JOURNAL OF APPLIED POLYMER SCIENCE VOL. 8, PP. 707-721 (1964)

Cut Growth and Fatigue of Rubbers. II. Experiments on a Noncrystallizing Rubber

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Synopsis

Tensile fatigue failure of a gum vulcanizate of noncrystallizing SBR can be accounted for by the growth of small flaws initially present in the rubber. Fatigue of crystallizing natural rubber was shown in Part I to be attributable to the same cause. Cut growth results are interpreted in terms of the tearing energy theory of Rivlin and Thomas. SBR exhibits cut growth under both static and dynamic conditions; in each case the rate is approximately proportional to the fourth power of the tearing energy. Variation of the dynamic cut growth rate with frequency can be explained by the summation of a time-dependent static component of growth and a cyclic component not dissimilar to that occurring in natural rubber. Fatigue failure, under both static and dynamic conditions, is predicted from the cut growth results. These predictions are found to account quantitatively for experimentally observed fatigue lives when a suitable value is assumed for the initial flaw size. Fatigue lives at different temperatures correlate well with cut growth results obtained by Greensmith and Thomas over the same temperature range. The results are compared to those obtained previously for natural rubber, and possible reasons for the differences in fatigue behavior of crystallizing and noncrystallizing rubbers are discussed.

Introduction

In Part I of this series[1] (subsequently referred to as I) the fatigue failure of a natural rubber gum vulcanizate, in the absence of heat build-up, was shown to be explicable in terms of the growth, by repeated tearing, of small flaws initially present in the rubber. This second paper describes cut growth and fatigue measurements on an SBR gum vulcanizate, and shows that, with some important differences, a similar theory can be used to interpret the results.

In contrast to strain crystallizing natural rubber, tearing in a vulcanizate of noncrystallizing SBR occurs at constant deformation.[2] This type of tearing is termed static cut growth. When the same deformation is applied and removed several times per second, the rate of growth is much greater than would result from static cut growth. Thus there is, additionally, a dynamic component to the tearing. Changing the magnitude of the deformation markedly affects the observed rate of growth in both cases.

These aspects of cut growth are examined and the results extended to the interpretation of fatigue failure in SBR under both static and dynamic conditions.

Tearing Energy Theory

A theory enabling the tearing of rubbers, for different shapes of test piece and under different modes of deformation, to be expressed in terms of a single parameter, the tearing energy, has been developed by Rivlin and Thomas.[3] The tearing energy T is defined as the elastic strain energy lost by a test piece per unit area of newly formed surface.

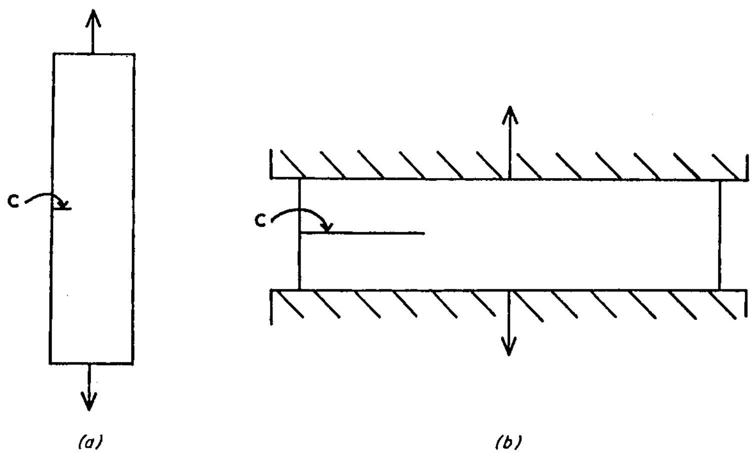

Fig. 1. Schematic diagrams of (*a*) tensile test piece; (*b*) pure shear test piece.

For a tensile test piece (Fig. 1*a*)

$$T = 2kWc \tag{1}$$

where c is the cut length, W the strain energy per unit volume in the bulk of the test piece (i.e., remote from the cut), and k a slowly varying function of the strain, determined empirically by Greensmith.[4]

For a pure shear test piece (Fig. 1*b*)

$$T = Wl_0 \tag{2}$$

where l_0 is the unstrained height of the test piece.

Experimental Details

Pure shear and tensile test pieces were cut from 1 mm.-thick molded sheets of an SBR gum vulcanizate. The mix formulation is given in Table I.

