required for the torque to rise one torque unit (either in dN-m or in-lb) above the minimum elastic torque M_L . One problem with this t_{s1} scorch parameter is that it is dependent on which torque units are being used. Another problem with t_{s1} occurs when comparing compounds with greatly different maximum elastic torque values (M_H). A one unit rise for a compound with a low M_H represents a much larger percent of the ultimate state of cure than for a compound with a relatively high M_H .

This curemeter scorch measurement is analogous to the Mooney scorch measurement described earlier. However, curemeter scorch times and Mooney scorch times do not always correlate exactly. This may be because the curemeter measures elastic torque (S') or the torque response that is in-phase with the applied strain. (The viscous torque (S'') is 90° out-of-phase with the applied strain and complex torque (S^*) is related to S' and S'' as follows: $(S^*)^2 = (S')^2 + (S'')^2$.) Decker *et al.* reported in 1963 that when the complex torque (S^*) is substituted for the elastic torque (S') and the curemeter oscillation frequency is increased, better correlations were achieved between the Mooney scorch and curemeter scorch tests.⁷

Rotorless curemeters were introduced in the 1980s. These instruments are replacing the oscillating disk curemeters because of their faster temperature recovery, improved test sensitivity and better precision.^{8,9} ASTM D 5289 describes how t_{s1} can also be applied to the rotorless curemeter for measuring scorch time.

The use of 'one unit rise' is somewhat arbitrary; however, scorch cannot be accurately determined from the time where minimum S' torque occurs ($t_{\min S'}$) because it does not correlate with true scorch.

CUREMETER: 10% CURETIME (t_{c10})

ASTM D 5289 also permits t_{c10} to be used as another method for measuring scorch. t_{c10} is the time to reach a 10% state of cure. It is defined as the time required to reach the elastic torque value of $(M_L + 0.10(M_H - M_L))$ where M_H represents the maximum elastic torque and M_L represents the minimum elastic torque (Fig. 2). One advantage of using t_{c10} over t_{s1} as a measure of scorch time is that it is independent of what torque units are selected. By definition, t_{c10} occurs at 10% of the distance from M_L to M_H . Therefore, t_{c10} may be preferred when comparing scorch characteristics of different compounds which vary significantly in maximum torque values. However, t_{c10} still cannot indicate exactly at what point a material will no longer process.

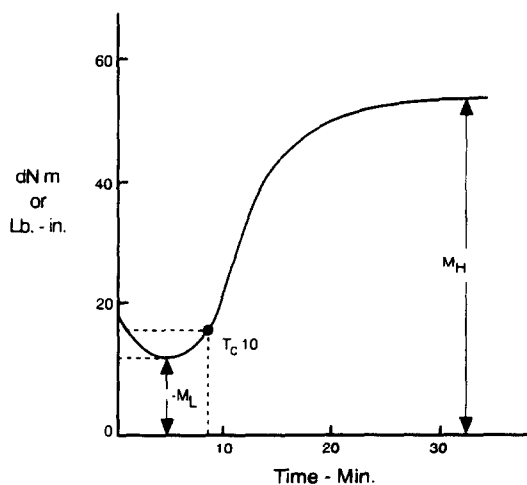


Fig. 2. Typical measurement of curemeter t_{c10} curetime in accordance with ASTM D 2084.

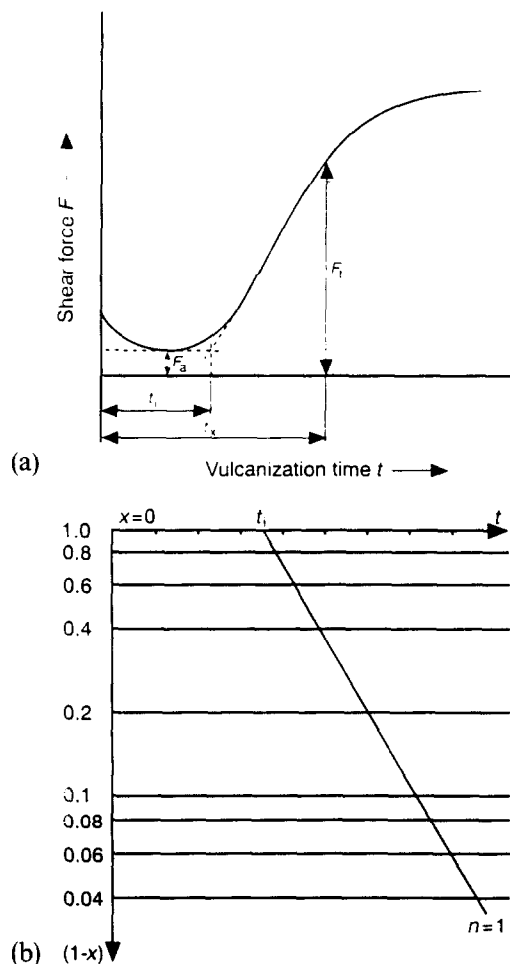


Fig. 3. (a) Illustration of the t_i incubation time in accordance with DIN 53529. (b) Determination of t_i incubation time from log of conversion $(1-x)$ versus time for a first-order reaction in accordance with DIN 53529.

CUREMETER: INCUBATION TIME (t_i) FROM REACTION KINETICS

Some curemeters have the isothermal conditions necessary to measure the incubation time (t_i) using reaction cure kinetics principles cited in the German standard DIN 53529 (Fig. 3). Software programs are available for use with some curemeters to automatically calculate the t_i values.^{10,11} As part of the calculations, a conversion rate constant at the cure temperature is determined from the slope in a plot of log of conversion ($\log(1-X)$)

versus time for a first-order reaction where X is the fraction converted or reacted. By extrapolating to $X = 0$, t_1 can be determined. This incubation time (t_i) includes the time lag attributed to chemical processes as well as the instrument thermal lag. A more isothermal rotorless curemeter minimizes the time for a new sample to reach the test temperature and improves the accuracy of t_i .

CUREMETER TIME TO PEAK VISCOUS TORQUE ($t_{\max S''}$)

The development of the rotorless curemeter also led to the precise measurement of viscous torque (S'') during a test in addition to the usual elastic torque response (S') which is also obtained from the oscillating disk curemeter. Sinusoidal deformation is an ideal way to measure the elastic and viscous torque responses from a rubber sample. In the MDR 2000[®] rotorless curemeter (MDR), the lower die oscillates while the transducer measures the torque response at the upper die to minimize noise. The elastic torque response (S') is in-phase with the applied strain and a viscous torque response (S'') is 90° out-of-phase with the applied strain. This relationship is illustrated in Fig. 4. Oscillating disk curemeters cannot measure S'' with precision because of friction associated with the rotor.

The shape of a typical S'' cure curve from a rotorless curemeter is very different from the conventional S' cure curve in either an oscillating disk curemeter or a rotorless curemeter. Figure 5 shows the shape of a typical S'' cure curve along with the corresponding S' cure curve. On approaching the onset of vulcanisation, the viscous torque (S'') typically starts to rise early in the cure process and reaches a maximum level shortly after the 'scorch time' is reached. The S'' curve then falls as the crosslink density increases to the ultimate state of cure. The cured viscous torque usually levels off at a significantly lower level. This is a characteristic pattern observed repeatedly

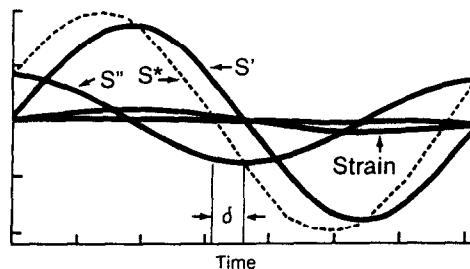


Fig. 4. RPA 2000[™] torque responses to a given sinusoidal applied strain.

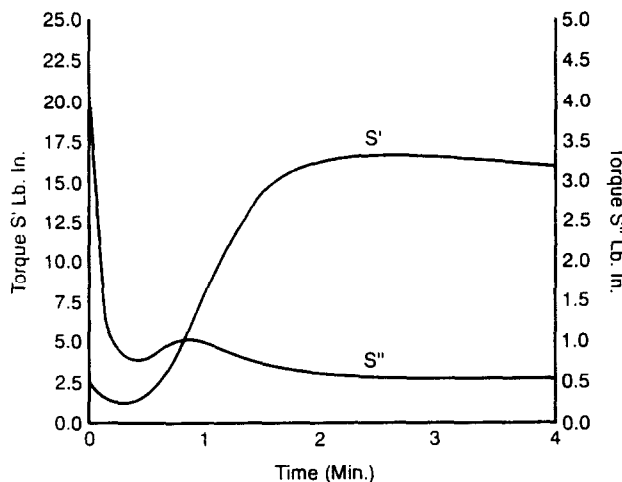


Fig. 5. Typical elastic torque (S') and viscous torque (S'') cure curves generated from a rotorless curemeter.

in a wide variety of formulations. The peak in S'' during cure has also been observed with other dynamic mechanical rheological testers.^{1,2}

The time at which the peak in S'' occurs ($t_{\max S''}$) during cure is usually shortly after the scorch time is reached. This paper presents data from our studies on the feasibility of using the $t_{\max S''}$ as a measure of scorch time.

NATURE OF $t_{\max S''}$

Most rubber compounds during cure show a peak in the S'' cure curve. After reviewing curemeter test results from a wide variety of compounds, an S'' peak usually occurred shortly after incipient cure (usually occurring between t_{C1} and t_{C40}). These compounds included a wide spectrum of formulations using general purpose elastomers (such as NR, SBR and BR) and speciality elastomers (fluoroelastomers, IIR and NBR). In some cases, the S'' peak was sharp and well defined. In other cases, the S'' peak was broad and not as well defined. Sometimes there was just a 'bulge' or distortion where the S'' peak might normally appear. However, an S'' peak usually appeared in some form. There were two broad classes of rubber compounds which did not show any sign of an S'' peak. One class was natural rubber compounds with low filler loadings. This is illustrated below in an experiment with six natural rubber isolator stocks. The second class was represented by rubber compounds with extremely high loadings of filler (in excess of 600 total parts per hundred (phr)). This latter group of compounds have relatively low rubber content. These compounds are discussed in the last section of this paper.

