Some Factors Affecting the Service Life of Natural Rubber Articles

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The paper is mainly concerned with failure under repeated stressing (fatigue) and creep behaviour.

Fatigue failure is a crack growth process from small naturally occurring flaws. A critical minimum severity of strain is necessary to produce any mechanical crack growth at all and this governs the 'fatigue limit', the strain below which the fatigue life is very great. Factors which influence this minimum severity include the molecular structure of the vulcanisate, the presence of oxygen and anti-oxidants and the minimum strain during the strain cycle. Ozone attack can be very important in fatigue at low strains.

Creep and stress relaxation are due to a combination of physical and chemical effects. The former is affected by humidity, pre-stressing and the nature of the vulcanising system, whereas the latter depends on the chemical stability of the vulcanisate.

Many factors can influence the useful life of a rubber article in service. It may, for example, become excessively hard or soft or become mechanically weak due to chemical changes. These effects are countered by suitable compounding to improve the chemical stability. The present paper is mainly concerned with two factors which are of particular concern in engineering applications: mechanical failure due to repeated stressing (fatigue), and the creep (or stress relaxation) which occurs under a constant load (or deformation). In both cases, chemical as well as physical processes are important.

FATIGUE FAILURE

Under repeated stressing, rubber components may eventually fail. If the stressing is vigorous and the component is large, failure may be due to excessive heat build-up, as may happen in large tyres at high speed. In this paper, however, cases where the temperature rise itself is a major factor are not being considered. Failure at normal temperatures under repeated stressing is due to the growth of small cracks across the specimen until finally rupture occurs. This is a mode of failure which must be guarded against

in engineering applications as it may be rapid and catastrophic.

As the failure is essentially a crack growth process from small flaws, the crack growth behaviour of the rubber and the size of the initial flaw are the essential factors determining fatigue life. The crack growth characteristics of vulcanised rubbers have now been quite extensively studied (Thomas, 1958; Lake and Lindley, 1964 and 1965). The form in which they have been expressed has been derived from earlier work on tear behaviour (RIVLIN AND THOMAS, 1953; Thomas, 1960). This approach considered the energetics of crack propagation. If a crack of length c in a strained test piece grows by a small amount thus producing an area δA of new surface, the amount of energy (U) in the strained test piece is reduced by δU . This lost energy can be considered as the energy available for driving the crack through the rubber, and a quantity $\delta U/\delta A$ can be defined which can loosely be described as the driving force trying to make the crack grow. This quantity is known as the tearing energy and is denoted by T. It has been shown that T governs the magnitude of the stresses around the tip of the crack (Thomas,

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1955; GREENSMITH et al., 1963), and also determines the amount or rate of crack growth that occurs in the strained test piece (THOMAS, 1958). There are several test pieces for which the relation between T and the applied forces or strains can be calculated, so that, using these, the tear behaviour can be described in terms of the tearing energy. The relation between, say, rate of crack propagation and T is then an intrinsic property of the material and independent of the particular type of test piece used to obtain it. The test piece used for most of the cut growth work is shown in Figure I, and for this the relation for T is

$$T = 2 kcW \qquad \dots (1)$$

where c is the crack length, W the strain energy density in the bulk of the test piece (calculable from the applied stress and the stress-strain curve) and k a slowly varying function of the strain, but which is approximately 2 for most cases (RIVLIN AND THOMAS, 1953; GREEN-SMITH, 1963).

For natural rubber vulcanisates, provided the value of T for catastrophic tearing T_c is not exceeded, there is a negligible amount of purely time dependent cut growth under a constant load. If the test piece is repeatedly stressed and completely relaxed, however, cut growth occurs during each loading cycle. The amount of crack growth depends on the maximum value of T attained each cycle. Thus the crack growth behaviour can be described by the relation between the cut growth per cycle, dc/dn, where n is the number of cycles, and this value of T.