The tensile strips were tested in a similar manner to that described in I for natural rubber. Additionally, in the static tests the cut growth rate dc/dt was approximated from readings of cut length c at time t by $\Delta c/\Delta t$, where Δc was the change in cut length measured over the time interval

TABLE I
Mix Formulation[a]

	Parts by weight
SBR(Polysar S)	100
Zinc oxide	5
Stearic acid	2
N-Cyclohexyl-2-benzthiazyl sulfenamide	1
Sulfur	1.75
Phenyl-β-naphthylamine	1

[a] Cure time: 50 min. at 140°C.

Δt. Provided $\Delta c/c$ was less than 0.2, the calculated error introduced by this approximation was less than 4%. The accuracy of the Δc measurement was always better than 10%.

The pure shear test pieces were approximately 30 cm. wide and 2.5 cm. high. A specific strain energy (W) versus strain (e) curve was obtained from a pure shear load-deflection test on one of the test pieces. Cut growth testing was carried out on a dynamic testing machine having a frequency range of 0.1 to 1000 cycles/min. Each test piece was set to be strained for one half of each cycle and relaxed for the other half. The growth of the cut, initially about 4 cm. long to avoid end effects, was measured with a traveling microscope.

Growth from a Razor Cut

In the majority of tests the initial cut was made with a razor blade. In Figure 2 the growth of such a cut under dynamic conditions is shown for

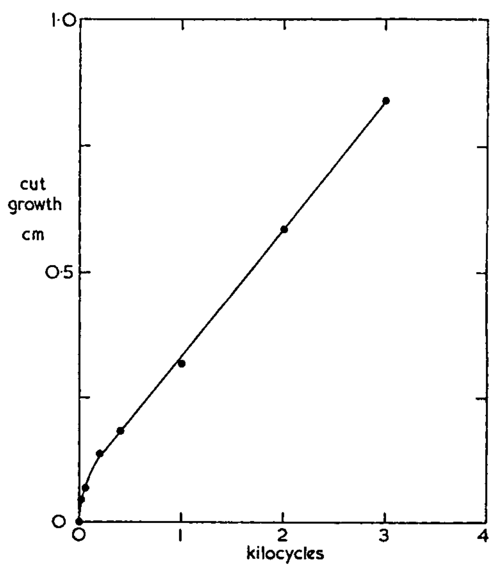

Fig. 2. Growth of a razor cut in a pure shear test piece.

a pure shear test piece in which the tearing energy is independent of the length of the cut. During the first few hundred cycles the rate at which the cut is growing decreases considerably, thereafter becoming substantially constant. From the appearance of the torn surfaces of the cut the slower, steady growth is referred to as rough growth, and the initial growth from the razor cut as smooth growth. In analyzing the experimental results the initial smooth growth is normally ignored.

Dependence of rate on T

Static cut growth tests were carried out on pure shear and tensile test pieces. The results are shown in Figure 3 as cut growth rate dc/dt against tearing energy T plotted on logarithmic scales. The line through the tensile points has the form

$$dc/dt = T^4/G_s \tag{3}$$

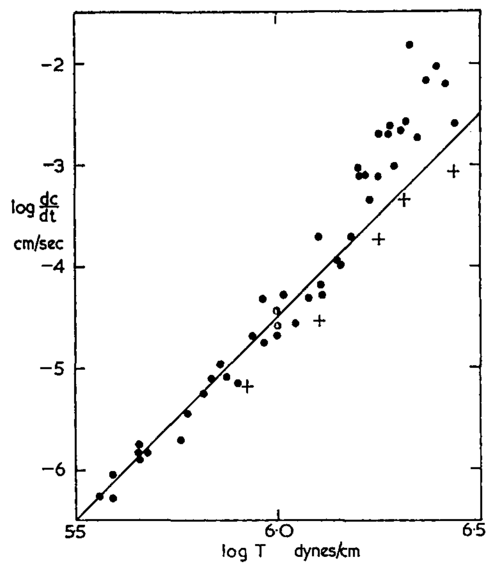

Fig. 3. Relationship between the cut growth rate dc/dt and the tearing energy T on static tests: (●) tensile test pieces; (+) pure shear test pieces; (——) eq. (3) with $G_s = 3.2 \times 10^{28}$ c.g.s. units.

where G_s, termed the static cut growth constant, is equal to 3.2×10^{28} c.g.s. units. The pure shear points lie below, but parallel to, the tensile results, the corresponding G_s value being 6.3×10^{28} c.g.s. units. According to theory these G_s values should be identical. In practice the difference corresponds to a change in T of less than 20%, which is not unsatisfactory in view of the variability of the elastic properties of SBR with time and temperature.

