

## ***Creep Behaviour of Rubbers Subjected to Repeated Loadings***

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*The creep behaviour of various rubbers has been studied when subjected to repeated loading conditions (cyclic creep), and compared to the creep behaviour under equivalent constant load conditions. It has been found that the repeated loading of strain crystallising rubbers under high stress levels can produce a substantial enhancement of creep rates over that observed in the static loading condition. The presence of crystallisation during stressing is thought to induce high stresses in the rubber network near the boundary region of the crystal sites and that these high stresses lead predominantly to the breakage of crosslinks. It appears from swelling and set measurements that crosslink breakage is responsible for approximately half of the total cyclic creep with visco-elastic effects accounting for the remainder.*

The creep of a rubbery material subjected to repeated loadings would normally be expected to be less than the creep of that rubber under the same load continuously applied. A load applied for half the time, for example, should produce approximately the same amount of creep as half the load applied continuously. This behaviour would be shown by a material which is visco-elastically simple, that is, one obeying approximately the Boltzmann superposition principle for linear visco-elastic systems. Previous work<sup>1,2</sup> have shown that for certain rubber vulcanisates, the creep under repeated stressing can in fact be considerably greater than the creep under a static load. It was suggested that this cyclic creep behaviour was partly due to the ability of these rubbers to strain crystallise. Further work is reported here using a greater range of materials than previously investigated and confirms the importance of crystallisation in producing the observed cyclic creep behaviour. Although the cyclic creep behaviour is most predominant in the tensile mode, the effect is also shown to be present in compression. The contributions of visco-elastic flow and mechanical rupture of bonds during repeated stressing of a vulcanisate are discussed in this paper together with the relative effect these two components have on the overall cyclic creep behaviour.

### EXPERIMENTAL

The tensile creep measurements were conducted on strips of rubber about 100-150 mm long, 6 mm wide and 1-2 mm thick depending on the desired loads and strains. The test pieces were firmly clamped at each end to eliminate slippage and the desired strain (typically 50%-750%) achieved by applying a suitable dead load.

The static creep measurements were conducted by applying a dead load to the test piece. The resulting extension after 1 min of loading is termed the initial extension. The creep is expressed as a percentage of this initial extension. With creep defined in this way, a plot of creep against the logarithm of time is shown to produce a straight line. The creep rate can therefore be quoted as so many percent per decade, that is the amount of creep that occurs during any factor of ten in time.

The cyclic creep measurements were conducted under a cyclic load, that is a load which is repeatedly applied and removed. The loading schedule is illustrated in *Figure 1*. The creep is fairly rapid immediately after the initial loading (*A, C*), but progressively reduces. The creep is measured at the same position during each loading cycle, (e.g. *D*<sub>1</sub>, *D*<sub>2</sub> etc.), when the rate