It has been found for natural rubber that the relation (THOMAS, 1958)

$$G \frac{dc}{dn} = T^2 \qquad \dots (2)$$

adequately describes the cut growth behaviour of T values between about 10^5 and 10^6 erg/cm². Using this relation and Equation (1) it is easily shown that the fatigue life N of a test piece cycled in simple extension and initially containing a flaw of size c_0 is given by GENT et al. (1964):

$$N = \frac{G}{(2kW)^2 c_0} \qquad \dots (3)$$

where, as for the test piece of Figure 1, W is the

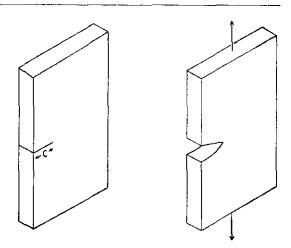


Figure 1. Strip test piece containing edge cut of length c.

strain energy density when the test piece is extended to its maximum.

Equation (3) has been found to represent quite well the dependence of fatigue life on strain over the expected range of applicability of Equation (2). From parallel fatigue and cut growth measurements the size of the initial flaw can be deduced to be about 2.5×10^{-3} cm. The size of these flaws is clearly of great importance in determining the life. Usually, although not invariably, the worst flaws are at the surface and are associated with the preparation of the sample by cutting or moulding. However, particles of dirt are frequently about the above size and have been observed to initiate failure from the body of the material. It seems probable that poorly dispersed compounding ingredients can also act in a similar way.

Natural rubber shows virtually no purely time dependent crack growth under a constant load, as mentioned above. This behaviour appears to be associated with strain-induced crystallisation which also gives it its characteristic high tensile strength. Non-crystallising rubbers such as SBR show time dependent crack growth so that under even a constant load failure may eventually occur.

Equation (2) is not valid at low tearing energies. Figure 2 shows a typical crack growth relation for T values up to about 10^5 erg/cm².

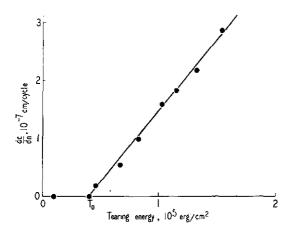


Figure 2. Relation between tearing energy and crack growth rate dc/dn for a natural rubber vulcanisate at low tearing energies. The intercept at T_0 gives the minimum tearing energy for any crack growth to occur.

There is an intercept on the T axis, and below this T value (T_o) there is virtually no crack growth due to mechanical stressing (Lake and Lindley, 1965). Corresponding to T_o , there is a fatigue limit for tensile stressing below which the life is virtually infinite. For the normal flaw sizes this fatigue limit is usually between 50 and 100% tensile strain. Its magnitude is of considerable practical significance as most rubber components subjected to repeated stressing will be designed to work below it. As soon as it is exceeded the fatigue life drops to a few hundred thousand cycles, which is often inadequate.

The magnitude of T_o is governed by the molecular structure of the vulcanisate and the strengths of the chemical bonds, and can be calculated approximately from a fundamental molecular approach (Lake and Thomas, 1967). It is not significantly influenced by mechanical hysteresis or crystallisation in contrast with tensile and tear strength. T_o increases by about a factor of two in vacuo, and some of this increase can be obtained in air by incorporating suitable anti-oxidants such as PBN or certain paraphenylenediamines. Thus oxygen facilitates the rupture of the molecules under stress.

Also, networks containing polysulphide crosslinks have generally higher T_0 values than those containing monosulphide or carbon-carbon cross-links (Lake and Thomas, 1967).

A test piece cycled below its fatigue limit in air may in practice have a finite life due to attack by ozone, which also produces crack growth. Ozone crack growth behaviour is shown schematically in terms of T in Figure 3. Provided a very small T value (T_z) is exceeded the rate of growth is independent of T but proportional to the ozone concentration (AN-DREWS et al., 1963). Presence of chemical antiozonants increases T_z and reduces the growth rate above Tz. Unfortunately under repeated stressing the enhancement of T_z does not occur, due probably to mechanical rupture of a protective surface bloom, but the rate reduction remains. Ozone crack growth is additional to that due directly to mechanical stressing and is, of course, very important at low T values. The crack growth behaviour under the combined action of ozone and mechanical stressing can be represented by

$$\frac{dc}{dn} = \varphi(T) + \frac{K.x(ozone\ concentration)}{(frequency\ of\ cycling)}...(4)$$

where $\varphi(T)$ gives the mechanical crack growth and the second term the ozone crack growth (LAKE AND LINDLEY, 1966). The parameter K