Dynamic cut growth measurements were made on the pure shear test pieces at 316 cycles/min. over a range of maximum strains. Several

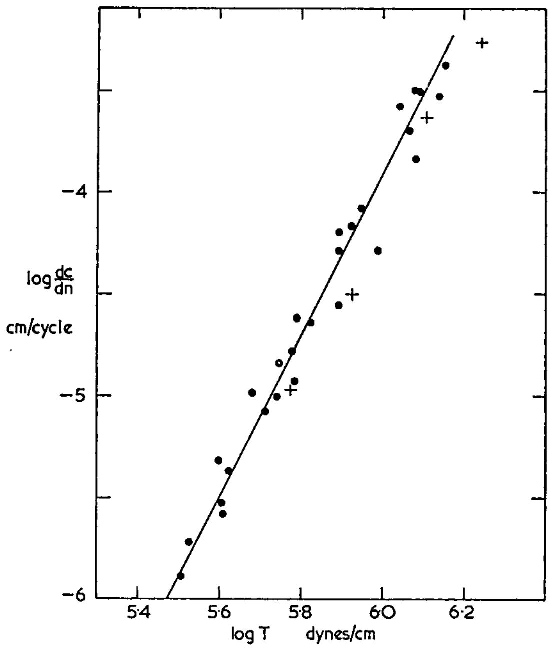

Fig. 4. Relationship between the cut growth rate dc/dn and the tearing energy T on dynamic tests: (●) tensile test pieces at 100 cycles/min.; (+) pure shear test pieces at 316 cycles/min.; (——) eq. (4) with $G = 0.8 \times 10^{28}$ c.g.s. units.

tensile strips were also examined, the tests being carried out on another machine at 100 cycles/min. As will be shown below there is little effect of frequency above 100 cycles/min. The results are shown in Figure 4. The line through the tensile points corresponds to

$$dc/dn = T^4/G \quad (4)$$

where n is the number of cycles and G, the dynamic cut growth constant, is 0.8×10^{28} c.g.s. units. The pure shear results are also consistent with this fourth power relationship, but are slightly lower than the tensile results.

These results show that: (*1*) in both static and dynamic tests the cut growth rate is proportional to the same (fourth) power of the tearing energy T, and (*2*) for any cycle the dynamic cut growth rate is at least ten times the static cut growth rate at the maximum strain.

The Frequency Dependence of G

The contribution of static cut growth to the total dynamic cut growth rate is given from eq. (*3*) by

$$(dc/dt)_{av} = F\,T^4/G_s \quad (5)$$

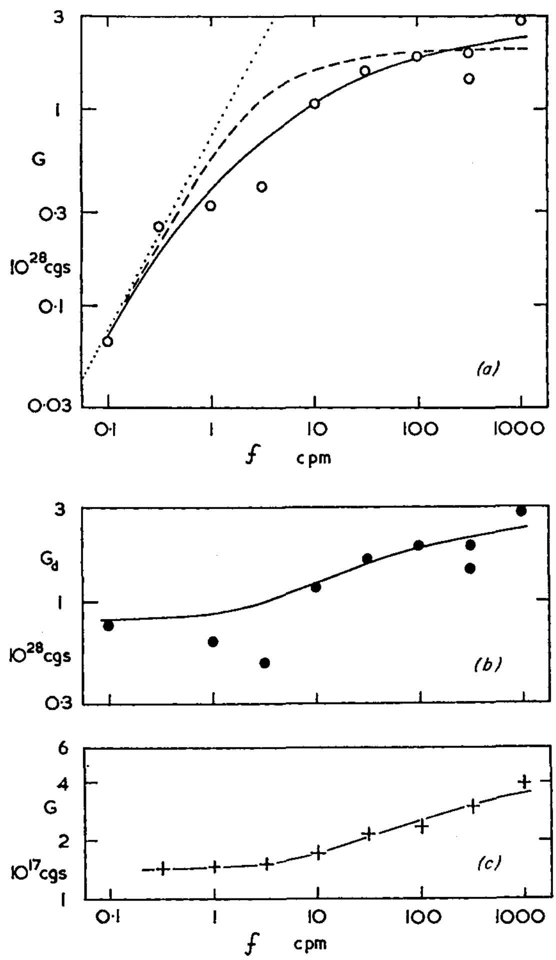

Fig. 5. Plots of (*a*) variation of the cut growth constant G, calculated from eq. (4), with frequency on pure shear tests, (· · ·) static result from Figure 3, (- - eq. (10) with constant G_d; (*b*) the dynamic component of the cut growth constant G_d derived from (*a*) by eq. (10); (*c*) variation with frequency of the cut growth constant G for pure shear test pieces of natural rubber.

where F is the time fraction of each cycle for which T^4 may be considered as operating, namely

$$F = \int_0^{t_1} T_t^4 \, dt / T^4 t_1 \tag{6}$$

where T_t is the tearing energy at time t, T the maximum tearing energy, and t_1 the time to complete one cycle. Approximate values of F have been calculated for the following (see Appendix): (*1*) test piece relaxed for half of each cycle (pure shear F = 0.145, tensile F = 0.156); (*2*) test piece relaxed instantaneously only once each cycle (pure shear F = 0.210, tensile F = 0.226).