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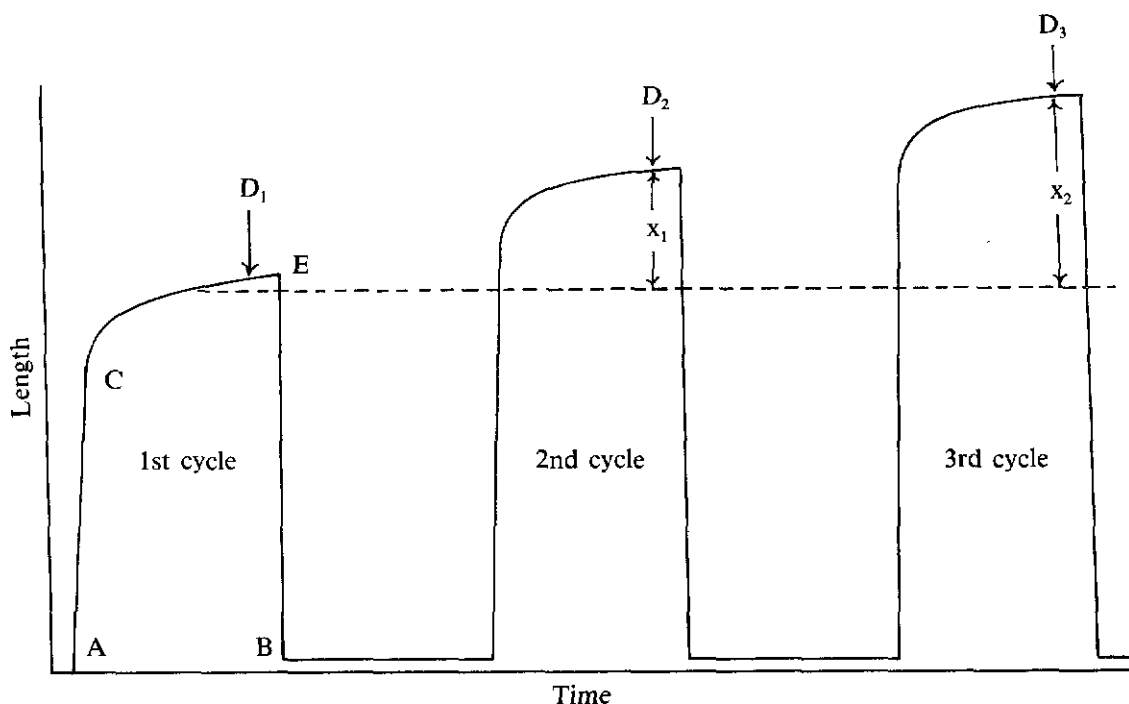


Figure 1. Schematic diagram of length versus time relation showing times at which the increases in length  $x_1$ ,  $x_2$ , etc. are measured.

is sufficiently slow to allow good accuracy in the length measurements. The creep ( $x_1$ ,  $x_2$ , etc.), is calculated as a percentage of the initial displacement which took place in the first cycle (e.g. at Position D).

All the measurements were carried out at a temperature of 22°C except where otherwise stated.

Table 1 shows the formulations of the various rubbers used.

## RESULTS

Since the amount of cyclic creep gives a linear dependence with the logarithm of the number of cycles of loading, and the static creep behaviour a linear dependence with the logarithm of time after loading, the two rates can both be expressed as percentage creep per decade of number of cycles and time respectively. As these rates have similar physical dimensions,

they can legitimately be compared. The absolute values would not be expected to be identical. As previously mentioned, in general, the cyclic rate should be less than the static rate as the load is only applied for a fraction of the time, about half in most of the following experiments. In fact, in many of the cases studied, the cyclic rates are substantially greater than the static ones. The numerical values of the cyclic and static creep rates can be realistically compared since the loading schedule is performed in a regular manner.

Figure 2 shows the static and cyclic creep behaviour of black filled natural rubber using three different vulcanising systems (Compounds A, B, C). It can be seen that the cyclic creep behaviour produces a linear dependence with the logarithm of the number of cycles for each of the vulcanising systems shown. A substantial increase in the magnitude of the cyclic creep rates, due to repeated stressing, over that of the static creep rates can be seen.

TABLE 1. FORMULATIONS OF VARIOUS RUBBERS USED

Item	Compound									
	A Cons	B SEV	C Dicup	D Gum NR	E Gum NR	F SBR	G EPDM	H P.C W	I P.C WRT	
Natural rubber, SMR 5	100	100	100	100	100					
Intol 1500						100				
NPR (WRT)									100	
NPR 4628 (W)								100		
Nordel 1470							100			
Zinc oxide	5	5		3	5	3.5	5	5	5	
Stearic acid	2	2		2	2	2.5	1	0.5	0.5	
CBS	0.6			0.3	0.6	1.1				
Sulphur	2.5	0.3		1.4	2.5	2	1.5			
Dicumyl peroxide			1.5							
Carbon black (Magecol 888)	30	30	30			30				
Nonox ZA									1.0	
Nonox HFN						1				
Flectol H									1.0	
TMTD		1.3							1.0	
MOR		2.75								
HAF							80			
Magnesium D								4	4	
Oktomine								2		
NA22								1	1	
Circosol 4240							55			
TMTM							1.8			
MBT							0.6			
Cure time (min)	60	60	60	30	40	60	35	35	35	
Cure temperature (°C)	140	150	150	140	140	140	150	153	153	
Vulcanisate hardness (IRHD)	50	47	47	43	44	47	50	44	44	