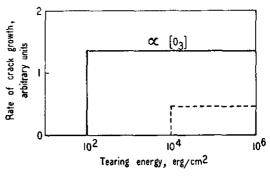


Figure 3. Schematic dependence of crack growth rate due to ozone on tearing energy. The dashed curve shows the effect of adding a chemical anti-ozonant. The rate of crack growth above the critical tearing energy is proportional to the ozone concentration.

depends on the particular vulcanisate and the form of the strain cycle. By using Equation (4), which can be determined by crack growth measurements, instead of Equation (2) the fatigue life in the presence of ozone can be predicted and compared with experiment, as shown in Figure 4. The agreement, using the flaw size deduced from high strain measurements of 2.5×10^{-3} cm, is satisfactory.

In practice, most spring applications do not allow the rubber to be completely relaxed. This can greatly enhance the fatigue life. This enhancement appears to be due to the presence of strain-induced crystallisation at the tip of the

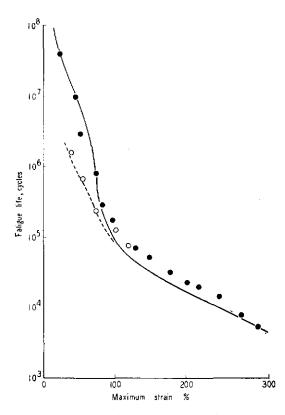


Figure 4. Dependence of fatigue life on strain in the presence of ozone. Curves are theoretical. Upper curve for ozone concentration of 0.3 p.p.h.m, lower (dashed) curve for concentration of 0.8 p.p.h.m. Points are experimental values:

• 0.3 p.p.h.m., ○ 0.8 p.p.h.m.

growing crack which does not disappear on partial relaxation and thus produces an effective strengthening of the material. The effective value of T_0 increases and the rate of crack growth at T values above it decreases. The effect of incomplete relaxation on fatigue is much less in a non-crystallising rubber such as SBR as $Table\ 1$ indicates (LAKE AND LINDLEY, 1966).

TABLE 1. EFFECT OF STRAIN CYCLE ON FATIGUE LIFE

Strain cycle	Fatigue life (kilocycles)		
	NR	SBR	
0-250 50-240	13 13 000	2 5	

Although the above work has been carried out in tension the results are qualitatively similar in other types of deformation such as shear or compression of a bonded block. Failure in these cases is associated with tensile components of the stress which tend to make a crack open and thus encourage its propagation.

There are several factors of importance in ensuring a long life for a component under repeated stressing. If complete relaxation occurs each cycle, as for example in a tyre sidewall, the deformation should not give T values above T_o . The life is then likely to be governed by ozone crack growth or production of large flaws by accidental mechanical damage. If possible, the component should be designed so that incomplete relaxation occurs, and then relatively large alternating deformations can be sustained. Again, ozone attack is likely to be of importance in determining the ultimate life. Incorporation of anti-ozonants is beneficial under static or alternating loads in reducing the ozone crack growth rate. If the component has only intermittent dynamic use an antiozonant may completely suppress growth during the static period.

For components used in compression ozone attack can be much less significant than mea-

surements in tension may suggest. A rubber disc 2 cm diameter and 3 mm thick bonded to metal on its major faces was compressed by 16% and exposed to ozone at 600 parts per million for 25 hours. This is equivalent to about fifty years at normal atmospheric concentrations. Although cracks grew, they penetrated only about 1 mm into the body of the rubber, equivalent to a rate of growth only about $\frac{1}{400}$ of that observed in tension. The reason is probably that the very small opening of the cracks under this strain makes access of ozone difficult. This effect is fortunate as many rubber components for long term applications are used in compression.