Table 1 gives the formulations for the six natural rubber isolator stocks which differ in Carbon Black and oil loadings. These compounds were tested for cure characteristics on a Monsanto RPA 2000™ Rubber Process Analyzer (hereafter referred to as 'RPA') at 0.1° arc strain, 50 cycles per minute (cpm) and 140°C . Figure 6 shows the S' and S'' cure curves for compound number 1. As can be seen, the S' and S'' cure curves are almost mirror images of each other with no visible S'' peak. There was also no S'' peak for compounds 2 and 3. Only at higher loadings of Carbon Black and oil do we start to see the S'' peak appearing at incipient vulcanisation. Thus, a natural rubber vibration isolator compound with low loadings of Carbon Black and oil does not give an S'' peak during the curing process. Other examples of compounds with no S'' peak will be discussed later.

TABLE 1
Vibration Isolator Stocks (Parts Per Hundred)

Ingredients	Compound number					
	1	2	3	4	5	6
SMR 10, NR	100	100	100	100	100	100
Zinc oxide	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
N330, HAF	10	30	50	25	45	75
Calcium carbonate	20	20	20	20	20	20
Aromatic oil	5	25	45	5	25	45
6PPD	2	2	2	2	2	2
CBTS	0.5	0.5	0.5	0.5	0.5	0.5
Sulphur	1.5	1.5	1.5	1.5	1.5	1.5
Total	146.0	186.0	226.0	161.0	201.0	251.0

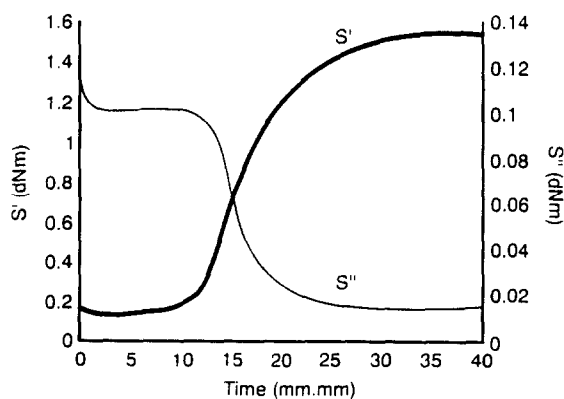


Fig. 6. Elastic torque (S') and viscous torque (S'') cure curves of vibration isolator compound with only 10 phr N330 and 5 phr oil. Test conditions: $\pm 0.1^\circ$ arc, 50 cpm, and 140°C .

The exact reason for the S'' peak during cure is not known. It is possible that the viscous torque S'' rises as it approaches the onset of vulcanisation because of crosslinks forming among rubber chains before an overall loose crosslinked matrix is established. Another possibility is that the rise is due to formation of rubber- S_x -Ac pendent groups which form during the cure induction period before the scorch time.¹³ These polysulfide groups could restrict flow, hindering the movement of rubber chains past one another. On the other hand, at some point during the vulcanization process, the S'' falls as a crosslinked matrix is established.

EFFECT OF OSCILLATION FREQUENCY on $t_{\max S''}$

An experiment was conducted to determine what effects changes in test oscillation frequency have on the S' and S'' cure curves. A model SBR formulation with 50 phr N330 Carbon Black was selected for this study (Table 2). The RPA was used to conduct cure tests at different preselected oscillation frequencies.

Figure 7 shows the S' and S'' cure curves resulting from performing the cure tests on the RPA at different frequencies of oscillation. As the frequency of the cure test increases, the scorch time as indicated by the S' curve and t_{s1} does not change greatly. However, as frequency of the cure test rises, the $t_{\max S''}$ time becomes shorter and shorter. At 2 cpm the $t_{\max S''}$ occurs where S' reaches $t_C 50$. However, when the same cure test is conducted at 100 cpm, the $t_{\max S''}$ occurs where S' reaches $t_C 20$ and at 1000 cpm the $t_{\max S''}$ occurs at $t_C 5$. Normally cure tests conducted with most curemeters are performed at 100 cpm. As can be seen from the results of this experiment, the $t_{\max S''}$

Table 2
Model SBR Formulation

<i>Ingredients</i>	<i>Parts per hundred</i>
SBR 1500	100
Carbon Black	50
Aromatic oil	20
Zinc oxide	3
Stearic acid	1.5
TMQ	1.0
TBBS ^a	1.25
Sulphur ^a	2.0
Total	178.75

^aCuratives added on the mill.

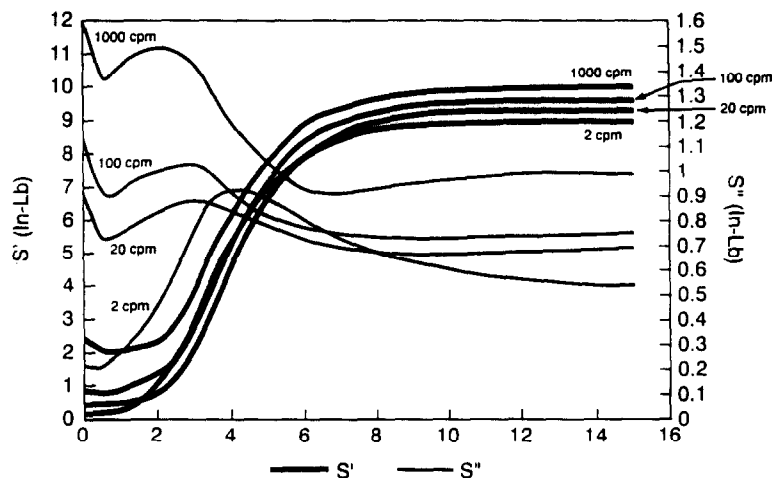


Fig. 7. Changes in the viscous (S'') and elastic (S') cure curves from changes in frequency in testing the model SBR compound with 50 phr N330 Carbon Black. Test conditions: $\pm 0.5^\circ$ arc and 170°C .

approaches the 'true scorch time' as the test frequency of oscillation is increased.

The before-cure S' and S'' torque responses are more sensitive to changes in frequency than the after-cure S' and S'' torque responses. This is somewhat analogous to what H. H. Winter reported for the behaviour of uncured and cured G' and G'' (before and after gel) for thermosets.¹⁴⁻¹⁶

As stated earlier, the scorch time as indicated by the S' curve did not change greatly under the conditions of this experiment (170°C and $\pm 0.5^\circ$ arc). A $\pm 0.5^\circ$ arc strain is a relatively low deformation ($\pm 7\%$). Therefore, the amount of heat energy added to the cavity is relatively small and easily dissipated with the rotorless curemeter die design. However, if a similar experiment were conducted with an oscillating disk curemeter at a $\pm 3^\circ$ arc ($\pm 42\%$ strain), the heat energy generated from the oscillating disk at increasing frequencies would effect scorch. Oscillating disk curemeters generally cannot dissipate heat as quickly as a rotorless curemeter design such as the MDR or the RPA. Also an oscillating disk curemeter has 50% more sample mass than a rotorless curemeter. In fact the annex to the ASTM standard for the oscillating disk curemeter (D 2084-92) documents the heat dissipation effect for $\pm 3^\circ$ arc and frequency variations from 10 to 900 cpm.

EFFECT OF TEMPERATURE ON $t_{\max S''}$

Another experiment was conducted with the same formulation given previously in Table 2. Cure tests were conducted with this compound using the RPA at different cure temperatures at 100 cpm and $\pm 0.5^\circ$ arc strain. Figures 8(a) and 8(b) compare the S' and S'' cure curves at 150 and 200°C, respectively. At 150°C, the $t_{\max S''}$ occurs at $t_c 12$ while at 200°C, the $t_{\max S''}$ occurs at $t_c 40$. Figure 9 shows the percent state of cure from the S' cure curve at which $t_{\max S''}$ occurs versus the cure temperature. As the cure temperature is lowered, $t_{\max S''}$ approaches the 'true scorch time'. This is similar to what