A factor of about 4 is found at this particular strain level (initially 150%), the value depending only slightly upon the vulcanising system used. Thus, the nature of the crosslinks, predominantly monosulphidic in the efficient vulcanising system (EV), and polysulphidic in the conventional sulphur system (CV), appear not to be a major factor in influencing the cyclic creep behaviour at these intermediate strain levels.

Previous work<sup>1,2</sup> have shown that a limited change in the loading schedule of a repeatedly loaded natural rubber gum vulcanisate does not produce a significant change in the cyclic creep rate. Some further measurements over a greater range of loading/unloading times have been conducted, and the results are shown in *Table 2*. The results confirm that the loading schedule can be considerably altered without greatly influencing the cyclic creep rate and that

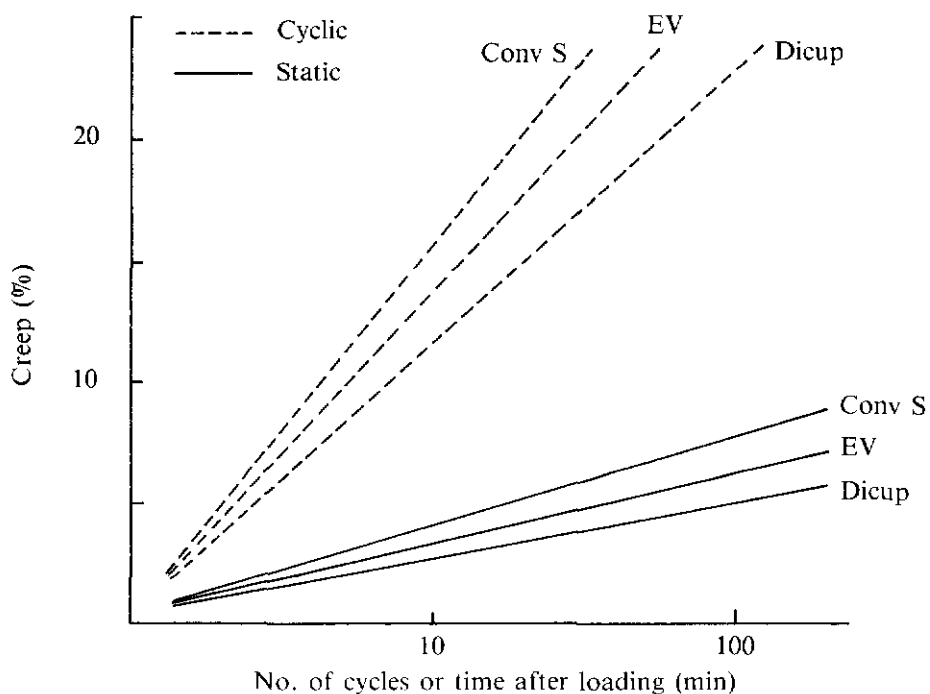


Figure 2. The static and cyclic creep behaviour of three natural rubber vulcanisates containing 30 parts of carbon black filler (Magecol 888). Compound A uses a conventional accelerated sulphur system (Conv S), B a soluble EV system (EV) and C dicumyl peroxide (Dicup). The initial test piece strain is 150%.