CREEP AND STRESS RELAXATION

Under constant load the deformation of a rubber sample increases steadily with time, the phenomenon of creep. At constant deformation relaxation of stress occurs. The two effects are aspects of the same molecular processes and the relation between them is governed by the force-deflection curve. If this is known, one can be calculated from the other (Gent, 1962a). Broadly speaking therefore, compounding and other changes which improve one will improve the other.

In springing and load-bearing applications generally, creep is probably the main disadvantage of rubber as compared with steel, and in severe conditions, such as at elevated temperatures, may limit the useful life. Understanding of the phenomenon, prediction of the amount of creep over long periods and improvements in materials are thus of importance.

There are two components of creep: (i) a physical effect which reflects the internal viscosity, and (ii) chemical effects which are due to cross-link and main chain scission. The physical component contributes a term approximately proportional to the logarithm of the time after loading (GENT, 1962a) and thus gets progressively less important at longer times. The chemical component is approximately proportional to the time, and at very long times becomes the dominating effect (GENT, 1962b). The two components have different variations of rate with temperature so that extrapolation of creep rates to service temperatures is not completely

straightforward. This question will be considered later.

Some of the factors affecting physical creep and stress relaxation will now be considered.

(i) Humidity

It was apparent from stress relaxation measurements carried out at room temperature that the atmospheric humidity was influencing the results appreciably. A systematic study was therefore made. The sample, about 1 mm thick, was immersed in a suitable aqueous solution for at least 20 minutes, rapidly extended while still in the solution and the subsequent stress decay followed. The apparatus was thermostated at 25°C.

Saturated aqueous solutions of various inorganic salts were used giving a range of relative humidities from 15% upwards. Check measurements with air at various humidities showed that the salts themselves did not influence the relaxation and presumably did not enter the rubber. Results for vulcanisates A (a conventional CBS-accelerated sulphur mix given in the Appendix) and B (a peroxide vulcanisate) are shown in Figure 5 for 100% extension. Varying the humidity has two effects:(a) the overall relaxation rate increases with humidity; and (b) the shape of the curves varies systematically, those at high humidity curving upwards. It has been reported that accelerated sulphur vulcanisates similar to this one give linear log(time) relations for physical relaxation (GENT, 1962a). It appears that this is somewhat fortuitous and arises because laboratory humidities are usually in the range 30-60% where this relation holds approximately.

The substantial magnitude of the effect of humidity shows the desirability of control of this variable during testing and also the possible important effect of atmospheric humidity on service performance.

(ii) Vulcanising System

Relaxation rates depend on the degree of cross-linking so that this must be held constant when other compounding variables are considered (GENT AND MASON, 1963). The moduli of the two rubbers shown in *Figure 5* are similar,

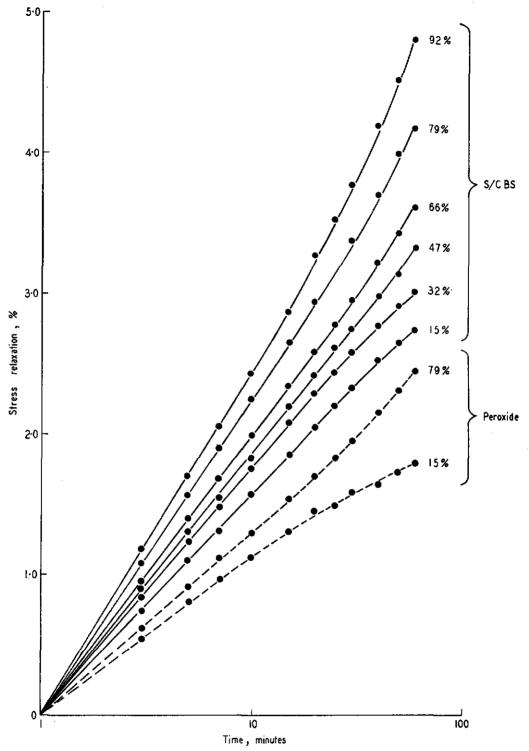


Figure 5. Effect of relative humidity on physical stress relaxation for natural rubber S/CBS and peroxide vulcanisates A and B respectively. The figures by each curve give the relative humidity.

and it can be seen that the overall rate and sensitivity to humidity are lower for the peroxide vulcanisate. Because the shapes of the curves are somewhat different, quantitative comparison is difficult, but arbitrarily taking the relaxation between 1 and 100 minutes the peroxide vulcanisate has a rate about 60% of that of the accelerated sulphur vulcanisate. This effect is present over a range of cross-link densities.