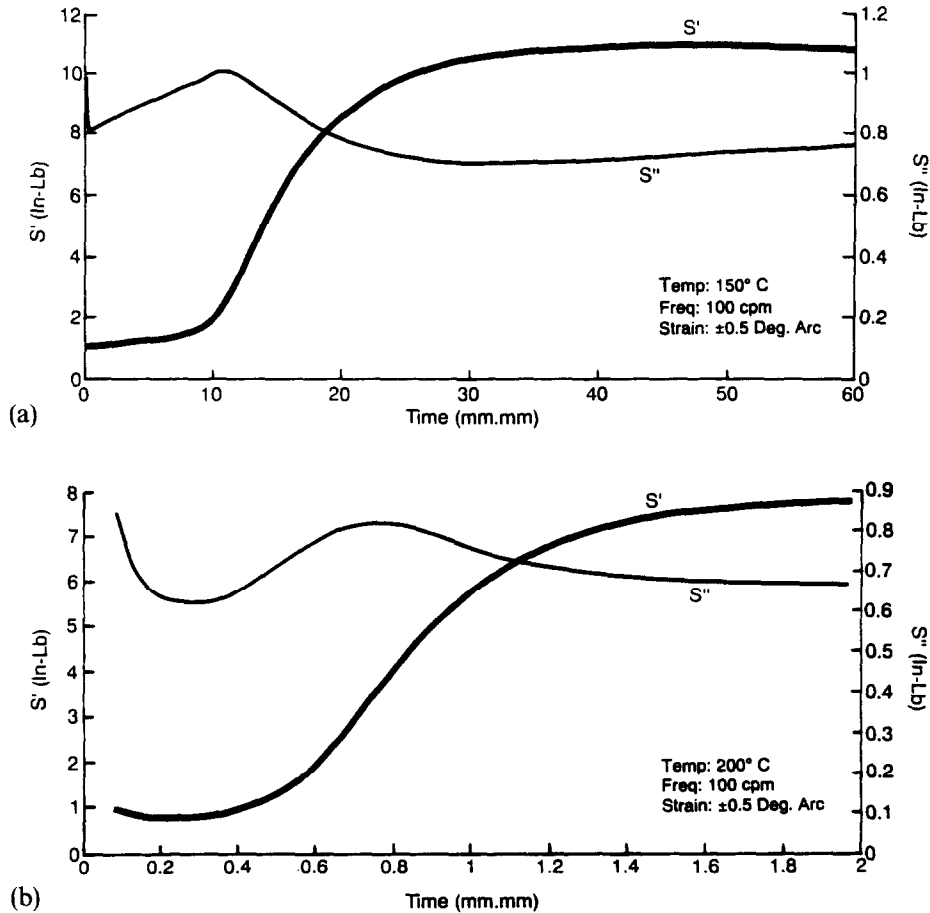


Fig. 8. Elastic (S') and viscous (S'') cure curves from testing at (a) low temperature, and (b) high temperature the model SBR compound with 50 phr N330 Carbon Black. Test conditions: ± 0.5 arcs 100 cpm and 150°C for 60 min.

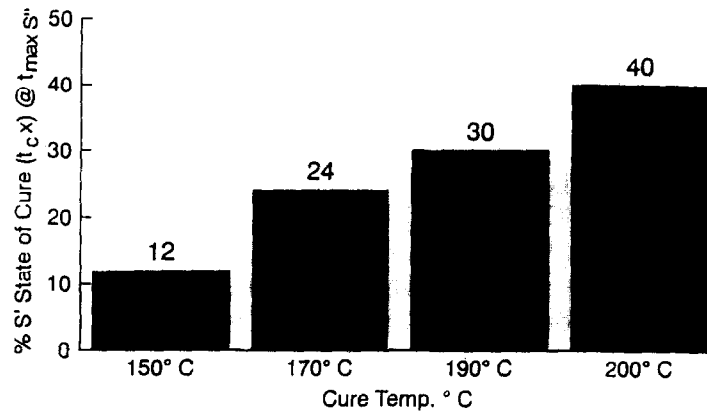


Fig. 9. Bar graph of the % state of cure from the S' curve curves at which $t_{max} S''$ occurs versus the cure temperature for the model SBR compound with 50 phr N330 Carbon Black. Test conditions: $\pm 0.5^\circ$ arc, and 100 cpm.

occurred from the effects of increasing test frequency discussed earlier. This is in agreement with the WLF shift^{17,18} which would predict that lowering temperature is equivalent to increasing frequency of oscillation.

EFFECTS OF STRAIN on $t_{max} S''$

One more experiment with the formulation given in Table 2 shows the effect of different strains on the S' and S'' cure curves. Again these tests were conducted using the RPA at different strains from $\pm 0.05^\circ$ arc ($\pm 0.7\%$ strain) to $\pm 2.0^\circ$ arc ($\pm 28\%$ strain). These cure comparisons were made at 170°C and 100 cpm. Figures 10(a) and (b) compare the S' and S'' cure curves at 0.05 and 2.0° arc strain. As noted from these cure curves, the $t_{max} S''$ remains unchanged. The $t_{max} S''$ occurred at about the same time (3.4 min) when the cure test was performed at $0.05, 0.1, 0.2, 0.5, 1.0, 2.0$, and 3.0° arc. However, the initial slope of the S' curve was affected significantly by the test strain conditions. This change in initial slope affects the calculation of t_{s1} values. The possible causes of this change in initial slope from changes in the applied strain are discussed later.

EFFECTS OF CARBON BLACK LOADING LEVELS ON THE S'' PEAK

The amount and type of Carbon Black used in a rubber compound affects the nature of the S' and S'' cure curves. An experiment used the RPA as a

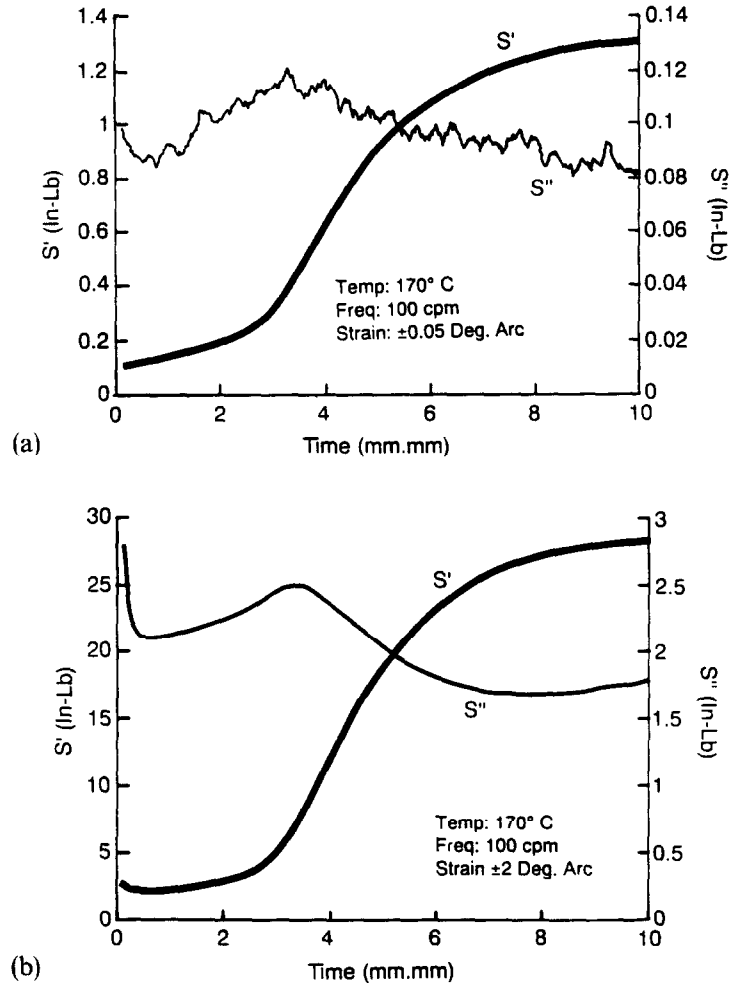


Fig. 10. Elastic (S') and viscous (S'') cure curves from testing at (a) very low strain, and (b) higher strain for the model SBR compound with 50 phr N330 Carbon Black.

rotorless curemeter at 100 cpm, 0.2° arc, and 170°C . The model SBR compound given in Table 2 was used; however the N330 Carbon Black was varied incrementally from 30 to 130 phr while keeping the aromatic oil level at 20 phr. Figure 11 gives the S' and S'' cure curves for the model SBR compound with increasing loadings of Carbon Black. As the Carbon Black concentration increases, the $t_{\max S''}$ values do not shift significantly (staying at about 3.4 min). However, the shape of the S'' peak changes greatly. At only 30 phr carbon black, the S'' peak is relatively sharp. As the concentration of Carbon Black increases, the width of the S'' peak becomes broader and broader. Moreover, the S'' cure curve on a percent basis, drops down less and less after the S'' peak with rising Carbon Black concentration. The difference

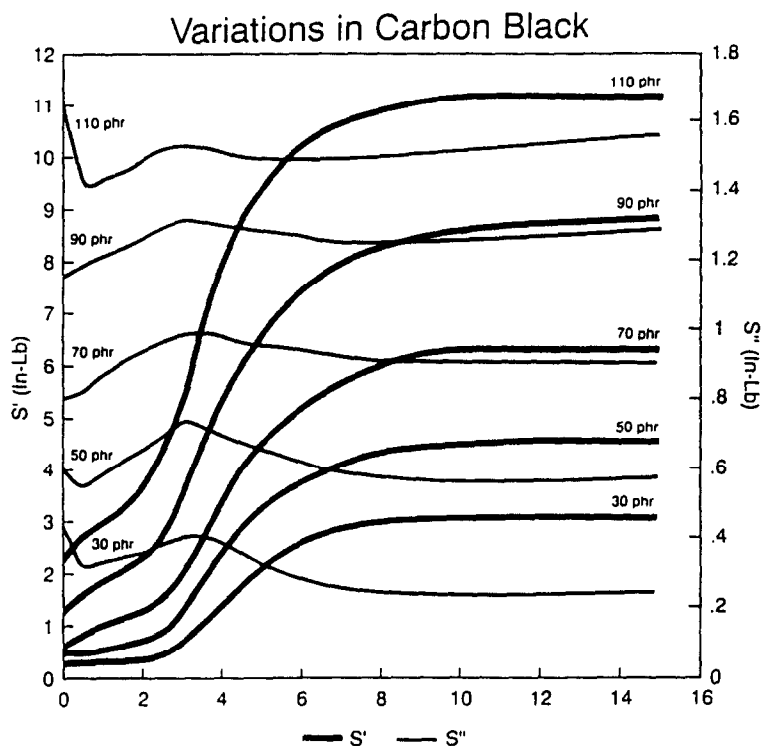


Fig. 11. Elastic (S') and viscous (S'') cure curves of model SBR compounds with 20 phr aromatic oil and variations in Carbon Black loadings as indicated. Test conditions: $\pm 0.2^\circ$ arc, 100 cpm, and 170°C .