TABLE 2. EFFECT OF LOADING SCHEDULE (COMPOUND E)

Time loaded (min)	Time unloaded (min)	Creep (% decade)	Load/unload ratio
Continuous		3.6	
1	1 440	11.9	0.0007
1	10	13.6	0.1
1	1	14.0	1
10	0.16	16.3	60
40	0.16	17.2	240

Initial test strain 350%

Temperature 22°C

it is the process of cycling itself which produces the increased cyclic creep when compared to that of the static creep behaviour. As the unloading period becomes increasingly longer however, the cyclic creep rates can be seen to

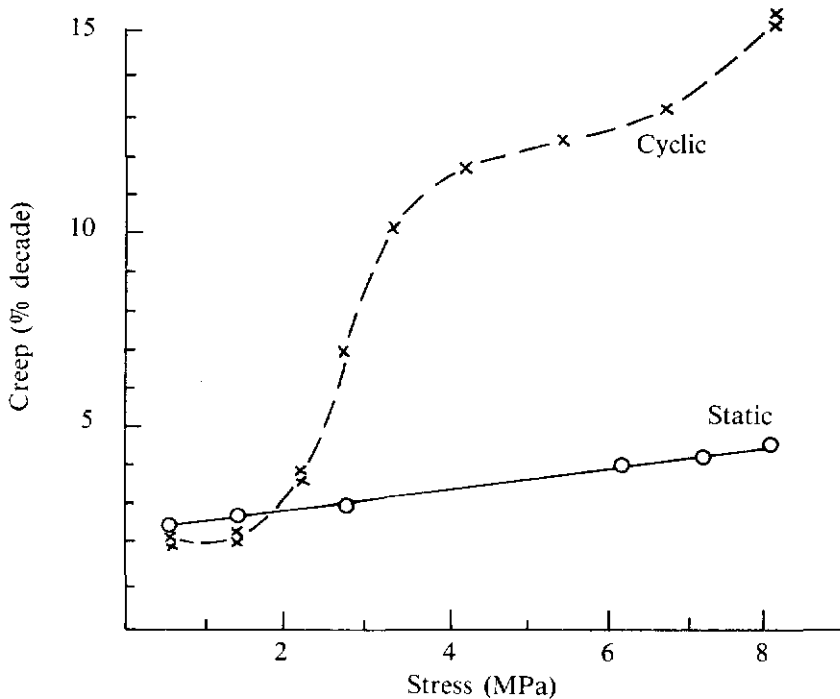
reduce. This reduction in creep is probably due to recovery within the rubber network, the more recovery allowed to take place as the unstrained time period increases, resulting in the reduction of the overall cyclic creep rate.

Earlier work<sup>1</sup> has shown that the cyclic creep of a natural rubber gum vulcanisate can substantially increase with increasing applied stress. Further cyclic creep measurements have been conducted using a range of stress levels (strain 50% to 750%), and some results are shown in *Figure 3*. It can be seen that at low stress levels (below 200% strain) the cyclic creep rate falls below that of the static value, as expected, assuming the material behaves as a linear visco-elastic system, however, as the applied stress increases a substantial increase in the cyclic creep rate takes place. This increase in the cyclic creep rate<sup>1</sup> appeared to reach a plateau, but the results reported here, which were continued to a somewhat higher stress level, indicate the creep rate continues to increase with increase stress.

At the stress levels and subsequent strains associated with the rapid increase in the cyclic creep rate, as shown in *Figure 3*, strain-induced

crystallisation is known to develop significantly. This suggests that the presence of crystallite development and melting during the repeated stressing process may play an important role in producing the observed increase in creep at higher stress levels. Thus subsequent work reported here has further investigated the role of strain crystallisation on the cyclic creep behaviour of various rubbers.

*Figure 4* shows the cyclic creep behaviour of a carbon black filled natural rubber vulcanisate (*Compound A*) repeatedly loaded until failure. The linear dependence of creep with the logarithm of the number of cycles of loading continues until a little before the failure of the sample. During the accelerating creep rate period, and just after the departure from the linear creep behaviour, cracks could be seen developing along the edges of the test pieces. The crack growth development usually took the form of a number of 'smaller' cracks growing



*Figure 3. The effect of increasing stress on the static and cyclic creep rates of a natural rubber gum vulcanisate (Compound E). The initial strain range is 50% to 750%. Similar creep rates occur at approximately 200% strain.*

