

Effect of γ -Radiation on the Dynamic Mechanical Properties of Styrene-Butadiene Rubbers

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Synopsis

Three different styrene-butadiene, gum rubbers were exposed to γ -radiation doses ranging from 0 to 200 Mrad. The crosslink densities, hardness, and the dynamic mechanical properties were evaluated. All specimens increased in hardness and crosslink density during radiation. This was reflected in an increased storage modulus, broadened loss tangent maximum, and increased relaxation spectrum. The dynamic data are presented in master curves which were developed by using the WLF equation.

Styrene-butadiene rubber (SBR) is considered to be one of the more radiation-resistant synthetic rubbers. This resistance is attributed to the "protective" influence of the aromatic styrene group which absorbs the energy. Examples of this protection can be found in the books by Chapiro,¹ Bolt and Carroll,² and Charlesby.³ Witt⁴ found that the G value for crosslinking (moles of crosslink formed per 100 e.v. of absorbed energy) was decreased by adding styrene to styrene-butadiene elastomers. Charlesby⁵ determined that the aromatic group protected four carbon units on either side of the aromatic structure.

However, many unexplained anomalies exist, and the actual mechanisms of radiation interaction are not understood. For example, natural rubber, which contains no aromatic structure, is less sensitive to radiation than SBR.⁶ Polybutadiene is affected more by high levels of radiation than SBR, yet the G value for gas formation has been reported as 0.23 for polybutadiene compared with 0.42 for a SBR of 10% styrene content.¹ Also, the G value for crosslinking of polybutadiene was found to be the same as that of a 70/30 copolymer of butadiene and styrene.¹ The data of Bauman and Glantz⁶ are frequently cited to illustrate the increased radiation protection of the styrene group. Their data⁶ show a reduction in solvent swelling with increasing styrene content at a given dose level. However, the change in swelling with change in irradiation level is the same for the SBR containing 50% styrene as that containing 10% styrene. The concept of protection by aromatic groups has been questioned in other work.⁷

Dynamic mechanical test methods offer an opportunity to clarify the reaction routes, since the dynamic data give information about both the molecular and the physical property characteristics of polymers. Some dynamic testing has been done on irradiated polymers.⁸ However, the past tests were usually carried out at constant frequencies over wide temperature ranges. More meaningful data can be obtained by measuring the dynamic properties over wide frequency ranges at several temperatures. The latter is experimentally difficult at the present time but was accomplished to a limited extent in this research.

In addition to the dynamic testing, the hardness, swelling characteristics, and the sol contents were determined for the irradiated rubbers. The relationship between crosslink density, calculated from swelling data, and the dynamic mechanical properties provides further information on the relationships between dynamic properties and molecular structure.

EXPERIMENTAL

Materials

Three peroxide-cured gum rubbers were supplied by Goodrich-Gulf Chemicals, Inc., for these tests. The individual rubber formulations and properties are given in Table I. The Ameripol 1500 contains 23% styrene. The Ameripol 1009 contains the same styrene content but is crosslinked through divinyl benzene groups. Ameripol 1013 contains 43% styrene.

Test buttons, $7/16$ -in. in diameter, were cut from sheets $1/4$ -in. thick. This particular sample size was chosen since it was the maximum that could be used to permit a uniform radiation dose to be imparted, and also this size was optimum for testing in the twin transducer. Normally samples of differing area/thickness ratio are used in different temperature-frequency regions to permit maximum quantity and quality of dynamic data to be obtained.¹¹ However, the uncertainty of irradiating samples of several different shapes to exactly the same dose made the use of multiple sample sizes undesirable.

Specimen pretreatment was performed as outlined in the ASTM procedure D1672-59T. The buttons were heated at 50°C. for 48 hr. to allow relaxation of internal stresses formed during the cure or specimen cutting. After heating, the buttons were cooled for 48 hr. over Drierite desiccant. Thereafter, except during irradiation, the samples were stored over desiccant at ambient temperatures. The postirradiation procedure was to evacuate the samples for 48 hr. to remove volatile reaction products, heat at 50°C. for 48 hr. to complete free-radical reactions and relieve stresses, and finally to stabilize the specimens at room temperature over desiccant for at least 48 hr. prior to testing.

Irradiation

The specimens were placed in an open top, glass chamber and the chamber including the specimens was irradiated in a 10,000 c.,⁶⁰Co source. The

TABLE I^{a,b}
Materials

Material	Constituent	Analysis	
		Minimum, wt.-%	Maximum, wt.-%
Ameripol 1009 ^c	(Hot fatty acid emulsion polymerization)		
	Volatile matter	—	0.75
	Ash, total	—	1.50
	Organic acid (MW 284)	3.75	6.00
	Soap (MW 306)	—	0.75
	Bound styrene	22.5	24.5
	Antioxidant ^d		
Ameripol 1500 ^c	(Cold rosin acid emulsion polymerization)		
	Volatile matter	—	0.75
	Ash, total	—	1.50
	Organic acid (MW 346)	5.00	7.25
	Soap (MW 384)	—	0.50
	Pound styrene	22.5	24.5
	Antioxidant ^e	1.0	1.75
	Mooney raw viscosity	46	58
Ameripol 1013 ^c	(Hot fatty acid emulsion polymerization)		
	Volatile matter	—	0.75
	Ash, total	—	1.50
	Organic acid (MW 284)	4.00	6.00
	Soap (MW 306)	—	0.01
	Bound styrene	42.00	44.0
	Antioxidant ^d		
	Mooney raw viscosity	40	50

^a Formulation information of Goodrich Gulf Chemicals, Inc.⁹

^b Cure information:¹⁰ curative, Di Cup 40C (Hercules Powder Co.), 5 parts per 100 parts of polymer; cure temperature, 310°F.; cure time, 45 min. The same cure system and cycle was used on all the samples. Ameripol 1009 contains divinylbenzene as the crosslinking agent.

^c Trade name of Goodrich-Gulf Chemicals, Inc., Cleveland, Ohio.

^d Trisnonylphenylphosphate antioxidant, quantity is proprietary.

^e Phenyl- β -naphthylamine antioxidant.

dose rate, determined using a ceric sulfate dosimeter, was between 1.66 and 1.57 Mrad/hr., depending on the sample location and date of the irradiation. Temperature was maintained at 25–30°C. by circulating air around the sample chamber. No radiation heat build-up was noted when the samples were removed from the source.