between the peak S'' and the cured S'' at M_H decreases with increases in Carbon Black loading. Figure 12 shows the decrease in this difference as the Carbon Black loading increases. The rise in S'' at M_H is because the compound is becoming less and less resilient in the cured state from increasing Carbon Black loading.¹⁹

EFFECT OF CARBON BLACK TYPE ON THE S'' PEAK

Another experiment determined the nature of S' and S'' cure curves resulting from the use of different types of Carbon Blacks at equal loadings. Grades N121, N330 and N326 were selected to represent the 'hard' Carbon Blacks (fine particle size) while N660, N683 and N762 represent the 'soft' Carbon Blacks (large particle size). Table 3 lists surface area and structure for these Carbon Blacks. These Carbon Blacks were tested in the ASTM SBR test recipe D3191 at 50 phr each as shown in Table 4. Again the RPA was used as

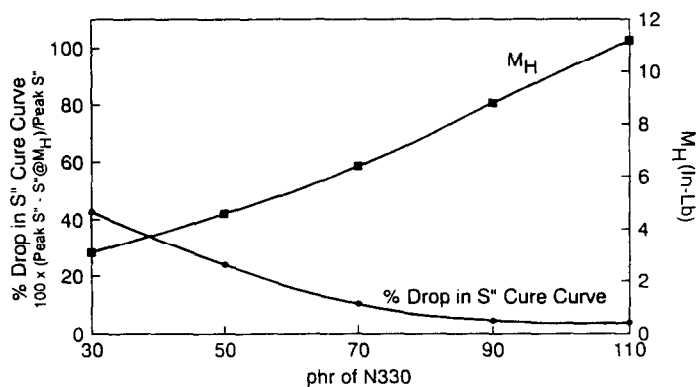


Fig. 12. Percent drop in S'' cure curve and rise in M_H with increasing loadings of N330 Carbon Black in the model SBR compound. Test conditions: $\pm 0.2^\circ$ arc, 100 cpm, and 170°C .

TABLE 3
Standard Reference Black Surface Area and Structure Comparisons

Standard reference black (ASTM D3324)	ASTM type	CTAB adsorption no. (surface area)	Compressed DBP absorption no. (structure)
A-4	N326	82.5	68.2
B-4	N330	78.7	86.6
C-4	N121	119.6	108.9
D-4	N762	24.9	58.7
E-4	N660	36.3	75.6
F-4	N683	40.5	87.8

TABLE 4
ASTM D3191

Ingredients	Parts per hundred
SBR 1500	100
Carbon Black	50
Zinc oxide	3
Stearic acid	1
TBBS	1
Sulfur	1.75
Total	156.75

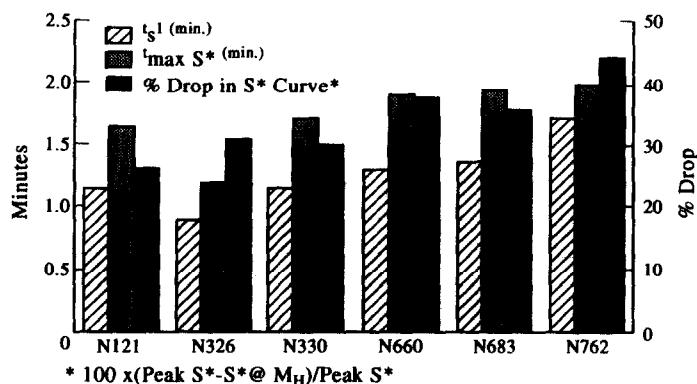


Fig. 13. Comparison of t_{s1} , $t_{max S^*}$ and % drop in the S'' cure curves for six standard SBR test compounds based on ASTM D3191 with 50 phr each of one of the six Standard Reference Blacks (ASTM D3324) as indicated. Test conditions: $\pm 0.5^\circ$ arc, 100 cpm, and 175°C .

a rotorless curemeter to cure these compounds at 100 cpm, $\pm 0.5^\circ$ arc, and 175°C . The results of this cure comparison in Fig. 13 show the scorch times were shorter for the 'hard' Carbon Blacks than those for the 'soft' Carbon Blacks. It was also observed that the difference between the peak S'' and the S'' at M_H was smaller for the 'hard' Carbon Blacks. This difference is somewhat analogous to the results of the previous experiment with variations in Carbon Black loadings.

EFFECTS OF OIL LOADING LEVELS ON THE S'' PEAK

An experiment measured the effects of increased oil loadings on the S' and S'' cure curves. The model SBR formulation (Table 2) had variations in aromatic oil loading from 0 to 80 phr with N330 Carbon Black constant at 70 phr. Again the RPA was used as a rotorless curemeter at 100 cpm, $\pm 0.2^\circ$ arc, and at 170°C cure temperature. As the oil concentration increases, the $t_{max S''}$ becomes longer and the S'' peak becomes broader and less sharp. Figure 14 compares the S' and S'' cure curves for the model SBR compound at different oil loadings.

EFFECTS OF CURATIVE VARIATIONS ON THE S'' PEAK

Curative variations also affect the S' and S'' cure curves.²⁰ The radial passenger tread formulation given in Table 5 was used to study the effects of curative variations on the S'' curve. An MDR was used for this experiment at 100 cpm, 0.5° arc and 190°C . Figure 15 shows the changes in

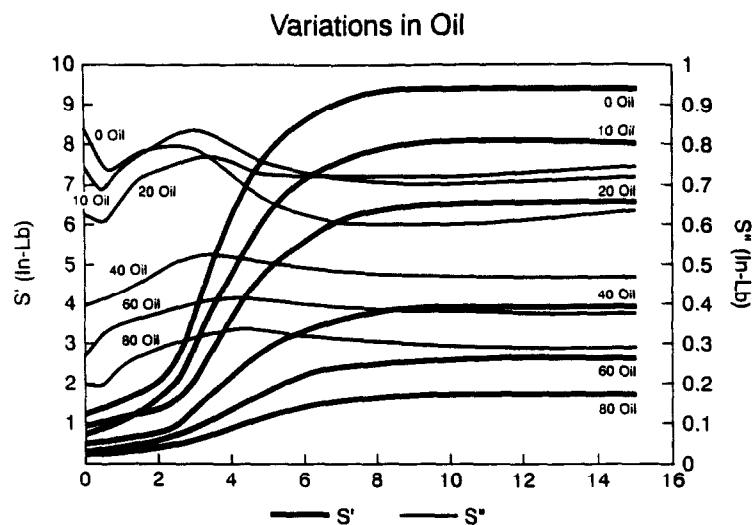


Fig. 14. Elastic (S') and viscous (S'') cure curves of model SBR compounds with 70 phr N330 Carbon Black and variations in aromatic oil loadings as indicated. Test conditions: $\pm 0.2^\circ$ arc, 100 cpm, and 170°C .

Table 5
Radial Passenger Tread Formulation

<i>Ingredients</i>	<i>Parts per hundred</i>
SBR 1502	80
Natural rubber	20
N299	50
Naphthenic oil	10
6 PPD	3
TMQ	1
Wax	2
Stearic acid	1.5
Zinc oxide	5
TBBS ^a	1.15
Sulfur ^a	2.5
Total	175.15

^a All curatives added on the mill.

the S' and S'' cure curves due to variations in accelerator levels. With a deficiency in curative (lower crosslink density), the sharpness of the S'' peak is reduced, but S'' consistently peaks at the same torque value (1.0 In-Lb) even with less curative. However, the deficiency of curative causes the S'' at M_H (the cured S'') to be relatively higher compared to results with compounds containing higher curative levels. This is because

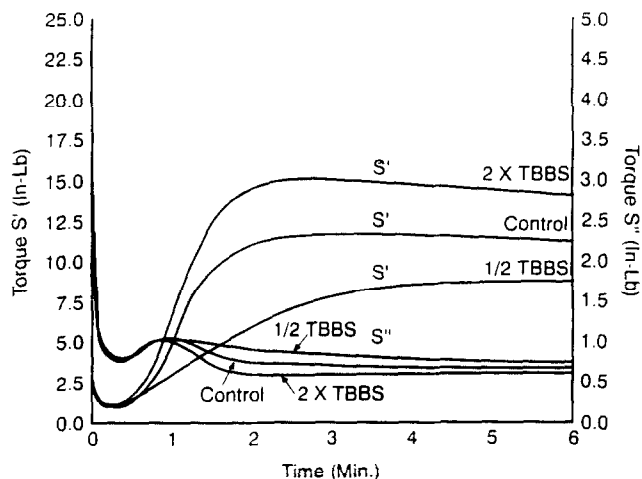


Fig. 15. Elastic (S') and viscous (S'') cure curves of radial passenger tread compounds with variations in TBBS accelerator as indicated. Test conditions: $\pm 0.5^\circ$ arc, 100 cpm, and 190°C .

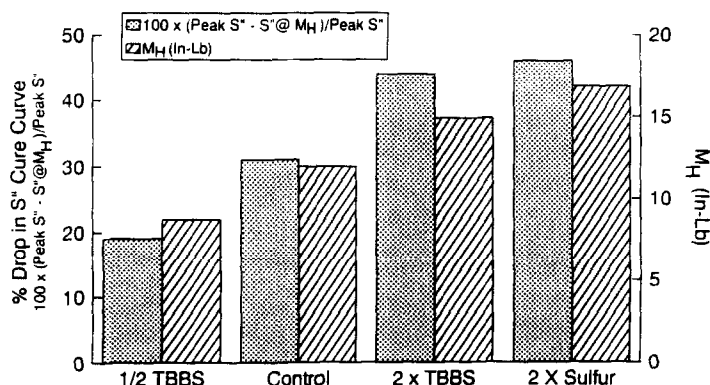


Fig. 16. Comparison of changes in percent drop in S'' cure curve and M_H from variations in TBBS accelerator and sulphur in radial passenger tread formulation as indicated. Test conditions: $\pm 0.5^\circ$ arc, 100 cpm, and 190°C .

the compounds having a deficiency of curative have a less dense sulfur crosslinked network and a higher viscous quality in the cured state. Figure 16 shows how the difference between the S'' peak and the cured S'' at M_H decreases for compounds as curative concentrations are reduced. A similar relationship was noted earlier with increasing levels of Carbon Black. However, with the Carbon Black effect, the cured S' and cured S'' moved in the same direction (not opposite directions) and the S'' peak did not remain the same height.