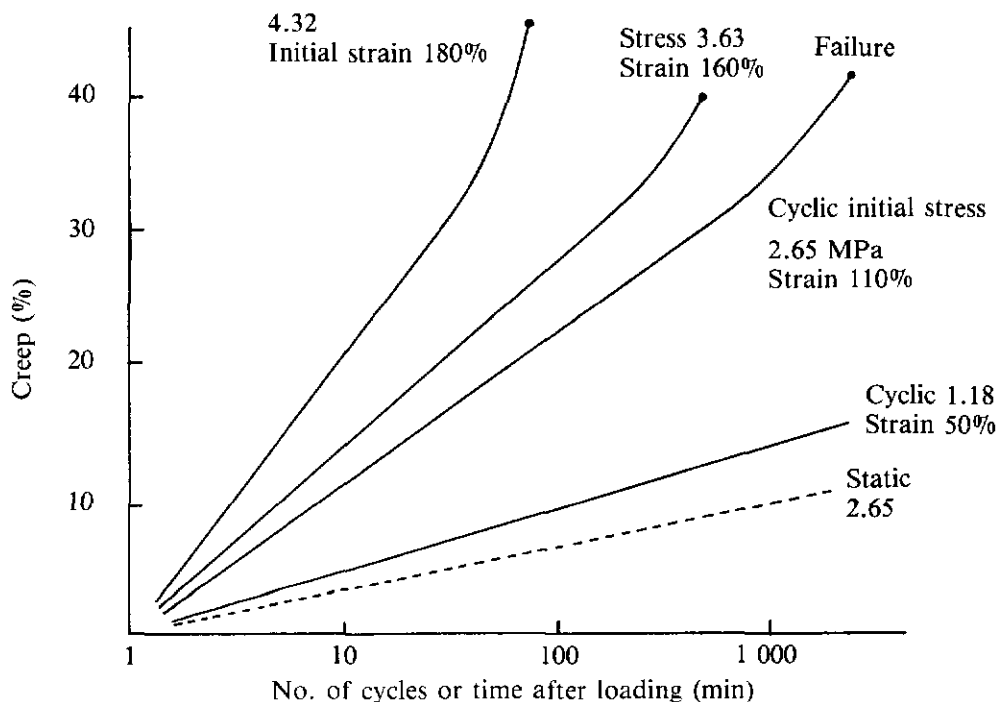


Figure 4. Static and cyclic creep measurements on a natural rubber vulcanisate containing 30 parts of carbon black, Compound A. The limit of time for the static measurements is minutes.

intermittently along the entire length of the test piece with a single dominant crack propagating until it caused the test piece failure.

If strain-induced crystallisation is contributing to the mechanism producing the enhanced cyclic creep behaviour it could be expected that a non-crystallising rubber should produce reduced cyclic creep rates. A 'well behaved' rubber *i.e.*, one following approximately the Boltzmann superposition principle, should produce cyclic creep rates which are lower than the creep rates produced under equivalent static loading conditions.

Figure 5 shows the cyclic and static creep behaviour of two non-crystallising polymers. Generally, the cyclic creep rates of both materials are considerably less than those found in the crystallising rubber, however, it is of interest that the cyclic creep rates are still a little greater than those measured in the static creep condition. Figure 6 shows the cyclic and static creep behaviour of two types of crystal-

lising polychloroprene rubber. The *W* type polychloroprene is known to readily strain crystallise, thus it could be expected that the cyclic creep rates would be considerably greater than the static creep rates if strain crystallisation played a major factor in producing the large creep rates, as found in the strain crystallising natural rubber vulcanisate. It can be seen that the cyclic creep rates are considerably enhanced, even at the moderately elevated test temperature of 35°C, where crystallite formation will be somewhat inhibited and crystallite melting encouraged. The *WRT* type polychloroprene vulcanisate is known to crystallise less readily than the *W* type, and this is reflected in the somewhat reduced cyclic creep rate of the *WRT* material.