Dynamic Tests

Testing over a wide range of frequencies and temperatures was needed to plot master curves of reduced modulus versus frequency. A forced vibration technique was considered to be the most applicable and a twin

electromagnetic transducer was constructed. The apparatus design is similar to that developed by Marvin, Fitzgerald, and Ferry¹² with minor modifications by Kurath.¹³

The apparatus was calibrated over the frequency range of 25–1400 cps. Data points throughout the temperature range were checked at extremes in amplitude and the amplitude changes did not alter the data readings.

The twin transducer apparatus was set in a controlled temperature chamber. Tests were conducted at temperatures ranging between -70 and 70°C . The specimens were stabilized for at least 6 hr. at the higher temperatures and at least 10 hr. at the lower temperatures prior to testing. Heat build-up in the sample was slight as expected because of the low strain amplitudes.

The storage (G') and the loss (G'') components of the complex shear modulus were calculated from electrical outputs of the transducer. The data obtained at several temperatures were reduced to -18°C . by using superposition procedures described by Ferry.¹⁴ Loss tangents ($\tan \delta$) were calculated from the measured values of G' and G'' with eq. (1).

$$\tan \delta = G''/G' \quad (1)$$

The relaxation distribution function (H) is defined by eq. (2):

$$G(t) = G_e + \int_{-\infty}^{\infty} H \exp \{t/\tau\} d(\ln \tau) \quad (2)$$

where G is the shear modulus, G_e is the contribution to the modulus at $\tau = \infty$, t is the time, and τ is the relaxation time. Values of H were calculated from points on the reduced storage modulus curves (Figs. 3, 4, and 5) by using the method of Williams and Ferry.¹⁵

Hardness Measurements

Four Shore A hardness values were measured on each specimen with the use of a Wallace electronic microindentation tester. A Zwick hardness tester was used to verify the measurements from the Wallace machine.

Swelling and Sol Measurements

When all the testing has been completed, the test buttons were placed in toluene at 26 – 30°C . After one week the toluene was decanted from the specimens and fresh solvent added to minimize any equilibrium concentrations of sol remaining in the gel.¹⁶

The effective crosslink density (ν/V) was calculated from the swelling data by using the Flory-Huggins relationship given in eq. (3):¹⁷

$$-[\ln(1 - \phi_2) + \phi_2 + u_1\phi_2^2] = V_1(\nu/V)(\phi_2^{1/3} - 1/2\phi_2) \quad (3)$$

where ϕ_2 is the volume fraction polymer in the swollen system, u_1 is the solvent-polymer interaction parameter, V_1 is the solvent molar volume, and ν/V is the crosslink density of polymer network between crosslinks

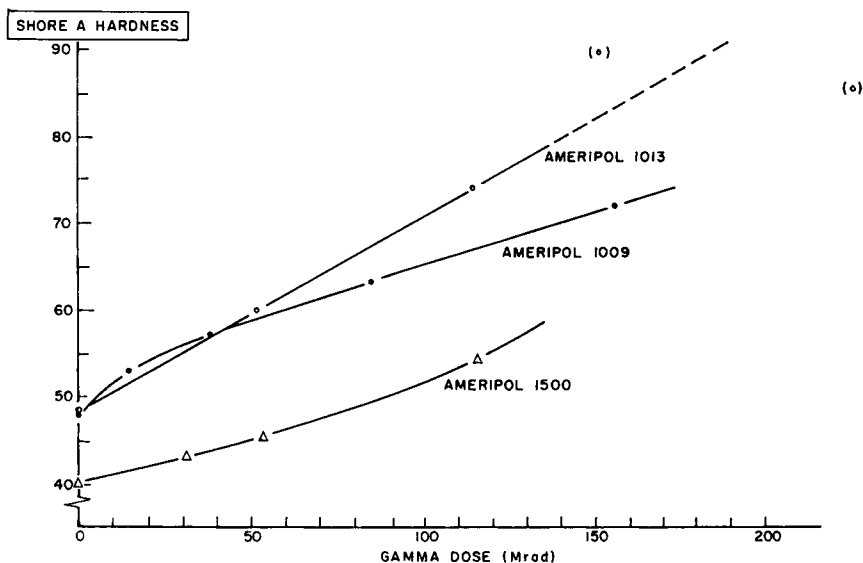


Fig. 1. Effect of γ -radiation on the hardness of styrene-butadiene rubbers.

per cubic centimeter of rubber. A solvent-polymer interaction parameter of 0.41 was used for these calculations.

The solvent was removed from the specimens by vacuum for 2 weeks at room temperature. The difference between the original and final weight was attributed to the quantity of sol extracted by the toluene.

RESULTS AND DISCUSSION

Hardness

Irradiation causes all the samples to increase in hardness as shown in Figure 1. The high styrene content Ameripol 1013 showed a more significant increase in hardness than the other two elastomers of lower styrene content. The two highest hardness values reported are questionable, since they were determined at the upper limit of the tester. Hardness value increases of 13–29% have been reported¹ for filled styrene-butadiene rubbers irradiated to 100 Mrad. The increase measured for these unfilled elastomers were 29, 34, and 43% for the Ameripols 1500, 1009, and 1013, respectively.

Crosslink Density

The swelling ratios and calculated crosslink densities of the irradiated rubbers follow the same pattern as the hardness value changes. The swelling ratios, solvent contents of the swollen rubbers, gel contents of the irradiated specimens, and the calculated crosslink densities are given in Table II. Crosslink densities are plotted against radiation dose in Figure 2.