Equation (5) for the static component of growth can thus be rewritten

$$(dc/dn)_s = FT^4/fG_s \tag{7}$$

where $f = dn/dt$, the frequency.

It is now possible to calculate the contribution of the static component $(dc/dn)_s$ to the total growth dc/dn of Figure 4, and it is found to be less than 3%. The remaining growth is therefore due to a dynamic component of cut growth $(dc/dn)_d$, having the form

$$(dc/dn)_d = T^4/G_d \tag{8}$$

where G_d is the dynamic-component cut growth constant. The total growth under dynamic conditions can be written as

$$dc/dn = (dc/dn)_s + (dc/dn)_d \tag{9}$$

By combining eqs. (4), (7), (8), and (9), the total cut growth constant G for dynamic growth will be given by

$$1/G = (F/fG_s) + (1/G_d) \tag{10}$$

The variation of G with frequency has been found experimentally by using pure shear test pieces. With a pure shear test piece, in which T is independent of the length of the cut, any error in the determination of T does not affect the relative values of G at different frequencies. The results are shown in Figure 5*a*, G being calculated from eq. (4). The dotted line corresponds to the static cut growth constant from Figure 3, in the form

$$G = fG_s/F$$

The broken line in Figure 5*a* is derived from eq. (10) with G_d assumed independent of frequency. The full line has been drawn through the experimental points.

In Figure 5*b* G_d has been obtained by using eq. (10) and the experimentally observed values of G and G_s. The line corresponds to the full line of Figure 5*a*. The variation of G_d with frequency is very similar to that of the dynamic cut growth constant of natural rubber which has no static component. The results of pure shear tests on the natural rubber gum vulcanizate used in I are shown in Figure 5*c* for comparison.

Static Fatigue of a Tensile Strip

The cut growth rate in tensile strips, unlike that in pure shear test pieces, is dependent upon the length of the cut, c. The theoretical manner in which c varies with time t is given by integrating the differential equation (3), $2kWc$ being substituted for T, whence

$$t = \frac{G_s}{3(2kW)^4}\left(\frac{1}{c_0^3} - \frac{1}{c^3}\right) \tag{11}$$

where c_0 is the initial length of the cut.

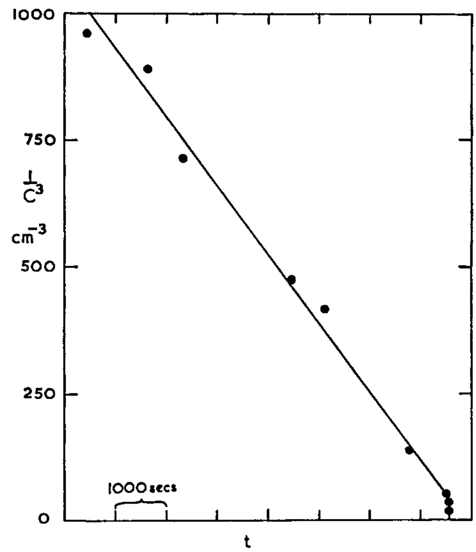

Fig. 6. Growth of a single cut during a static tensile test at 50% strain. The static cut growth constant G_s determined from the slope of the line and eq. (11) is 3.4×10^{28} c.g.s. units.

In Figure 6 the results of one test used for the data of Figure 3 have been replotted in this way. The linear relation between t and the reciprocal of the cube of the cut length given by eq. (11) is found to be obeyed, and the slope of the line gives $G_s = 3.4 \times 10^{28}$ c.g.s. units. The initial growth from the razor cut has been ignored. There is, however, a departure immediately prior to failure. This can be best explained by reference to Figure 3, where the upward departure of the experimental points from the line at high tearing energies is due to a transition from rough to smooth growth as T approaches a value at which tearing occurs catastrophically.[3,5]