It is known that the process of isomerising natural rubber considerably suppresses the development of strain-induced crystallisation<sup>3</sup>. Isomerised natural rubber could, therefore, be expected to act 'well behaved' in the manner of

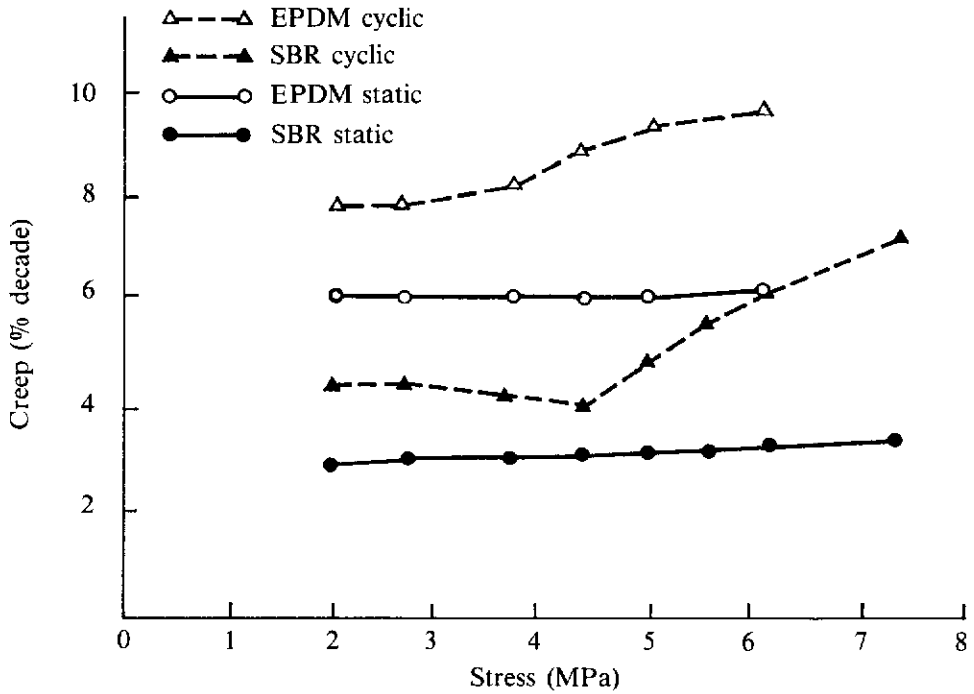


Figure 5. Static and cyclic creep rates of two non-strain crystallising polymers containing carbon black filler. The initial strain range is 110% to 280% for Compound G (EPDM) and 90% to 260% for Compound F (SBR).