TABLE II
Effect of Gamma Radiation on the Swelling Characteristics
of Styrene-Butadiene Rubbers

Material	Gamma dose, Mrad	Volume fraction toluene in the swollen rubber	Sol, wt.-%	Crosslink density, mole/cm. ³ × 10 ⁸
Ameripol 1009	0.0	0.73	7.8	29
	15.7	0.72	6.5	31
	38.6	0.69	5.5	42
	85.4	0.63	4.9	67
	156	0.55	4.2	122
Ameripol 1500	0.0	0.82	7.0	10
	31.1	0.80	6.6	16
	53.9	0.78	9.2	19
	118	0.73	5.5	31
Ameripol 1013	0.0	0.76	4.2	21
	51.0	0.66	1.9	55
	114	0.56	1.2	110
	152	0.43	0.1	250
	228	0.41	-2.2	290

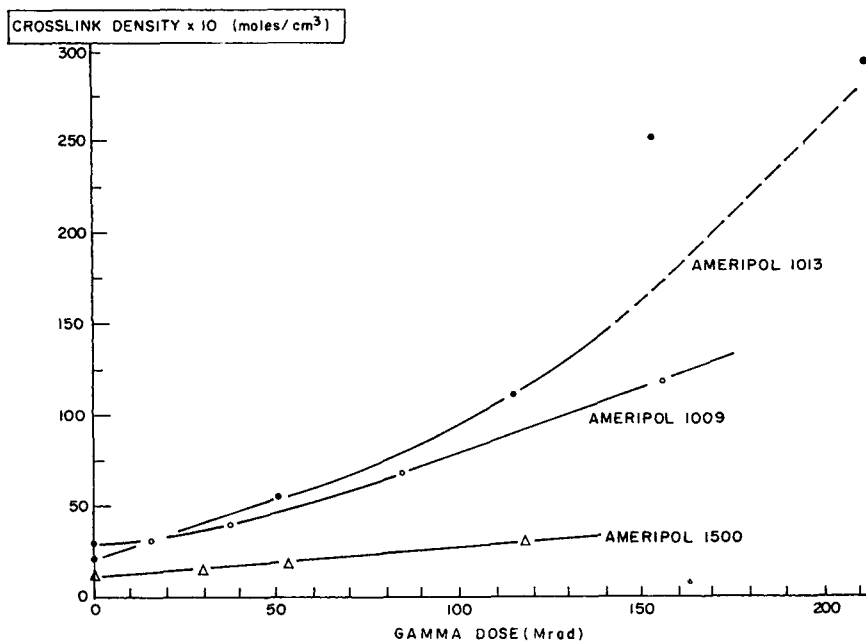


Fig. 2. Effect of γ -radiation on the crosslink density of styrene-butadiene rubbers.

The low styrene content Ameripols 1500 and 1009 both appeared to swell uniformly and remained intact throughout the swelling and drying processes. The high styrene 1013 rubber disintegrated during swelling. The degree of disintegration increased with an increased irradiation level. The Ameripol 1013 was obviously oxidized during the drying process. This is apparent in the negative sol content (final weight after drying was greater than the original weight) determined for the Ameripol 1013 irradiated to 274 Mrad.

The crosslink density of all the materials is increased by γ -irradiation as shown in Figure 2. The Ameripol 1013 is crosslinked to the greatest extent and the Ameripol 1500 the least. The rate of crosslinking appears to increase at the higher dose levels. It should be pointed out that swelling measurements do not differentiate between a true chemical crosslink and one caused by chain entanglements. Consequently, branching or chain rupture that would lead to entanglements could increase the measured crosslink densities.

Storage Modulus

Irradiation increases the storage modulus of the styrene-butadiene rubbers as shown in Figures 3-5. This is in agreement with the previ-

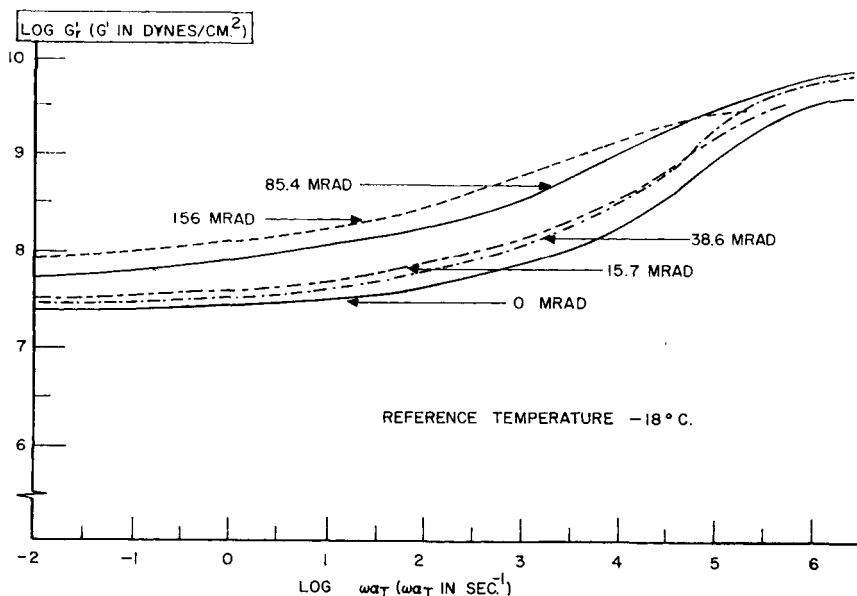


Fig. 3. Effect of γ -radiation on the storage modulus of styrene-butadiene rubbers: Ameripol 1009.

ously mentioned crosslink density increases and hardness changes. Similar increases in dynamic moduli have been reported¹⁸ for irradiated SBR filled with carbon black.

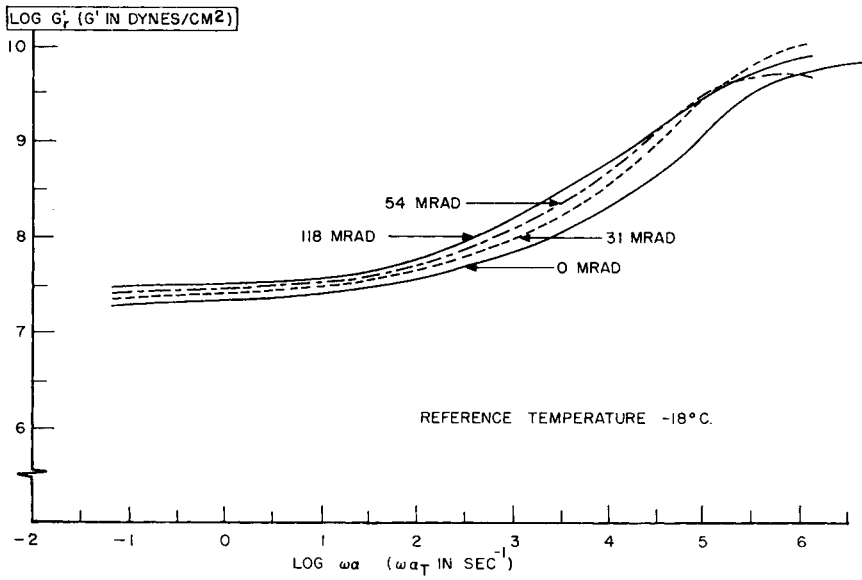


Fig. 4. Effect of γ -radiation on the storage modulus of styrene-butadiene rubbers: Ameripol 1500.