A correct choice of vulcanising system can therefore help in designing a compound with a low physical relaxation rate. Usually a compromise between different properties has to be made, for example, the superior behaviour of a peroxide vulcanisate in creep may have to be balanced against its generally somewhat inferior strength properties.

(iii) Effect of Pre-stressing

It is known that the presence of filler can increase the relaxation rate of natural rubber vulcanisates by a factor of two or more, depending on the nature and loading of the filler. The stiffness of filled vulcanisates can be substantially reduced by pre-stressing and there is some limited published data suggesting that pre-stressing can also influence relaxation rates (Gent, 1962c).

The creep behaviour of vulcanisate C, a CBS-accelerated sulphur mix containing 40 parts per hundred rubber (p.p.h.r.) lampblack, was measured over a range of extensions using fresh samples for each point and taking care not to over-strain them on applying the load.

Under the test conditions quite accurately linear log (time) plots were obtained so that the creep rate could be expressed as a percentage increase in deformation per decade of time, the deformation at one minute being taken as the initial value.

A number of test pieces were then strained to 100% for 1 hour and allowed to recover unstressed for 24 hours so that subsequent recovery was negligible. They were then each tested at a strain in the range 60 to 150%. The results of these two series of tests are shown in *Figure 6*. It is apparent that there is a very substantial reduction in creep rate in the region of the pre-strain value.

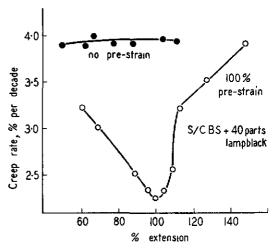


Figure 6. Creep rate as a function of strain for vulcanisate C, containing 40 p.p.h.r. lampblack, for virgin test pieces and ones pre-strained to 100%.

This effect is not solely due to the filler. A corresponding gum rubber was pre-stressed to a similar *stress* to that applied to the black-filled rubber and the above experiments were repeated with the results shown in *Figure 7*.

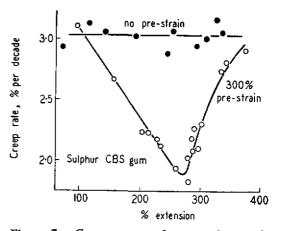


Figure 7. Creep rate as a function of strain for an accelerated sulphur gum vulcanisate similar in composition to the base material of the black-filled vulcanisate C. Results for virgin and perstrained test pieces. The 300% pre-strain entailed a stress similar to that required to prestrain vulcanisate C to 100%.

The pre-strain in this case was 300%. The results are qualitatively remarkably similar to those for the black-filled rubber suggesting that part at least of the effect is due to changes in the gum matrix. A similar suggestion has been made for the stress-softening observed with filled rubbers (HARWOOD et al., 1967).

The effects described above emphasise the difficulties of making valid assessments of creep behaviour. They also suggest that a fundamental molecular interpretation of the relaxation of natural rubber vulcanisates of the types discussed is likely to be difficult.

Chemical relaxation processes, which are relatively important at long times, will be superimposed on the physical component. To estimate the long-term relaxation at service temperatures it is necessary to separate the two components and to examine their temperature dependence individually so that a reasonable extrapolation may be made.

For a vulcanisate whose physical creep is proportional to log (time) the total fractional creep $\triangle e/e_0$ is well described by the equation

$$\frac{\triangle e}{e_0} = A \log \left(\frac{t}{t_0}\right) + B(t - t_0) \dots (5)$$

where t_0 is the time after loading of the first reading, when the strain is e_0 and $\triangle e$ the increase in strain at time t. The agreement with experiment is shown in *Figure 8*. The parameters A and B give respectively the physical and chemical components.

The relative importance of physical and chemical relaxation cannot be readily expressed in terms of their amounts as the amount of physical creep depends strongly on the time of the first measurement after stretching. It is therefore better to compare the two rates of relaxation, and on this basis it appears that for a typical vulcanisate the rates become comparable after one to two years at 10°C.