The S'' peak also exists with other cure systems as well. It occurs with conventional, semi efficient and efficient vulcanization cures as well as peroxide and phenolic resin cures.

As will be discussed later, the addition of *N*-(cyclohexylthio) phthalimide to a compound based on a sulfenamide cure causes the $t_{\max S'}$ as well as the t_{s1} , t_{c10} and t_i to increase in length.

VISCOELASTIC CHANGES BEFORE SCORCH

When measuring scorch time with a Mooney viscometer, the viscosity usually decreases from an initial peak, 'levels off' and then starts to rise when it approaches the scorch time. By contrast, the oscillating disk curemeter may display a gradual rise after allowing for the short time necessary for the sample to reach the set temperature. The contrast of Mooney scorch versus the oscillating disk curemeter test was reported by Decker *et al.* and is shown in Fig. 17. Rotorless curemeters are even more sensitive to viscoelastic changes which occur before the scorch time. This gradual rise in the S' torque before scorch will effect t_{s1} and t_{s2} scorch times since they are defined as the time required to achieve a one unit or two unit rise above a minimum. The gradual rise of S' is partially attributable to a small number of crosslinks which form very early before scorch.²² However, other causes may also be partially responsible for this upward drift in the S' before scorch. These causes are discussed below.

CARBON BLACK EFFECTS ON INITIAL S' SLOPE BEFORE SCORCH

A comparison of Fig. 10(a) with 10(b) shows the S' initial slope before

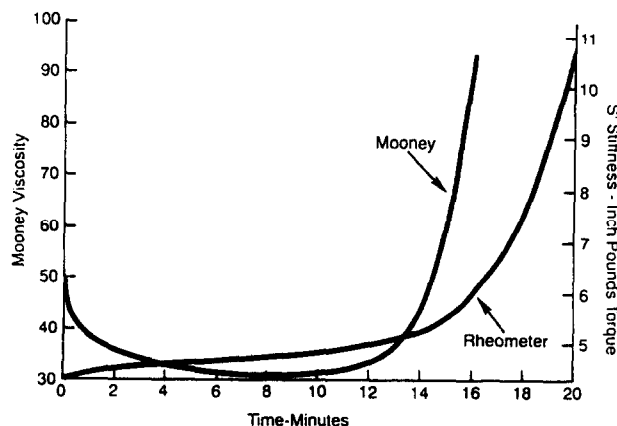


Fig. 17. Comparison of oscillating disk curemeter S' cure curve and Mooney viscosity scorch curve as reported in Ref. 21.

scorch increasing more with a lower applied strain (see 'Effects of Strain on $t_{\max S''}$ '). Carbon Black when mixed in a rubber formulation forms an aggregate–aggregate network which breaks up when disturbed by shearing forces, but the network reforms after sufficient relaxation time.^{23–25} The reformation of this network after prior shearing of a compound in the uncured state has been measured with the RPA as an increase in S' with time at low strain (a time sweep). This technique was developed by Coran²⁶ and later by Burhin.²⁷

Another experiment determined if the destruction and reformation of

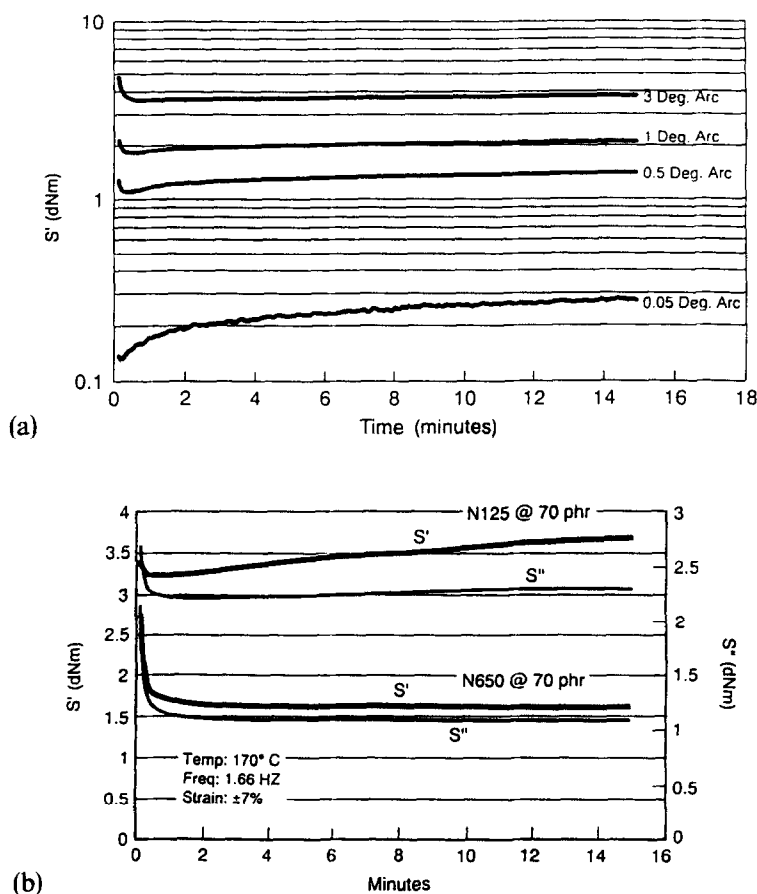


Fig. 18. (a) Comparison of elastic torque response (S') just after closing the RPA dies with different strain amplitudes as indicated above for the model SBR compound with 50 phr N330 Carbon Black and no curatives. (b) Comparison of elastic torque response (S') just after closing the RPA dies for the model SBR compound with N125 (small particle size Carbon Black) versus N650 (large particle size Carbon Black) each at 70 phr with no curatives. Test conditions: 100 cpm and 170°C.

the Carbon Black aggregate–aggregate network might contribute to an increase in the angle of the initial slope for S' before scorch (as shown in Fig. 10). The formulation used for this study is the same model SBR compound used earlier (Table 2), but without any curatives (no sulfur or accelerator). Again the RPA was used as a rotorless curemeter under the same 'cure' conditions of 100 cpm and 170°C. Repeat tests were performed on different samples of the same mixed stock at progressively higher strains. Since no curatives were present in this compound, any rise in the S' torque cannot be caused by vulcanisation.

Figure 18(a) shows the RPA S' responses at different strain amplitudes for the model SBR compound without curatives. The S' does rise significantly at very low strains. As the strain amplitude increases, however, the percent rise in S' decreases. The rise in S' is possibly due to the reformation of the Carbon Black aggregate network. As the amplitude of strain is increased, there is less chance for this Carbon Black network to reform. Further experiments have shown that this 'Carbon Black' effect decreases with an increase in frequency of oscillation at $\pm 0.5^\circ$ arc strain or a decrease in temperature at $\pm 0.5^\circ$ arc. Also N125 (fine particle size Carbon Black) was compared with N650 (large particle size Carbon Black) both at 70 phr in the SBR model compound. Figure 18(b) shows that the N650 'soft' Carbon Black does not increase S' with time under the same test conditions used before. This may be because the large particle size Carbon Blacks do not form this aggregate–aggregate network in the same manner as the 'hard' Carbon Black grades.

However, this 'Carbon Black' effect is not great enough to fully account for the initial S' slope for the SBR compound with curatives. Much of the initial rise in S' is probably caused by chemical crosslinking.

COMPARISON OF SCORCH INDICES

A series of experiments were performed to compare the following scorch indices:

- t_{s1} —curemeter scorch time (time for a one unit rise above the minimum elastic torque (M_L))
- t_{c10} —curemeter time to 10% state of cure
- t_i —curemeter reaction kinetics incubation time
- $t_{\max S''}$ —curemeter time to peak viscous torque S''
- $t_{\min S'}$ —curemeter time to minimum S' torque

SENSITIVITY TO VARIATIONS IN CTP

An experiment was performed to measure the test sensitivity of each scorch index listed above to changes in CTP (*N*-(cyclohexylthio) phthalimide) concentrations in selected compounds. CTP is a common scorch inhibitor which should affect scorch indices. Four different formulations were selected for this study. Two of the formulations (Tables 6 and 7) were based on SBR with low and high loadings respectively of N330 Carbon Black while the remaining two formulations (Tables 8 and 9) were based on natural rubber with low and high loadings respectively of N220

TABLE 6
SBR Formulation with Low
Carbon Black Loading

<i>Ingredients</i>	<i>Parts per hundred</i>
SBR 1500	100
N330	10
Zinc oxide	3
Stearic acid	1.5
TMQ	1.0
CBTS ^a	1.25
Sulfur ^a	2.0
CTP ^a	0-0.5
Total	118.75-119.25

^aAll curatives added on the mill.

TABLE 7
SBR Formulation with High
Carbon Black Loading

<i>Ingredients</i>	<i>Parts per hundred</i>
SBR 1500	100
N330	70
Aromatic oil	20
Zinc oxide	3.0
Stearic acid	1.5
TMQ	1.0
CBTS ^a	1.25
Sulfur ^a	2.0
CTP ^a	0-0.5
Total	198.75-199.25

^a All curatives added on the mill.