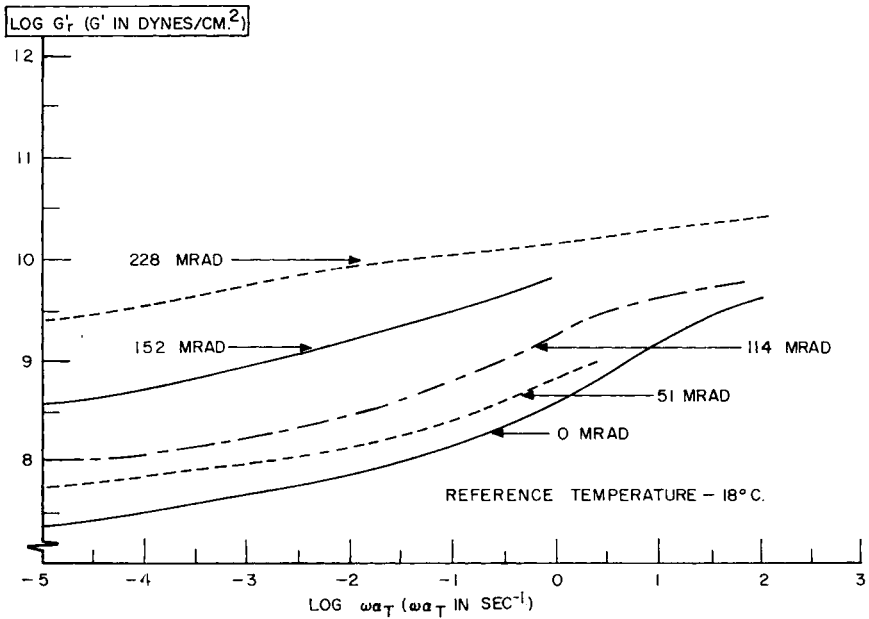


Fig. 5. Effect of γ -radiation on the storage modulus of styrene-butadiene rubbers: Ameripol 1013.

The storage modulus increases at a greater rate at the higher dose levels reflecting the similar change in the crosslink density. The change in modulus per unit dose is dependent on the rate of strain; the change being the greatest in the transition region of the polymer. The properties in this time region reflect the short-range diffusional motion of side groups or very short chain segments.¹⁹ The shift to lower frequencies on increased radiation reflects an increase in the relaxation times brought about by an increase in the monomeric friction coefficient (as is discussed later).

The modulus in the plateau region of the reduced curve is also significantly increased by irradiation of the Ameripol 1009, is only slightly increased in the Ameripol 1500, and appears to also be greatly increased for the Ameripol 1013 although a true plateau is not defined for the 1013. Chain entanglements and crosslinks control material responses in this time region.

Loss Modulus

The loss moduli of the three rubbers increase during irradiation as shown in Figures 6-8. The modulus increase continues at all radiation levels

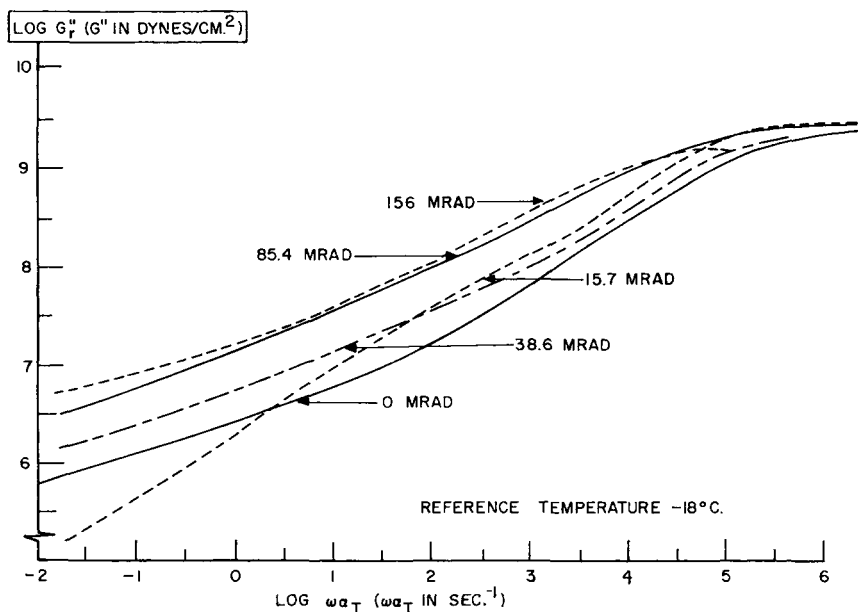


Fig. 6. Effect of γ -radiation on the loss modulus of styrene-butadiene Rubbers: Ameripol 1009.

for the 1013 rubber; however, the loss modulus no longer increases after a dose of about 50 Mrad for the Ameripols 1009 and 1500.