CONCLUSIONS

It is usually important to make the chance of an engineering component failing by fatigue remote, as such a failure may be rapid and catastrophic. The work described above enumerates some of the relevant factors. The magnitude of the fatigue limit, its variation

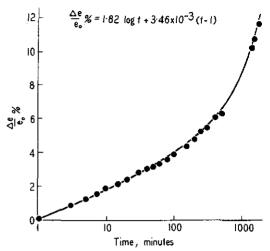


Figure 8. Creep at $70^{\circ}C$ of an accelerated sulphur vulcanisate. Full curve is a relation of the form $\triangle e/e_0 = [A \log (t/t_0) + B(t-t_0)]$ where A and B represent physical and chemical creep components respectively. Their values have been chosen to give the best fit to the experimental points.

with vulcanising system and anti-oxidant, and the benefits of incomplete relaxation of stress during cycling are all important. Attack by ozone is particularly important in tension, but for compressive stresses may be much less so.

Many engineering components, such as engine and anti-vibration mountings and bridge bearings, are used primarily in compression and have relatively small surface to volume ratios. In these cases not only is ozone attack of less concern, but also the attack on the rubber by mineral oils may be unimportant as diffusion may limit the penetration very substantially (SOUTHERN, 1967).

There are also indications that chemical creep due to oxidation, which is often the most important chemical creep process, is reduced in bulky components (CUTHBERT, 1967), probably again due to diffusion effects.

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

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APPENDIX

Item	Vulcanisate		
Item	A	В	C
RSS1 (SMR5)	100	100	100
ZnO `	5		5
Stearic acid	5 2		
Lauric acid	_	-	1
Sulphur	2.5		2.7
CBS	0.6	-	0.54
Nonox ZA	1.5	i _	_
Flectol H	_	1.5	1.5
Lampblack	_	_	40
Dicumyl peroxide	-	3.5	_
Vulcanisation	40' at	10' at	30' at
conditions	140°C	100°C	140°C
		+ 60' at	
	1	150°C	

DISCUSSION

Chairman: Dr. A. A. Watson

Dr. P. Thirion asked about the effect of humidity on stress relaxation and the time of recovery of the free strain. Mr. Thomas replied that the humidity effect was due probably to the non-rubbers rather than the polyisoprene itself, because the diffusion of water into rubber was controlled by the non-rubbers present. Dr. C. E. Scott enquired if similar humidity effects were shown by synthetic eis-polyisoprene whose non-rubber constituents were greatly reduced. Mr. Thomas replied that humidity affected synthetic eis-polyisoprene as well as natural rubber, but comparative data—especially on synthetic—were lacking. Mr. S.W. Sin asked if the effect of humidity

could be due to water acting as a physical plasticiser. Mr. Thomas said that plasticisation often reduced stress relaxation rates; perhaps water softened some components and imparted a stiffness dependent on time, which would contribute to the creep. Mr. Thomas said that work was in progress on EV vulcanisation systems, which Mr. J. Payne suggested would show less creep than the conventional use of sulphur; Dr. J.I. Cunneen added that EV compounds should be better than conventional sulphur vulcanisates in long-term creep where chemical effects were important, but the behaviour of EV systems and conventional vulcanisates were very similar in short-term creep.

Mr. R.A. Billett said that lampblack—mentioned in the paper—was virtually unobtainable outside Britain. Mr. Thomas said lampblack was the material used in the experiments, but any soft non-reinforcing black could be used. Dr. P.M. Sorgo stated that carbon blacks were very specific in their applications and should not be used indiscriminately. However, a black with the desired properties—such as large particles and non-reinforcing—could always be obtained. Dr. A.A. Watson considered that medium thermal black would provide a good replacement if lampblack was not available. He asked if it was known what gave rise to the extra creep. Mr. J. O'Connell commented that lampblack increased stiffness with minimum loading. Mr. Thomas informed Mr. I.K. Yeo that creep behaviour in rubber under compression was under investigation.