TABLE 8
Natural Rubber Formulation with
Low Carbon Black Loading

<i>Ingredients</i>	<i>Parts per hundred</i>
SMR 10, natural rubber	100
N220	10
Zinc oxide	5
Stearic acid	2.5
TMQ	2.0
6 PPD	2
Sulfur ^a	1.5
MBS ^a	1.6
CTP ^a	0-0.5
Total	124.6-125.1

^a All curatives added on the mill.

TABLE 9
Natural Rubber Formulation with
High Carbon Black Loading

<i>Ingredients</i>	<i>Parts per hundred</i>
SMR 10 natural rubber	100
N220	60
Aromatic oil	15
Zinc oxide	5
TMQ	2
6PPD	2
Stearic acid	2.5
Sulfur ^a	1.5
MBS ^a	1.6
CTP ^a	0-0.5
Total	189.6-190.1

^a All curatives added on the mill.

Carbon Black. As reported earlier, there were some problems noted when $t_{\max S}$ was used to measure scorch times with natural rubber compounds. These natural rubber formulations will determine how well $t_{\max S}$ can detect changes in scorch characteristics for these 'more difficult' compounds.

Master batches of these four formulations were prepared using a laboratory BR Banbury. The four master batches were then subdivided into five weighed portions each. The curatives were added to each of these portions on the mill along with incremental additions of CTP at 0.0, 0.05,

0.1, 0.25, and 0.5 phr for each of the four series of mixes. An MDR was used at 100 cpm, 0.5° arc, and 170°C to test the scorch properties for each of the final mixes. The scorch properties recorded by the MDR were t_{s1} , t_{c10} , t_i , $t_{\max S^*}$ and $t_{\min S^*}$. Each series of tests was repeated four times in order to measure repeatability and relative sensitivity.

'Sensitivity' was calculated by a method analogous to signal-to-noise ratios used in the electronics industry. The 'signal' in this calculation is the difference between the maximum and minimum of the test measurement averages within a group of compound variations. The 'noise' is the pooled standard deviation (SD_p) of the test replicates for each compound. The formula for this calculation is given in eqn (1):²⁸

$$\text{'Sensitivity'} = (\max \bar{X} - \min \bar{X}) / SD_p \quad (1)$$

A high 'sensitivity' value indicates the curemeter scorch parameter is more sensitive to compound scorch changes than a scorch parameter with a low 'sensitivity' value. A curemeter scorch parameter that produces a greater 'signal' from a compounding change and/or has reduced 'noise' due to random test variation will have increased sensitivity to the compound variation. The calculated sensitivity values were all normalized with t_{s1} being assigned 100.

Sensitivity to CTP Variations in SBR—Fig. 19 compares the test sensitivity for each of the scorch indices for both low and high Carbon Black loaded SBR formulations. The t_{s1} index gave the best (highest) sensitivity of all the scorch indices studied in both SBR formulations. t_i consistently gave the second best sensitivity to scorch differences while

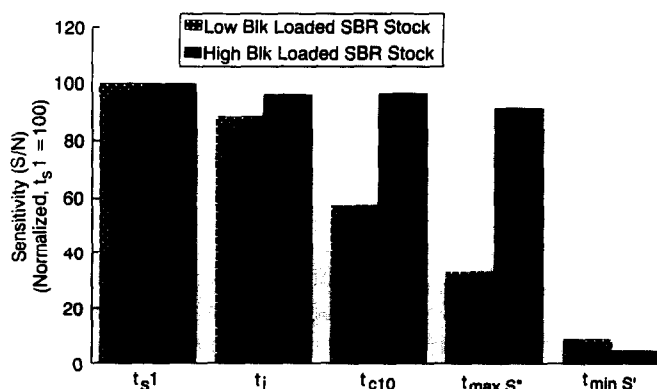


Fig. 19. Comparison of test sensitivity of the five different scorch indices to given CTP variations for both the low and high carbon black loaded SBR test compounds. All sensitivity (signal-to-noise ratio) values have been normalized with t_{s1} being arbitrarily assigned a value of 100. Test conditions $\pm 0.5^\circ$ arc, 100 cpm, and 170°C .

t_{C10} was consistently third. The $t_{\max S^*}$ gave very good sensitivity to scorch variations in the highly loaded SBR compound but was less sensitive to scorch changes in the SBR compound with low loading of Carbon Black. Also the time to minimum torque ($t_{\min S^*}$) was studied. This parameter was found to have no sensitivity to changes in scorch for the SBR compounds.

Figure 20 compares the time ranking of the scorch indices for compound number 1 in Table 6 (SBR with low Carbon Black loading) versus compound number 1 in Table 7 (SBR with high Carbon Black loading). All index times given in Fig. 20 are arranged in ascending order. As can be seen, the relative position of t_{S1} changes compared to the other indices. For the compound with low Carbon Black loading (and lower M_H torque), the t_{S1} lags behind the other indices. However, for the compound with high Carbon Black loading, the t_{S1} occurs much earlier. This is an example where t_{C10} may be the preferred scorch index when comparing compounds with significantly different maximum torque values (M_H).

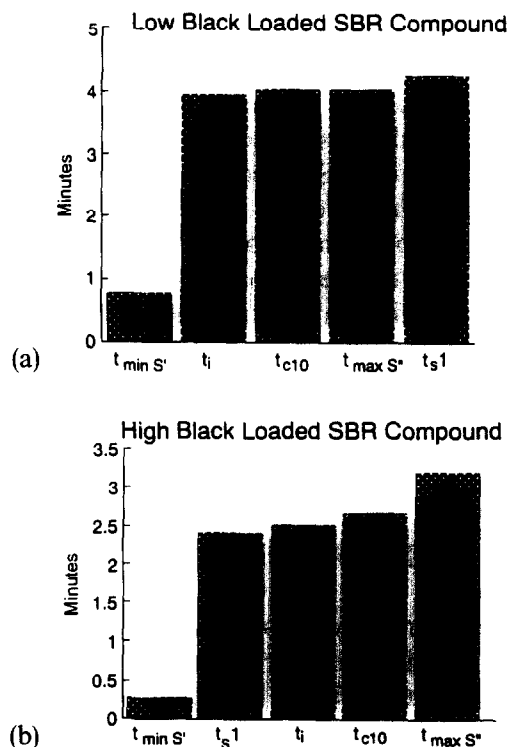


Fig. 20. Comparison of the five scorch indices in ascending order of total lapse time in minutes for (a) the low and (b) the high Carbon Black loaded SBR test compound without CTP. Test conditions: $\pm 0.5^\circ$ arc, 100 cpm, and 170°C .

Figures 21(a) and (b) show the correlations between t_{s1} and $t_{\max S''}$, respectively, for the low and high loaded SBR formulations. There is some scatter for the SBR compound with low loading of Carbon Black. However, the correlation is nearly perfect for the highly loaded SBR compound.

Tables 10 and 11 give the matrix of correlation coefficients for all possible one-on-one comparisons of the scorch indices for the low load SBR compound and the high load SBR compound.

Sensitivity to CTP Variations in NR—Fig. 22 compares the test sensitivity for the scorch indices for both the low and high Carbon Black loaded NR formulations. The t_{s1} , t_i , and t_{C10} indices all had very good sensitivity to scorch changes. However, the $t_{\max S''}$ was not as sensitive to changes in scorch for these NR compounds. No $t_{\max S''}$ actually occurred for the low Carbon Black loaded NR stock. Just a 'bulge' appeared where the S'' peak would be expected to appear. With the higher loading of Carbon Black, the S'' peak was observed, but it was not well enough defined to enable scorch time values to be measured accurately.

Figure 23 compares the time ranking of the scorch indices for compound number 1 in Table 8 (NR with low Carbon Black loading) versus compound number 1 in Table 9 (NR with high Carbon Black loading). All index times given in Fig. 23 are arranged in ascending order. Again the relative position of these indices is in the same order as noted in the earlier SBR experiment. Similar to the SBR compounds, the relative position of t_{s1} changes compared with the other indices when going from the low Carbon Black loading to the high Carbon Black loading NR compounds. Again t_{C10} may be the preferred scorch index when comparing compounds with significantly different maximum S' torque values (M_H).

Tables 12 and 13 give the correlation coefficient matrix for all possible comparisons of the scorch indices for the low and high loaded NR compounds, respectively.

COMPARISON OF S'' PEAK IN A VARIED SELECTION OF RUBBER COMPOUNDS

The purpose of this experiment was to determine how the nature of the S'' peak changes when going from one production compound to the next. To make this a 'real world' comparison, 14 different generic tire compounds²⁹ and 26 general products compounds³⁰⁻⁴⁵ were randomly selected from the literature. Tables 14 and 15 give a general description for each of these compounds as well as noting the nature of the measured S'' peak. A MDR at 100 cpm, $\pm 0.5^\circ$ arc, and 170°C was used to measure the S'' peak.