The loss modulus is a measure of the energy dissipated per cycle of sinusoidal deformation. The dynamic viscosity n' calculated from the loss

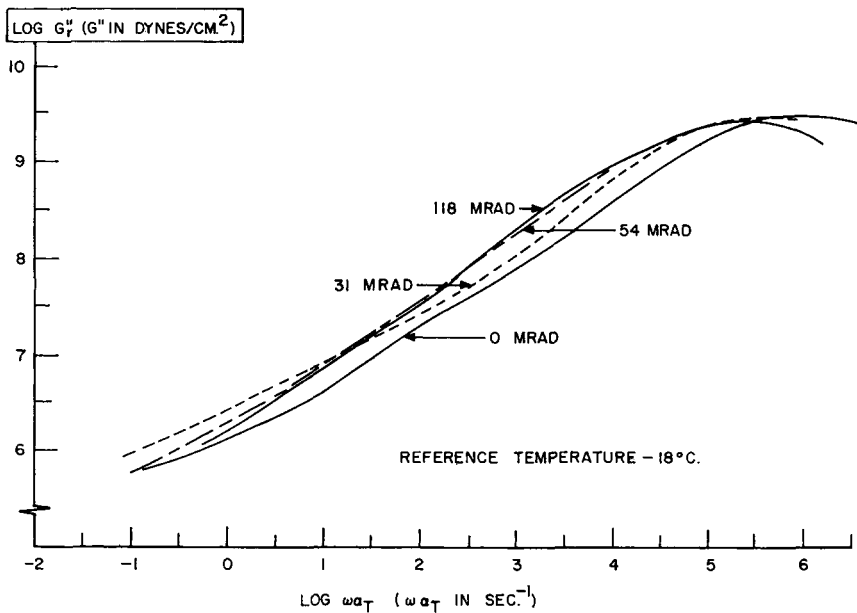


Fig. 7. Effect of γ -radiation on the loss modulus of styrene-butadiene rubbers: Ameripol 1500.

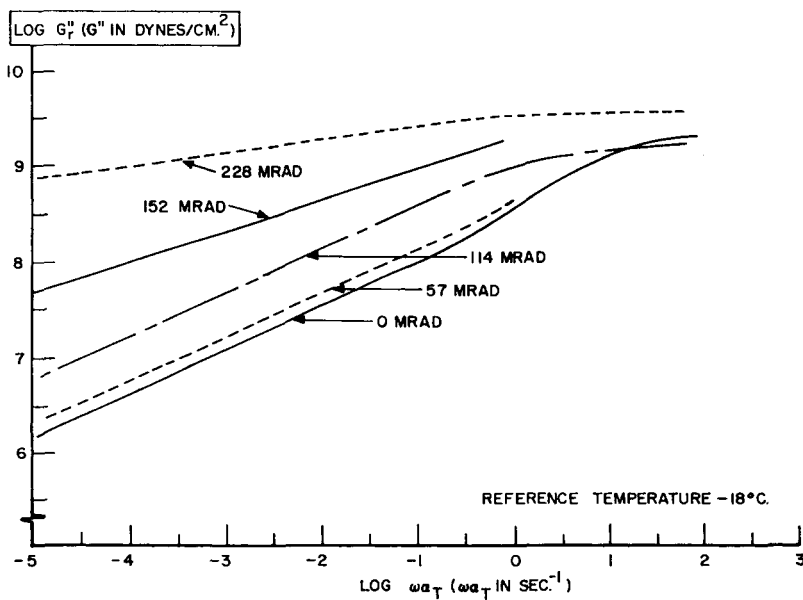


Fig. 8. Effect of γ -radiation on the loss modulus of styrene-butadiene rubbers: Ameripol 1013.

modulus is a quantity which aids in the interpretation of the loss modulus curves.

$$n' = G''(\omega)/\omega \quad (4)$$

The irradiation increases the crosslink density, which makes the rubber more viscous. The loss modulus and viscosity increase with irradiation to a dose level of about 50 Mrad for the Ameripols 1009 and 1500 and thereafter the loss modulus remains constant. This would indicate that scissioning reactions may become important. The increasing crosslink density and storage modulus at doses above 50 Mrad might then be attributed to entanglement networks. The continuing increase in the loss modulus of the Ameripol 1013 suggests that the higher styrene content is limiting the scission reaction and promoting crosslinking.

Loss Tangent

The loss tangent provides a measure of the ratio of the energy dissipated to the energy stored during cyclic deformation. The loss tangent does not have a clear molecular interpretation but is of practical interest because it locates time-temperature regions of high energy dissipation.

The loss tangent curves are decreased in height and broadened by exposure of the materials to γ -radiation, as shown in Figures 9-11. The loss maxima are also shifted to lower frequency levels by the irradiation. This

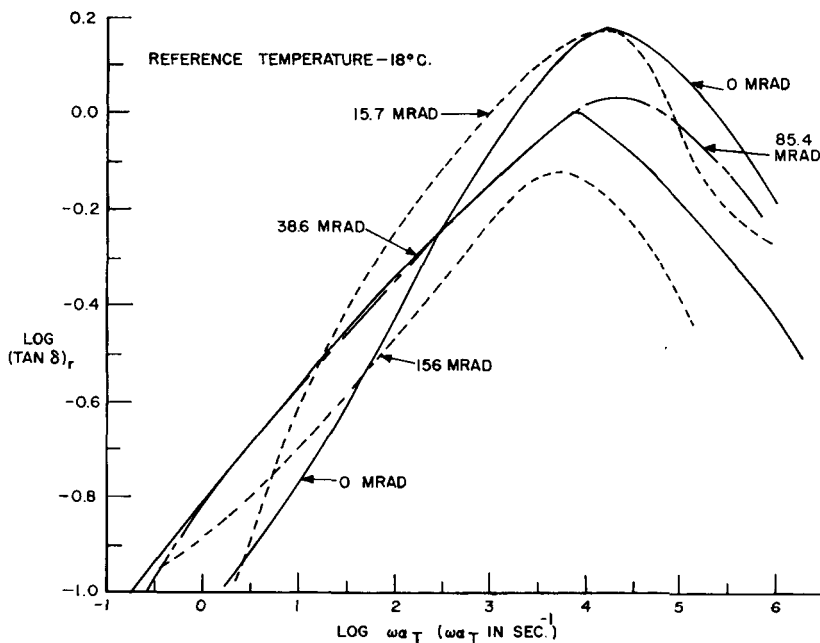


Fig. 9. Effect of γ -radiation on the loss tangent of styrene-butadiene rubbers: Ameripol 1009.