If a tensile strip of SBR is left under load it will eventually fail by the growth of a cut across it. Growth may commence not only at intentionally introduced cuts but also at molding imperfections, at flaws produced during cutting or from ozone cracks. In eq. (11), t is the time taken for a cut to grow from a length c_0 to a length c. However, when c is a few times greater than c_0, almost the whole of the life of the test piece will have elapsed. The static fatigue life, t_f, is then given by

$$t_f = \frac{G_s}{3(2kW)^4 c_0^3} \tag{12}$$

This relationship can be verified experimentally by (*1*) testing at constant strain, thereby keeping $2kW$ constant, and varying c_0 and (*2*) keeping c_0 constant and varying the strain. Verification by (*1*) using test pieces containing razor cuts of different length is unsatisfactory due to the rapid initial smooth growth. It is possible, however, to utilize the results of the

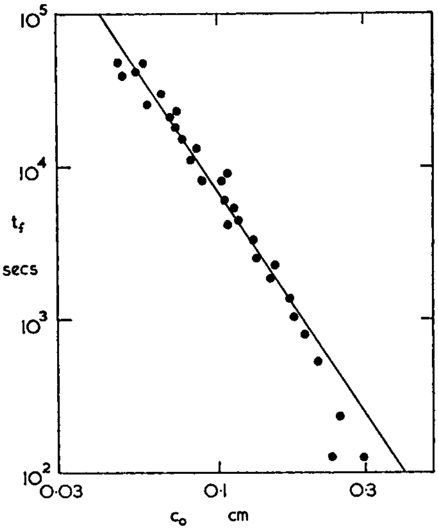

Fig. 7. Relationship between time to failure t_f and length of cut c_0 of static tensile tests at 50% strain. The line corresponds to eq. (12) with $G_s = 3.2 \times 10^{28}$ c.g.s. units.

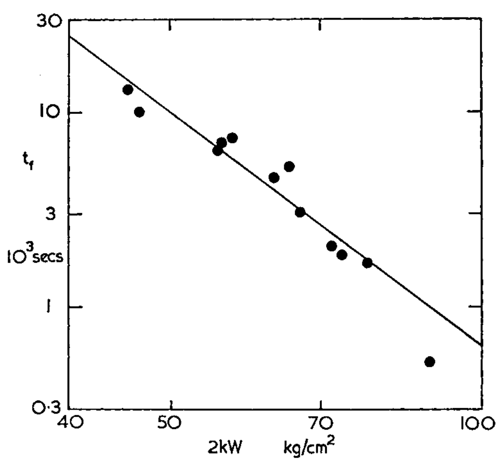

Fig. 8. Dependence of fatigue life t_f on the strain energy $2kW$ of static tensile test pieces. The line has a slope of -4 in accordance with eq. (12), and corresponds to $c_0 = 5.5 \times 10^{-3}$ cm. Each point is the average of three experimental results.

cut growth tests by plotting the time prior to failure (equivalent to t_f) against the measured length of the cut (equivalent to c_0). The results of three tests at 50% strain are shown in Figure 7.

The full line through the experimental points represents eq. (12) with $G_s = 3.2 \times 10^{28}$ c.g..s units, and confirms the prediction that $t_f \propto c_0^{-3}$. The deviation from this line at high cut lengths arises from the departure of the experimental results in Figure 3 from the approximate cut growth relationship, eq. (3).

To try to avoid the large effect which a razor incision has on the initial growth of a cut, test pieces without inserted cuts were used to examine the dependence of t_f on strain energy, W. The results of these static fatigue tests, carried out at high strains, are shown as $2kW$ versus t_f in Figure 8. The value of k at these strains is not known, but extrapolation of Greensmith's results[4] indicates a value of about 1.7.

Despite the large scatter, a line of slope −4, as indicated by eq. (12), is a reasonable fit to the experimental points. A value for the length c_0 of the initial flaws of 5.5 × 10^{-3} cm. is necessary for the line to correlate with eq. (12). This value is of similar order to that of 2.5 × 10^{-3} cm. obtained in I for natural rubber. It must be emphasized that these values are effective flaw sizes, and may arise due to inhomogeneities or impurities in the rubber, or to flaws introduced during die stamping or molding. Physically, the latter do not appear to be as large as the theoretical values, but it may be that, as in the case of growth from a razor cut, the initial growth from these flaws is smooth. The difference between the effective flaw sizes for natural rubber and SBR may be due to the greater effect of smooth growth in SBR.

Much of the scatter in the fatigue results of SBR could arise from variations in c_0; for example, if c_0 varies by a factor of 2, then t_f will vary by a factor of 8.