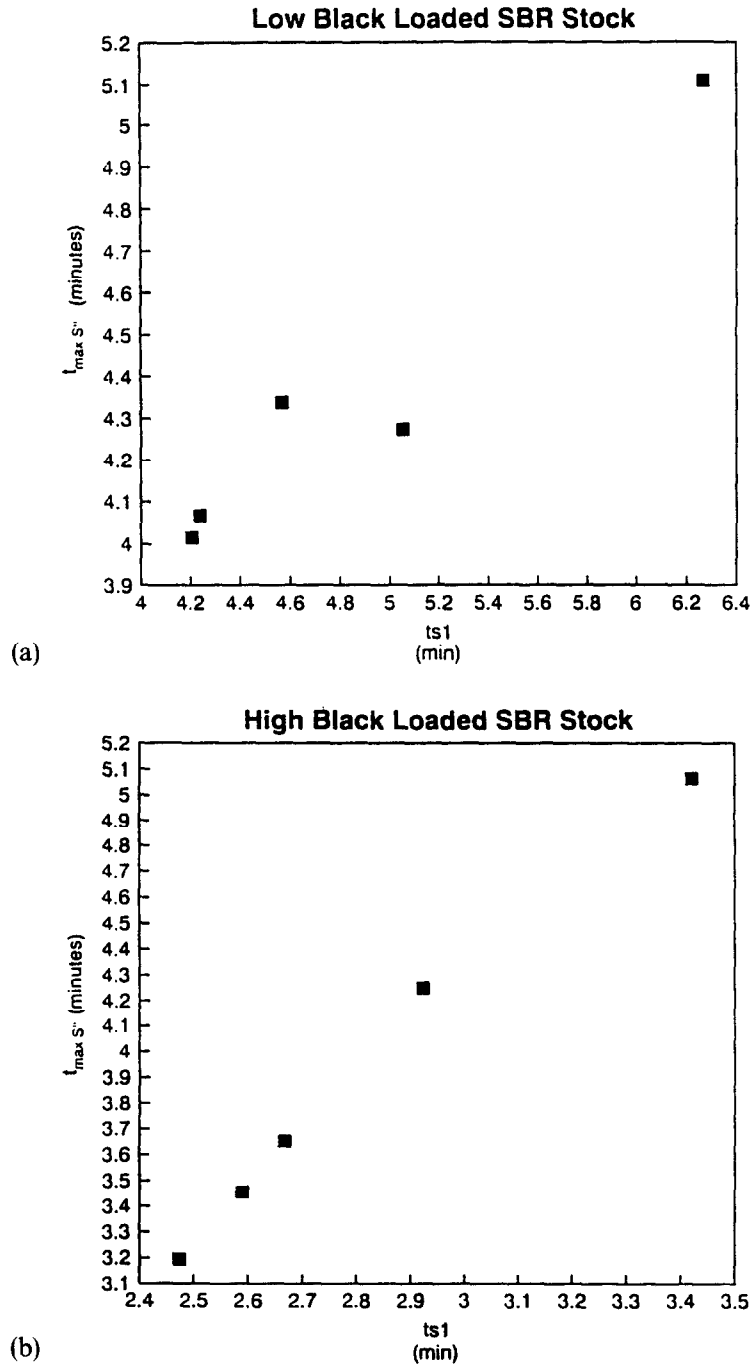


Fig. 21. Scatter plot of $t_{max S''}$ versus t_{s1} for CTP variations in (a) the low loaded and (b) the high loaded Carbon Black SBR test compound. Test conditions: $\pm 0.5^\circ$ arc, 100 cpm, and 170°C .

TABLE 10
Correlation Matrix of Scorch Indices for Low Black Loaded SBR Compound

	$t_s 1$	$t_c 10$	t_i	$t_{\max} S''$	$t_{\min} S'$
$t_s 1$	1				
$t_c 10$	0.994	1			
t_i	0.999	0.999	1		
$t_{\max} S''$	0.971	0.984	0.977	1	
$t_{\min} S'$	0.303	0.294	0.311	0.267	1

TABLE 11
Correlation Matrix of Scorch Indices for High Black Loaded SBR Compound

	$t_s 1$	$t_c 10$	t_i	$t_{\max} S''$	$t_{\min} S'$
$t_s 1$	1				
$t_c 10$	0.999	1			
t_i	0.999	0.999	1		
$t_{\max} S''$	0.997	0.998	0.999	1	
$t_{\min} S'$	-0.63	-0.62	-0.63	-0.61	1

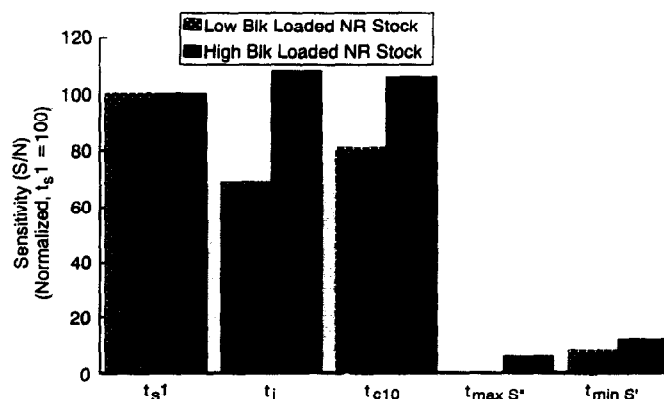


Fig. 22. Comparison of test sensitivity of five different scorch indices to given CTP variations for both the low and high carbon black loaded natural rubber test compounds. All sensitivity (signal-to-noise ratios) values have been normalized with $t_s 1$ being arbitrarily assigned a value of 100. Test conditions: $\pm 0.5^\circ$ arc, 100 cpm, and 170°C .

All the tire formulations, with the exception of the apex compound, displayed an S'' peak. The Apex compound may not have had an S'' peak because it contained 12 phr of a reactive phenolic reinforcing resin. By contrast, six of the 26 general product compounds were found to either not display an S'' peak during cure or only show a 'bulge' where the S'' peak should have appeared. In almost every case, these compounds were either

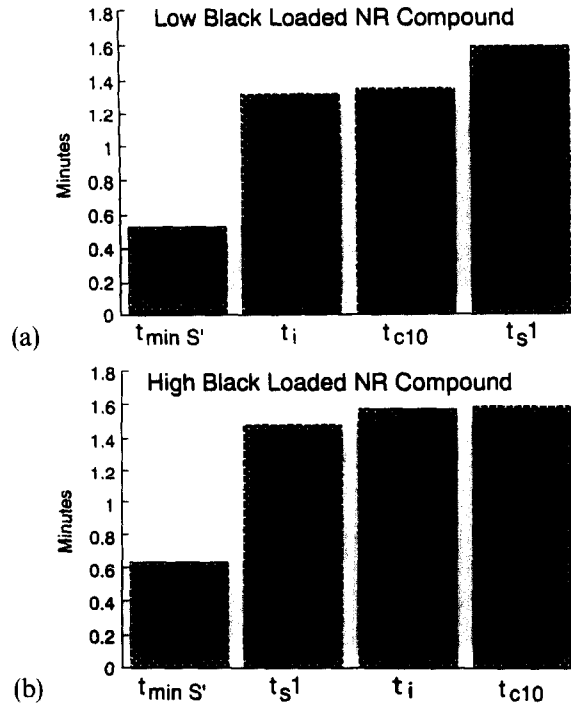


Fig. 23. Comparison of the five scorch indices in ascending order of total lapse time in minutes for (a) the low and (b) the high Carbon Black loaded natural rubber test compound without CTP. Test conditions: $\pm 0.5^\circ$ arc, 100 cpm, and 170°C .

TABLE 12
Correlation Matrix of Scorch Indices for Low Black Loaded Natural Rubber Compound

	t_{s1}	t_{c10}	t_i	$t_{max S''}$	$t_{min S'}$
t_{s1}	1				
t_{c10}	0.999	1			
t_i	0.999	0.999	1		
$t_{max S''}$	—	—	—	1	
$t_{min S'}$	0.964	0.966	0.968	—	1

TABLE 13
Correlation Matrix of Scorch Indices for High Black Loaded Natural Rubber Compound

	t_{s1}	t_{c10}	t_i	$t_{max S''}$	$t_{min S'}$
t_{s1}	1				
t_{c10}	0.999	1			
t_i	0.999	0.999	1		
$t_{max S''}$	0.824	0.818	0.811	1	
$t_{min S'}$	0.325	0.310	0.302	0.514	1

TABLE 14
Tire Compounds

Compound	Total Parts per hundred	Rubber base	Parts per hundred filler	Parts per hundred oil	S" peak	S" peak shape	@ % S' State of cure	% Drop $100 \times \frac{(\max S'' - S'' @ M_H)}{\max S''}$	Comments
Black sidewall	164	NR 65 BR 35	N330 20 N550 20	4	Yes	Not sharp	5	46	
Steel tire cord skim	189	NR	N326 45 Silica 15	0	Yes	Not sharp	12	—	RF resin HMMM & Co. Naph.
Bead insulation	235	SBR	N660 100	20	Yes	Fairly sharp	15	35	
Apex compound	205	NR 60 IR 20 SBR 20	N326 65	12	None	—	—	—	Reinforcing resin 12 parts per hundred, marching modulus
Carcass stock	179	NR 50 SBR 30 BR 20	N660 50	18	Yes	Sharp	14	56	

Inner-liner	241	CIIR 65 NR 35	N660 70 Whiting 50	12	Yes	Extremely broad	35	7	
Truck undertread	175	NR 80 BR 20	N550 50	12	Yes	Moderately sharp	5	56	
White sidewall	175	NR 30 EPDM 10 CIIR 60	Clay 40 TiO ₂ 25	0	Yes	Very broad	14	26	
Truck tread	172	NR 60 BR 40	N330 50	10	Yes	Moderately broad	5	28	
OTR tread	184	NR	N220 40 Silica 20	3	Yes	Not well defined	5	9	Coupling agent
Performance tread	167	SBR	N330 70	37	Yes	Very broad	5	36	
Passenger tread	220	SBR 70 BR 30	N339 70	14	Yes	Very broad	12	12	
ASTM tread	200	SBR 75 BR 25	N351 64	20	Yes	Moderately sharp	20	23	
Innertube	205	11R	N660 70	25	Yes	Moderately sharp	6	28	