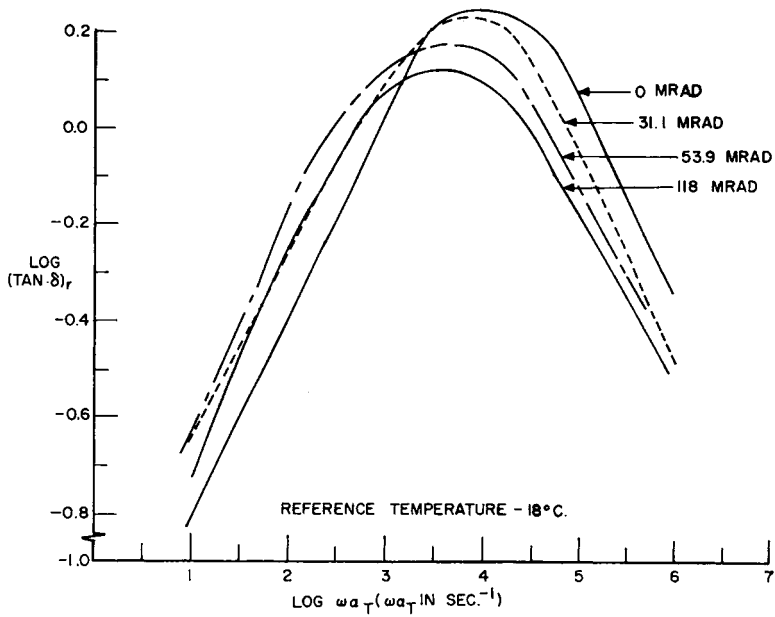


Fig. 10. Effect of γ -radiation on the loss tangent of styrene-butadiene rubbers: Ameripol 1500.

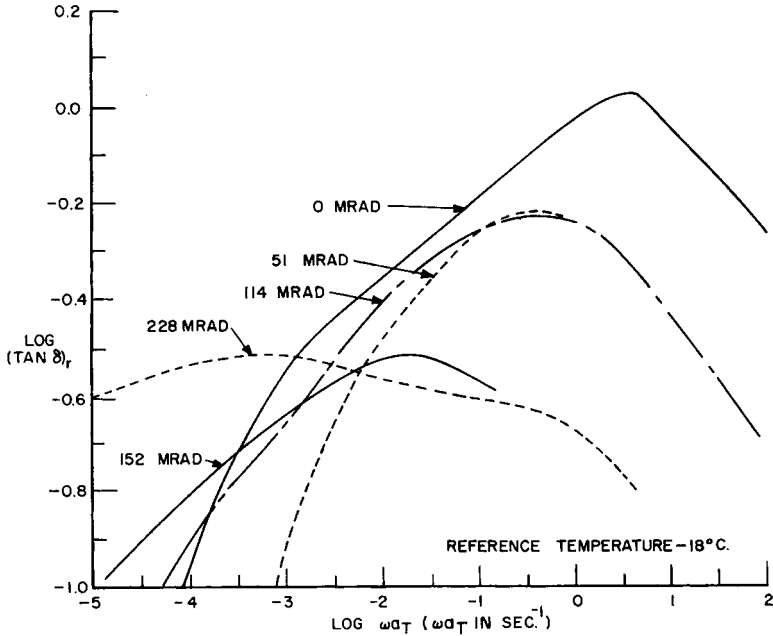


Fig. 11. Effect of γ -radiation on the loss tangent of styrene-butadiene rubbers: Ameripol 1013.

overall shift is due to an increased rigidity of the polymer network and is typical for systems of increasing crosslink density.²⁰ The width of the loss tangent curve has also been attributed to heterogeneity of the polymer structure.²¹

Relaxation Spectra

The relaxation spectra changes caused by radiation are shown in Figures 12–14. The effect of radiation is to raise the level of the spectrum and to extend the spectrum to considerably longer times. This is in accord with the prior data because increasing crosslink density requires an increased number of coordinated molecular movements for relaxation to occur.

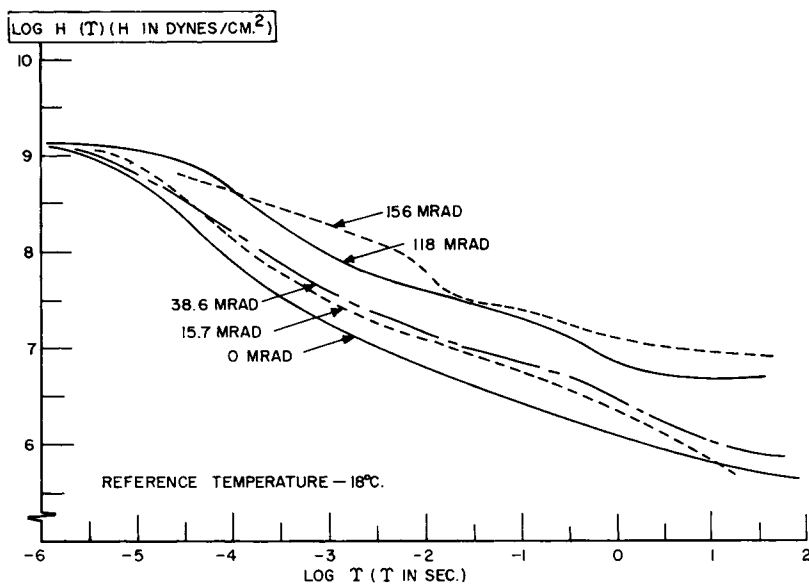


Fig. 12. Effect of γ -radiation on the relaxation spectra of styrene-butadiene rubbers: Ameripol 1009.

The extension of the spectra to long times is particularly evident in the Ameripol 1009 and 1013 systems. These spectra and the loss tangent data at long times show the opposite trend as the data of Stratton²² and Ferry,²³ in which the spectra of natural rubbers were extended to longer times and the loss tangent increased as the crosslink density decreased. Trapped coupling entanglements were suggested²³ as a possible explanation to the natural rubber data. A similar explanation could be considered for the materials used in this research, since entanglement couplings have been shown^{9,21} to play important roles in the loss properties of styrene-butadiene rubbers.