Dynamic Fatigue

The dependence of the dynamic cut growth rate on tearing energy eq. (4), leads to a relationship between cut length c and number of cycles n, similar to that of static cut growth, namely

$$n = \frac{G}{3(2kW)^4}\left(\frac{1}{c_0^3} - \frac{1}{c^3}\right) \tag{13}$$

As c becomes large compared to c_0, n tends towards a limiting value which is termed the fatigue life, N.

$$N = G / 3(2kW)^4 c_0^3 \tag{14}$$

In dynamic cut growth tests, the number of cycles to failure was also recorded to give fatigue life (N) values from the various observed lengths of the cut (c_0) during rough growth. These results are shown in Figure 9. The full line corresponds to eq. (14) with G equal to 0.8 × 10^{28} c.g.s. units.

The fatigue of dumbbell test pieces, with no inserted cuts, was carried out over a range of strains of 80–300%. The results N versus $2kW$ are shown in Figure 10. The line through the points has a slope of −4, vide eq. (14), and from its position a value of the effective initial flaw size c_0 is found to be 5.6 × 10^{-3} cm., as in the static tests.

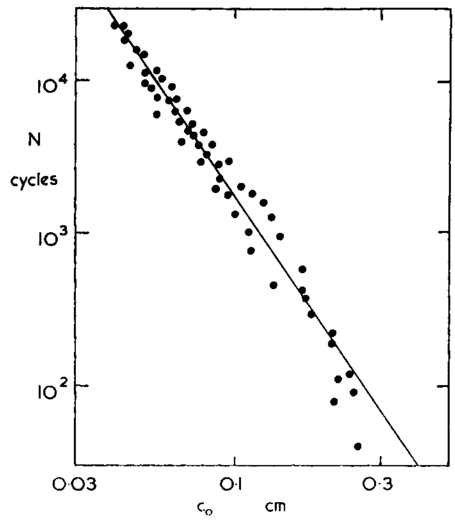

Fig. 9. Relationship between number of cycles to failure N and length of cut c_0 in tensile tests at 100 cycles/min. and 50% maximum strain. The line corresponds to eq. (14) with $G = 0.8 \times 10^{28}$ c.g.s. units.

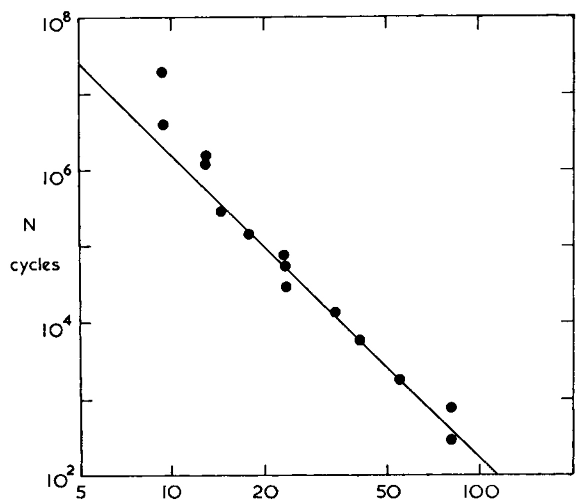

Fig. 10. Dependence of fatigue life N on the strain energy $2kW$ of dumbbell (tensile) test pieces at 100 cycles/min. The line has a slope of -4 in accordance with eq. (14), and corresponds to $c_0 = 5.6 \times 10^{-3}$ cm. Each point is the average of three experimental results.

Effect of Temperature

Temperature variation is known to have a considerable effect on the tear strength of SBR gum vulcanizates.[2] The results of fatigue tests carried out at constant strain over the temperature range 0–100°C. are

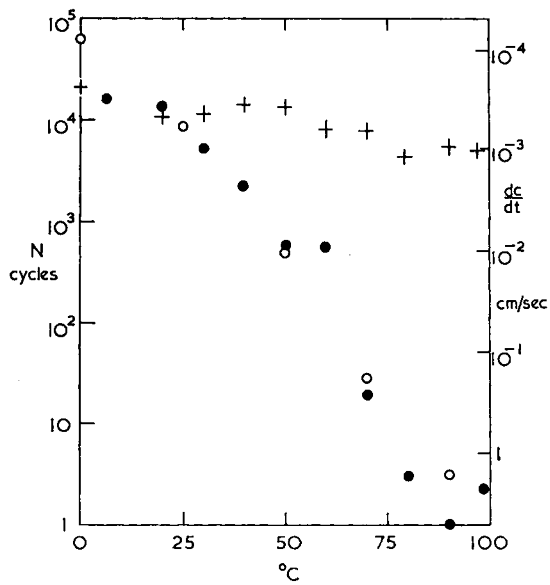

Fig. 11. Effect of temperature on the dynamic fatigue life of dumbbell test pieces at 100 cycles/min.: (●) SBR at 175% maximum strain; (+) natural rubber at 250% maximum strain (left-hand scale); (○) cut growth rates at T = 1 kg./cm. obtained by Greensmith and Thomas[2] on another SBR gum vulcanizate (right-hand scale).