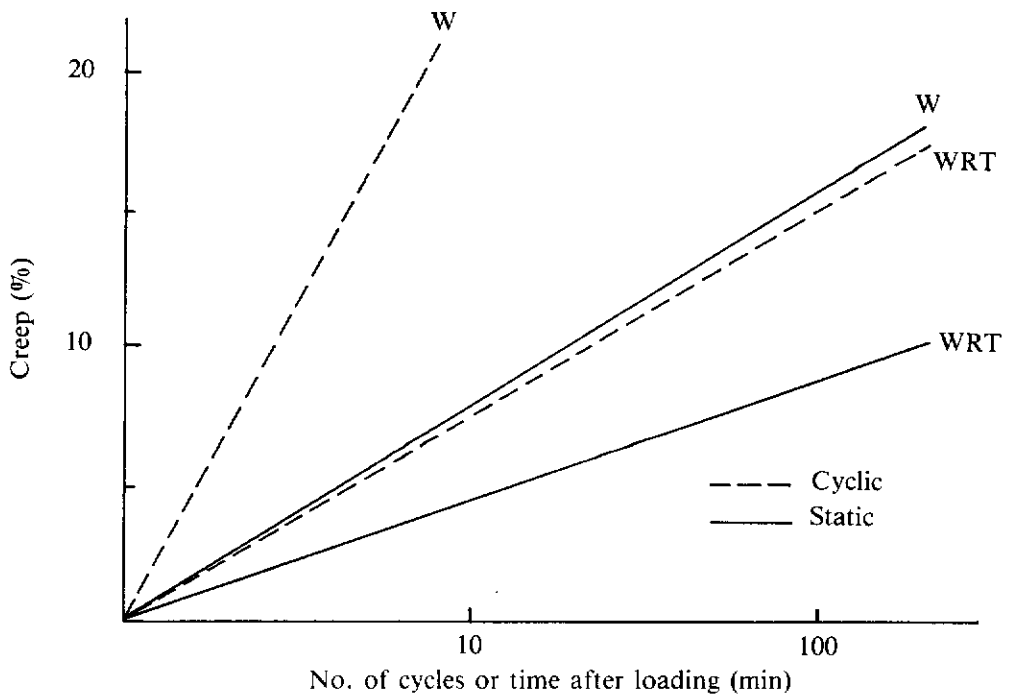
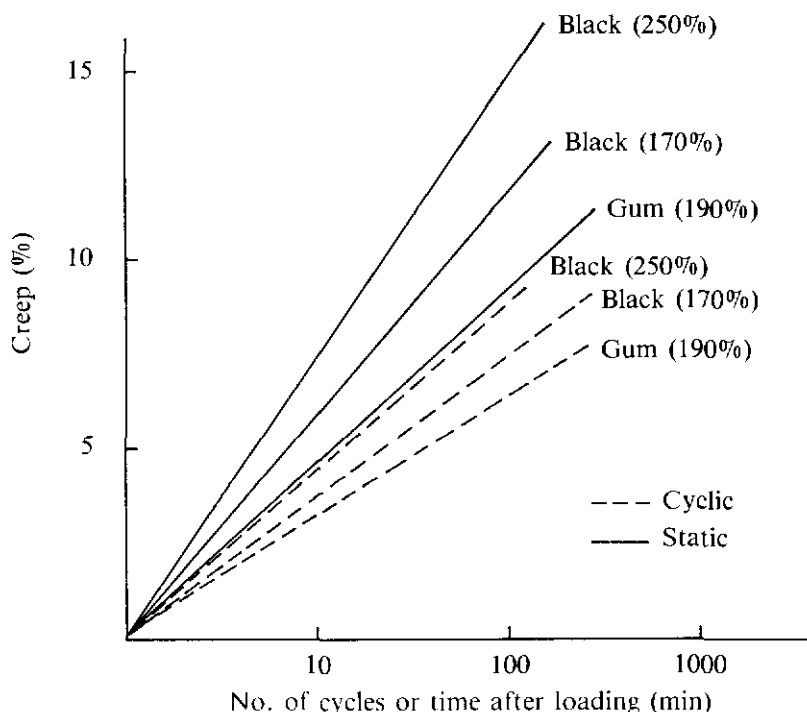


Figure 6. Static and cyclic creep behaviour of two types of strain crystallising polychloroprene rubber. Compound H (W) initial strain 230% and I (WRT) initial strain 240%.

a linear visco-elastic material whereby the cyclic creep rate behaviour should be less than the normal static creep behaviour. Some results are shown in *Figure 7* of isomerised gum and black filled rubbers subjected to cyclic and static loading conditions. From *Figure 7* it can be seen that the materials do show a cyclic creep rate below that of the static creep rate, and therefore behave in the expected well behaved manner, indeed these rubbers are the only materials tested here where the cyclic creep is appreciably less than in the static creep condition. Thus, these results further indicate that the presence of strain-induced crystallisation contributes to the observed increase in creep under repeated loading conditions.