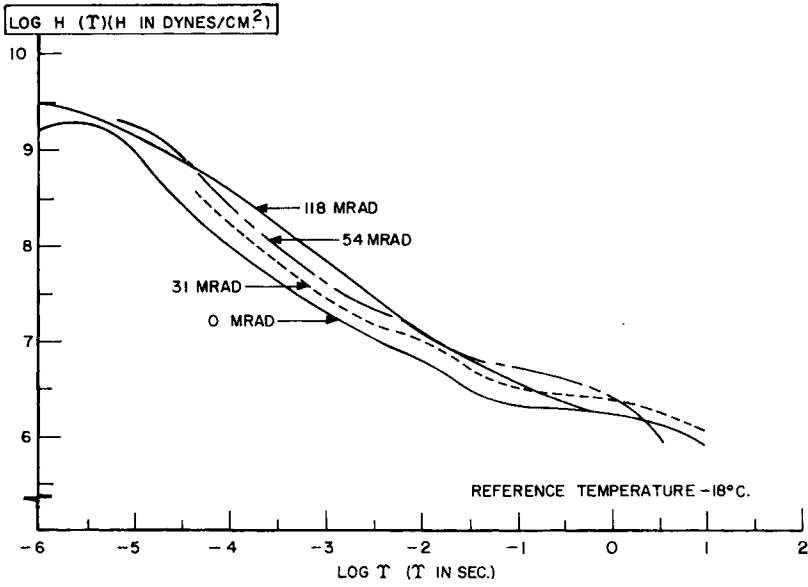


Fig. 13. Effect of γ -radiation on the relaxation spectra of styrene-butadiene rubbers; Ameripol 1500.

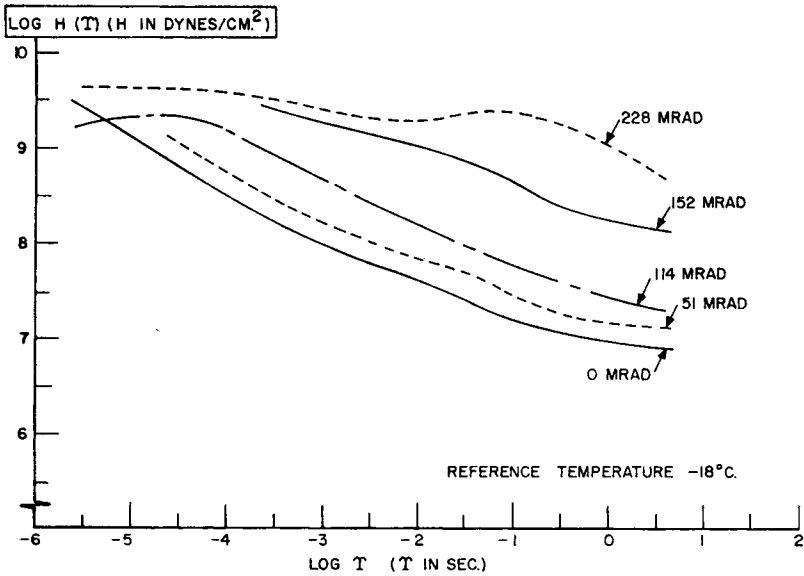


Fig. 14. Effect of γ -radiation on relaxation spectra of styrene-butadiene rubbers; Ameripol 1013.

TABLE III
Relative Monomeric Frictional Coefficients of Irradiated
Styrene-Butadiene Rubbers

Rubber	Gamma dose, Mrad	Log τ	Log H	ζ_0 irradiated
				ζ_0 unirradiated
Ameripol 1009	0.0	-2.90	7.22	1.0
	15.7	- .295	7.45	2.6
	38.6	-3.05	7.62	7.8
	85.4	-2.95	7.95	23
	156	- .230	8.05	170
Ameripol 1500	0.0	-2.50	7.03	1.0
	31.1	-2.70	7.30	2.2
	53.9	-2.65	7.43	4.5
	118	No region of slope -0.5		
Ameripol 1013	0.0	-3.50	8.22	1.0
	51.2	-3.45	8.43	2.9
	114	-3.45	8.90	26
	152	No region of slope -0.5		
	228	No region of slope -0.5		

The monomeric frictional coefficient ζ_0 , calculated from the relaxation spectra, is a measure of the force involved in pushing a chain segment through its local surroundings. The monomeric frictional coefficients of the irradiated copolymers were determined relative to the coefficients of the nonirradiated copolymers. Values are reported in Table III. The reported changes are not considered to be too significant, since there was no defined portion of the relaxation spectra that had the theoretical slope of -0.5 . Also Ferry¹⁴ has questioned the meaning of the frictional coefficient for highly crosslinked systems as these.

Nevertheless, it is interesting to compare the calculated values. The coefficient increases in all cases as would be expected with increasing cross-link density. The values for all three systems increase approximately the same amount to dose levels on the order of 100 Mrad. The coefficient does increase at a higher rate with increasing dose. This is particularly noticeable above doses of 50 Mrad. This, in conjunction with the loss modulus data, suggests the importance of entanglement networks in the response of SBR systems.

Time-Temperature Shift Factors

Figures 15-17 show that, within the accuracy of these data, γ -radiation has no effect on the time-temperature shift factors. This is to be expected for highly crosslinked systems.

The data can be correlated by using the WLF equation. The solid lines in Figures 15-17 represent calculated values from the WLF equation with constants derived from a least-squares analysis of the data. The derived constants are given in Table IV.

TABLE IV
Time-Temperature Relationships for Styrene-Butadiene Rubbers

	Ameripol 1009	Ameripol 1500	Ameripol 1013
WLF equation			
Reference temperature, °C.	-18	-18	-18
C_1	-12.08	-8.53	-13.57
C_2	118.1	94.01	74.08
T_s , °K.	264	256	289
T_s , °K. ^a	266	266	287

^a Data of Williams.²⁴

A convenient form of the WLF equation was suggested by Williams:²⁴

$$\log a_T = -8.86(T - T_s)/(101.6 + T - T_s) \quad (5)$$

where a_T is the time-temperature shift factor, T is the test temperature, and T_s is the reference temperature needed to fit the equation to the data. The only variable in the equation is T_s . T_s values have been determined²⁴ for a series of butadiene-styrene rubbers on the basis of stress relaxation data.^{25,26} Values for T_s for the 23% and the 44% styrene rubbers determined from interpolation of the literature data²⁴ are compared with values obtained from this experimental data in Table IV. There is excellent agreement between the two sets of data.