Table 15
General Product Compounds

Compound ID (Ref.)	Total Parts per hundred	Rubber base	Parts per hundred filler	Parts per hundred oil	S'' peak	S'' peak shape	@% S' state of cure	% Drop $100 \times (\max S'' - S'' @ M_H / \max S'')$	Comments
Highly extended low cost EPDM compound ³⁰	819	EPDM	N650 250 N774 100 Whiting 200	165	None	—	—	—	MBTS/ TMTD/ ZnDBC/ Sulfur
Rubber bands ³¹	115	NR	TiO ₂ 1.5 Whiting 2	1	Not well defined	A 'Bulge' no actual peak	—	—	MBTS/ TMTD DTDM/ Sulfur
Printing rubber, 45 duro ³²	191	NR	Barytes 40 Clay 15	12	Yes	Sharp	10	80	CBTS/ Sulfur and ZnDBC Kicker
High damping, low creep NR compound No NBR ³³	132	NR	N762 25	0	Yes	Weak, not sharp	3	75	
High damping low creep NR compound with 25 PHR NBR ³³	127	NR 75 NBR 25	N762 20	0	Yes	Sharp	10	62	

High damping low creep NR/NBR ³³	127	NR 50 NBR 50	N762 20	0	Yes	Sharp	32	34	
Idler roll compound ³⁴	189	NBR	N330 60	6	Yes	Sharp	30	10	
Fluoro-E lastomer check out stock	124	FKM	N550 10 N326 5	0	Yes	Broad	65	4	Dihydroxy crosslinker
Pharmaceutical closure ³⁵	207	CIIR	Whiting 100	0	Yes	Moderate	15	53	TMTD
Hot water bottles ³⁶	200	NR	Whiting 90	0	Yes	Moderate	5	65	TMTD/ MBTS/ DTDM Cure
Acid hose tube ³⁵	175	11R	N550 35 N770 25	0	Yes	Not well defined	5	44	MBT/ TMTD/ TeDEC
Radiator hose ³⁷	513	EPDM	N650 130 N762 95 Whiting 40	130	Not well defined	'Bulges' not a peak	—	—	TMTD/ DTDM/ ZnDBC/ ZnDMC/ Sulfur

Table 15—Contd.

Compound ID (Ref.)	Total Parts per hundred	Rubber base	Parts per hundred filler	Parts per hundred oil	S" peak shape	S" peak shape	@% S' state of cure	% Drop $100 \times (\max S'' - S'' @ M_H / \max S'')$	Comments
Heat-resistant conveyor belt cover ³⁸	165	CIIR	N330 50	5	Not well defined	A small bulge	—	—	MBTS/TMTD cure
V-belt friction oil-resistant jacketed ³⁵	163	CR	N550 30	20	Yes	Sharp	40	38	ZnO and MgO No accelerator
Hose tube compound ³⁹	280	NBR	Talc 40 Silica 90 N990 5	20	Yes	Moderate	38	—	CBS/DIDM/TMTD Marching modulus
Oil field compound ³⁵	216	NBR	N231 35 N774 50 Silica 15	0	Yes	Sharp	20	33	MBTS/TMTD/Sulfur cure
Red sheet packing ⁴⁰	640	SBR	Clay 240 Whiting 250 Iron oxide 10	25	No	—	—	—	CBS/DOTG/Sulfur
SBR gum compound	113	SBR	0	0	Yes	Broad	5	63	CBTS and Sulfur
NR gum compound	114	NR	0	0	Not well defined	'Bulges' not a peak	—	—	CBTS and Sulfur

Hydraulic hose cover ⁴¹	266	CR	N762 55 N990 65	28	Yes	Moderately sharp	12	28	Cured with DPG, TMTM, ZnO, Sulfur
Black EPDM roofing ⁴²	311	EPDM	N660 85 Clay 100	95	Yes	Sharp	20	18	MBT/TMTM/Sulfur, cure
Peroxide cured SBR compound	167	SBR	N330 52	10	Yes	Broad	12	42	Iupercu 130XL at 4 pts
Resin cured SBR compound	182	SBR	N330 52	10	Yes	Extremely broad	30	18	SP 1045 at 10 pts Neoprene at 5 pts ZnO 5 pts
Eraser ⁴³	612	NR	MgO 30 Lithopone 100 BaSO ₄ 80 Whiting 150 TiO ₂ 10 Factice 100	30	None	—	—	—	"Marching Modulus" Followed by Reversion
Leather innersole footwear ⁴⁴	229	SBR	Clay 115 TiO ₂ 10	0	Yes	Sharp	10	16	
Translucent soling ⁴⁵	208	SBR	SiO ₂ 65	12	Yes	Sharp	15	20	

(i) very highly loaded compounds in excess of 500 total phr (such as the red sheet packing, the 'low cost' EPDM sheet stock or the highly loaded EPDM radiator hose), or (ii) natural rubber compounds with very low filler loadings (such as rubber band stock or NR gum stock). It is interesting to note that when the 'high damping, low creep NR compound' is based on 100 phr natural rubber, the S'' peak is 'weak'. However, when 25 and then 50 phr of NBR is partially substituted for the NR in to this compound, the S'' peak becomes very sharply defined. The rest of this very diverse selection of compounds gave S'' peaks during cure. These included compounds based on non-sulfur cure systems (peroxide and resin cures) and speciality rubbers such as fluoroelastomers.

The tables also give a general description of the shape of the S'' peak, the % S' state of cure at which the S'' peak occurs, and the % S'' drop from the S'' peak ($S''_{\max}$) to the cured $S''@M_H$ as calculated from eqn (2) below:

$$\% S'' \text{ drop} = 100 \times (S''_{\max} - S''@M_H) / S''_{\max} \quad (2)$$

Directionally, the higher the total phr loading of a compound, the broader the S'' peak and the smaller the % drop in S'' . Also the base elastomer has a large effect on the % S'' drop with higher T_g (less resilient) elastomers such as NBR, fluoroelastomers and butyl giving a lower % drop in S'' after the peak. Again, compounds based on less resilient elastomers (having a higher glass transition temperature T_g) also have their peak S'' occurring at a higher percent state of cure as measured by S' than the more resilient rubber based compounds. Figure 24 illustrates how the

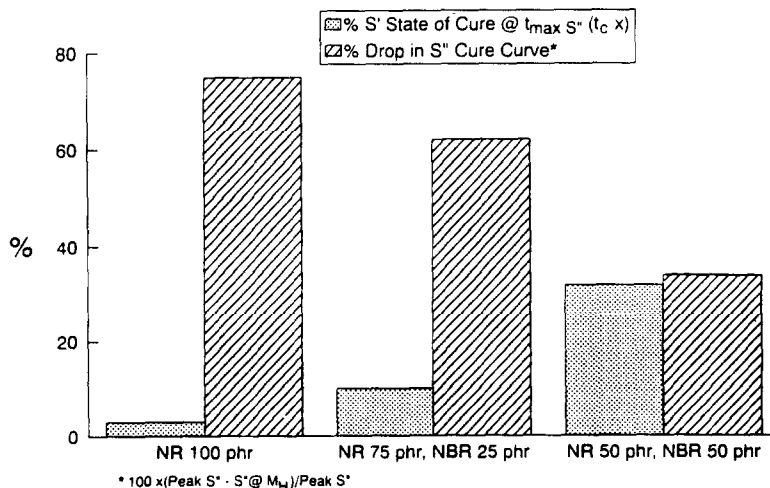


Fig. 24. Bar chart comparison of changes in percent drop in S'' cure curve and percent S' state of cure @ $t_{\max} S'$ from changes in the NBR content in the high damping, low creep compound. Test conditions: 0.5° arc, 100 cpm, 170°C.

TABLE 16
High Damping, Low Creep Compound

<i>Ingredients</i>	<i>Parts per hundred, rubber compound number</i>		
	<i>1</i>	<i>2</i>	<i>3</i>
Natural rubber	100	75	50
NBR (33% ACN)	0	25	50
N762 Carbon Black	25	20	20
Zinc oxide	5	5	5
TMQ	2	2	2
Sulfur ^a	0.6	0.6	0.6
MBS ^a	1.4	1.4	1.4
TMTD ^a	0.6	0.6	0.6
Total	134.6	129.6	129.6

^a Curatives added on the mill.

% S'' drop decreases and the percent state of cure at the occurrence of the S'' peak increases as the concentration of NBR is increased and the NR is decreased for the compound given in Table 16. The T_g of the base rubber appears to have a great effect on the nature of the S'' cure curve.

CONCLUSIONS

The $t_{\max S''}$ can be used as a valid alternative test parameter for measuring scorch time for many rubber compounds.

The $t_{\max S''}$ approaches the 'true' scorch time when measured with a higher curemeter oscillation frequency or a lower temperature. Also $t_{\max S''}$ occurs closer to the 'true' scorch time for compounds based on elastomers with low T_g values such as natural rubber as opposed to elastomers with high T_g values such as NBR or FKM.

Increasing filler loadings cause the % drop in the S'' cure curve after cure to decrease and the shape of the S'' peak to broaden (become less sharp). As curative concentration increases in a rubber compound, the % drop in the S'' cure curve increases.

The initial slope (before scorch) of the S' cure curve is affected by both vulcanization chemistry before scorch and possibly the physical reformation of a Carbon Black aggregate-aggregate network shortly after closing the die cavity.

Only two types of rubber compounds did not show an S'' peak. These two types of compounds were as follows:

- (a) rubber compounds which contain extremely high loadings of fillers (total phr in excess of 500); and
- (b) compounds based solely on natural rubber and with very low loadings of fillers (such as NR gum stock).

The t_{C10} may be preferred over the t_{s1} when comparing scorch characteristics of compounds which differ greatly in their maximum S' torque (M_H) values.

This study has also shown that the S'' cure curve of the RPA 2000TM analyzer or the MDR 2000[®] curemeter contains useful information on the base polymer, filler, and cure systems of the compound.

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