shown in Figure 11. The very large reduction in fatigue life correlates well with the increase in static cut growth rate measured over the same temperature range by Greensmith and Thomas[2] on another SBR gum vulcanizate. In their tests the tearing energy was determined for an imposed rate of cut propagation. Their values of rate for a tearing energy of 1 kg./cm. are fitted to the fatigue life results of Figure 11 at 25°C.

The behavior in fatigue and tearing is very similar, a not unexpected finding in view of the connection between cut growth and fatigue. By means of a rate—temperature transform, Mullins[6] has shown the dependence of tearing on internal viscosity for noncrystallizing SBR. He points out that the transform does not apply when structural changes occur at the tip of a growing tear as, for example, in the case of strain-crystallizing natural rubber. The effect of crystallization can be gauged from the natural rubber results in Figure 11, where the reduction in fatigue life is a factor of 4 over the range 0–100°C., compared with 10,000 for SBR.

Discussion

It has been shown that fatigue of a gum vulcanizate of noncrystallizing SBR, in the absence of heat build-up, can be accounted for in terms of cut growth from small flaws initially present in the rubber. In I the fatigue of crystallizing natural rubber was similarly explained.

Although the same failure mechanism applies to both types of rubber there are important differences between the behavior of the crystallizing and noncrystallizing rubbers examined, as follows: (*1*) static cut growth and fatigue occur in SBR, but do not in natural rubber except at tearing energies close to the catastrophic tearing energy; (*2*) for SBR the cut growth and fatigue behavior changes markedly with temperature, whereas for natural rubber there is very little variation; (*3*) the cut growth rate of SBR is proportional to the fourth power of tearing energy, whereas that of natural rubber is proportional to the square.[1,7]

A plausible explanation for the lack of static cut growth in natural rubber has been advanced by Andrews,[8] who attributes this to the gross hysteresis induced by crystallization, creating a substantially "frozen" strain pattern at the tip of a cut. In dynamic cut growth the relaxation of a test piece to zero strain on each cycle enables the crystallites to melt so that the strain pattern moves forward as the cut grows. For SBR hysteresis is much less, since there is no crystallization, and consequently the strain concentration moves with the growing cut even though the test piece is not relaxed.

The cut growth (and hence fatigue) properties of SBR at different temperatures have been correlated by Mullins[6] using a rate–temperature transformation. This transformation is not applicable to natural rubber owing to the occurrence of crystallization. The ability of natural rubber to crystallize at high strains changes relatively little over the temperature range 10–80°C.,[9] and since the cut growth process is governed by crystallization a substantially constant fatigue life over this temperature range results.

As the rate functions are determined empirically the disparity in the dependence of cut growth rate on tearing energy for the two rubbers cannot be explained at present. However, in view of the difference in the nature of the strain concentration at the tip for crystallizing and noncrystallizing rubbers, it appears not unreasonable to attribute the disparity to the ability or otherwise of a rubber to crystallize under strain.

APPENDIX

Evaluation of F

The fraction F of each cycle for which T^4 may be considered effective is given by

$$F = \frac{\int_0^{t_1} T_t^4 \, dt}{T^4 t_1} \tag{15}$$

where t_1 is the time to complete one cycle.

For the tensile strips, in which $T = 2kWc$, an approximation to F can be evaluated assuming that (*1*) k is a constant and (*2*)

$$W = Ae^{1.5} \tag{16}$$

where A is a constant and e the strain. This is a simplification of the relationship

$$W = 4.64\ e^{1.46}$$

which is a good approximation to the experimental results. It is also assumed that (*3*) e has a sinusoidal relationship with time.

Assumptions *1* and *2* are not valid at strains much smaller than the maximum, but under these conditions T_t^4 is very small compared with T^4.

Two forms of the relation between e and t allow easy evaluation.