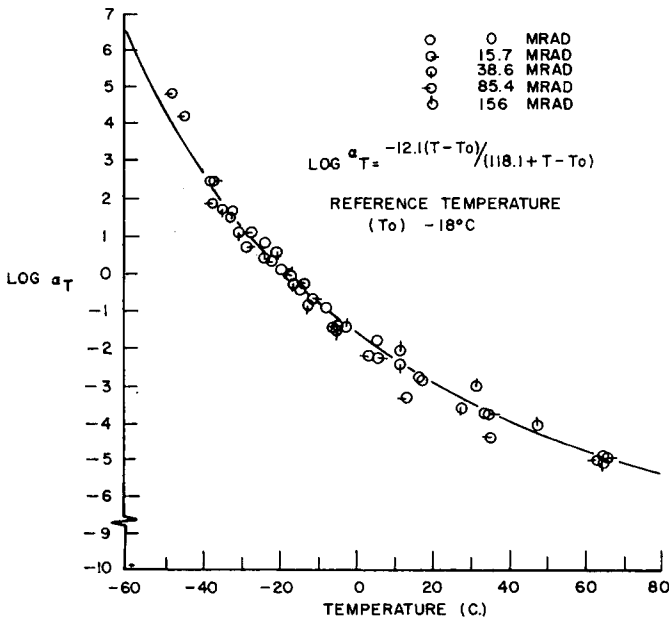


Fig. 15. Time-temperature shift factor for styrene-butadiene rubbers; Ameripol 1009.

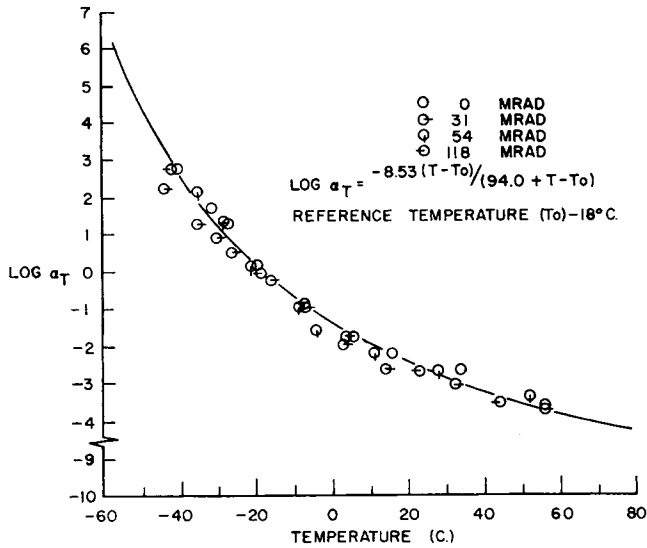


Fig. 16. Time-temperature shift factor for styrene-butadiene rubbers: Ameripol 1500.

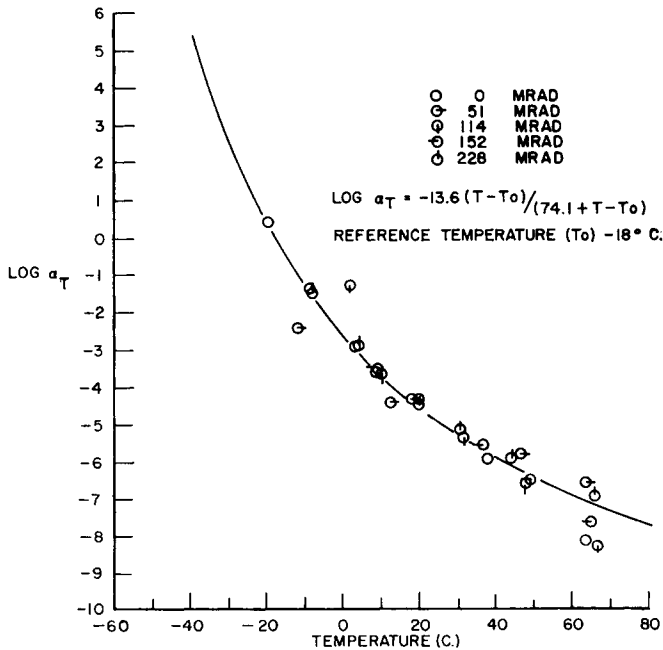


Fig. 17. Time-temperature shift factor for styrene-butadiene rubbers: Ameripol 1013.

CONCLUSIONS

Hardness values, crosslink densities, and dynamic moduli of styrene-butadiene elastomers are increased as a result of γ -radiation. The changes in dynamic properties correlate very well with the changes in crosslink

densities. The increase in property values is primarily due to crosslinking but chain entanglements also appear to be important.

The effect of crosslinking of Ameripol 1009 is more evident than the crosslinking of the Ameripol 1500 in their dynamic properties. This is possible because a "looser" network in the divinylbenzene-crosslinked 1009 is more restricted by a radio-induced crosslink than the 1500 network structure.

The copolymer of high styrene content was apparently crosslinked to a greater extent by radiation than the rubbers of lower styrene content. Analysis of the dynamic data suggests that the aromatic group enhances the crosslinking reaction and minimizes the scission effects. This is evident in the continued increase in the loss modulus on irradiation of the 43% styrene copolymer whereas the loss moduli of the 23% styrene rubbers remained essentially constant at doses above 50 Mrad. Further work is needed to define the mechanism of "protection" against radiation by aromatic structures in polymer chains.

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Résumé

Trois différentes gommes de caoutchouc, à base de styrène-butadiène, ont été exposées à des radiations- γ à des doses variant de 0 à 200 Mrads. Les densités de pontage, la dureté et les propriétés mécaniques dynamiques ont été évaluées. Ces échantillons croissaient en dureté et en densité de pontage en cours de radiation. Ceci était reflété par un module croissant, un maximum de tangente de perte élargie, et un spectre de relaxation accru. Les données dynamiques sont représentées sous forme de courbes-maitresses qui sont développées en utilisant l'équation WLF.

Zusammenfassung

Drei verschiedene ungefüllte Styrol-Butadien-Kautschukproben wurden γ -Strahlungsdosen im Bereich von 0 bis 200 Mrad ausgesetzt. Die Vernetzungsdichte, die Härte und die dynamisch-mechanischen Eigenschaften wurden bestimmt. Alle Proben nahmen während der Bestrahlung an Härte und Vernetzungsdichte zu. Dies äusserte sich in einer Erhöhung des Realteils des komplexen Moduls, einer Verbreiterung des Maximums des Verlusttangens und einer Vergrößerung des Relaxationsspektrums. Die dynamischen Daten werden in Masterkurven dargestellt, welche mittels der WLF-Gleichung entwickelt wurden.

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