(*a*) The test piece relaxes instantaneously only once each cycle. (This is a limiting condition.)

$$e = e_{\mathrm{m}}\,(1 - \cos\theta)/2 \tag{17}$$

where $\theta = 2\pi ft$ and e_{m} is the maximum strain; (*b*) the test piece is relaxed for half of each cycle. (For the majority of tests this may be considered as the other limiting condition.)

$$\begin{aligned} e &= e_m \sin\theta \qquad 0 < \theta < \pi \\ e &= 0 \qquad\qquad \pi < \theta < 2\pi \end{aligned} \tag{18}$$

For case (*a*), combining eqs. (15), (16), and (17) yields

$$F = \frac{\int_0^\pi (1 - \cos\theta)^6\, d\theta}{2^6 \pi} = 0.226$$

For case (*b*), combining eqs. (15), (16), and (18) yields

$$F = \frac{\int_0^{\pi/2} \sin^6\theta\, d\theta}{\pi} = 0.156$$

Taking F as 0.188 (the geometric mean) gives a maximum error of $\pm$ 20%.

Two values of F for the pure shear test pieces can also be obtained. For these test pieces the strain energy W is found experimentally to be approximately proportional to $e^{1.75}$.

For case (*a*) when the test piece relaxes instantaneously only, $F = 0.210$, and for case (*b*) when the test piece is relaxed for half of each cycle, $F = 0.145$.

This work forms part of the research program of the Natural Rubber Producers' Research Association.

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Résumé

Le manque de fatigue à la tension d'une gomme vulcanisé de SBR non cristallisé peut se justifier par la croissance de petites imperfections initialement présentes dans le caoutchouc. Dans la première partie, on a montré que la fatigue du caoutchouc naturel cristallisé est attribuable à la même cause. Des résultats de rupture de croissance sont interprêtés à l'aide de la théorie de l'énergie de rupture de Rivlin et Thomas. Le SBR montre une rupture de croissance à la fois sous des conditions statiques et dynamiques; dans chaque cas, la vitesse est approximativement proportionnelle à la fème puissance de l'énergie de rupture. Une variation de la vitesse de rupture de croissance dynamique avec la fréquence peut s'expliquer par la sommation d'un composant de croissance statique dépendant du temps et d'un composant cyclique semblable à celui se présentant dans le caoutchouc naturel. Le manque de fatigue, sous des conditions à la fois statique et dynamique, est prévu à partir de résultats de rupture de croissance. On a trouvé que ces prévisions se justifient quantitativement pour les temps de fatigue observés expérimentalement lorsqu'une valeur convenable est envisagée pour la dimension de l'imperfection initiale. Les temps de fatigue à diverses températures s'accordent bien avec les résultats de rupture de croissance, obtenus par Greensmith et Thomas dans la même gamme de température. On compare les résultats à ceux obtenus ultérieurement pour le caoutchouc naturel, et on discute les raisons probables de comportement à la fatigue différent des caoutchoucs cristallisés et non-cristallisés.

Zusammenfassung

Die Spannungsermüdung eines ungefüllten Kautschukvulkanisates aus nicht-kristallisierendem SBR kann auf das Wachstum kleiner, anfänglich vorhandener Fehlstellen im Kautschuk zurückgeführt werden. Im Teil I wurde gezeigt, dass die Ermüdung von kristallisierendem Naturkautschuk den gleichen Ursachen zuzuschreiben ist. Ergebnisse bezüglich des Schnittwachstums werden auf Grund der Reissenergie-Theorie von Rivlin und Thomas gedeutet. SBR zeigt Schnittwachstum sowohl unter statischn als auch dynamischen Bedingungen; in beiden Fällen ist die Geschwindigkeit etwa der vierten Potenz der Reissenergie proportional. Die Abhängigkeit der dynamischen Schnittwachstumsgeschwindigkeit von der Frequenz kann durch die Summierung einer zeitabhängigen statischen Wachstumskomponente und einer cyclischen, der in Naturkautschuk auftretenden nicht unähnlichen Komponente wiedergegeben werden. Ermüdungsschäden unter statischen und dynamischen Bedingungen lassen sich aus den Ergebnissen beim Schnittwachstum ableiten. Die experimentell erhaltenen Ermüdungslebensdauern lassen sich so unter Annahme eines geeigneten Wertes für die Anfangsgrösse der Fehlstellen quantitativ wiedergeben. Ermüdungslebensdauern bei verschiedener Temperatur stehen in befriedigender Korrelation zu den Ergebnissen von Greensmith und Thomas bezüglich des Schnittwachstums im gleichen Temperaturbereich. Die Ergebnisse werden mit den früher bei Naturkautschuk erhaltenen verglichen und mögliche Gründe für die Unterschiede im Ermüdungsverhalten von kristallisierenden und nichtkristallisierenden Kautschukarten diskutiert.

Received February 11, 1963