The work described so far has been confined to creep behaviour in tension, because this mode is where the effect of cyclic creep would be expected to be most prominent and therefore easiest to study. However, the cyclic creep

behaviour would also be expected to be present under compression and shear deformations although to a lesser extent. Since in practice the majority of manufactured rubber articles will operate in some combination of compression and shear modes, it is of interest to know the degree by which the enhanced cyclic creep rate behaviour measured in tension occurs in these other modes of deformation. Some results of the stress relaxation of a natural rubber vulcanisate with two carbon black filler loadings is shown in *Figure 8*, as a function of the applied compression. It can be seen that at smaller compressive strains (less than 30%), the cyclic stress relaxation is less than that of the ordinary static conditions but as the compressive displacement increases the cyclic creep becomes greater, a behaviour similar to that found in tension. The particular value of compressive strain when the cyclic creep rate becomes greater than the static creep rate will depend on the filler loading. Since the filler



*Figure 7. Static and cyclic creep behaviour of two isomerised natural rubber vulcanisates. The filled material contains 30 parts HAF carbon black. Figures within brackets indicate initial strain.*



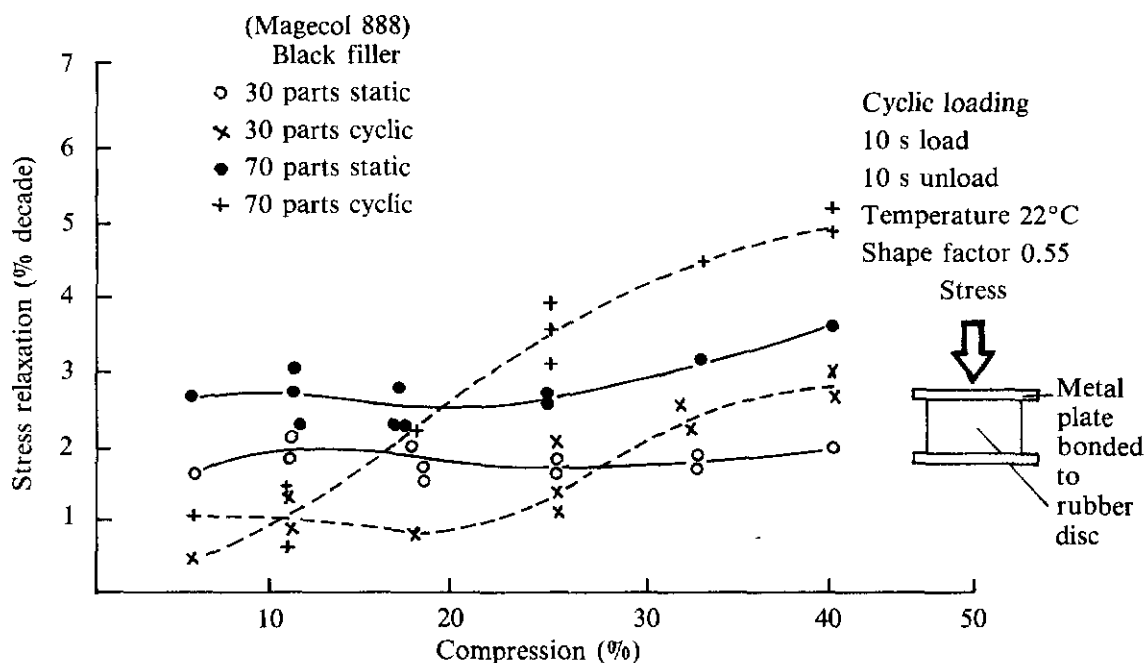


Figure 8. Static and cyclic stress relaxation of plate bonded natural rubber discs in compression. Compound E plus 30 or 70 parts carbon black filler.

loading determines the proportions of the elastic rubber phase to the inelastic carbon black phase 'strain amplification' effects will influence the cyclic creep rate values.

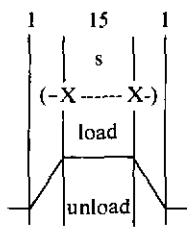
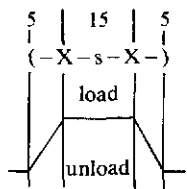
### Set and Bond Breakage on Repeated Stressing

Ordinary creep at room temperature is regarded as a visco-elastic mechanism. Previous work using natural rubber has shown that set, resulting from ordinary creep is recoverable by swelling and deswelling the rubber in a good solvent. Measurements of cyclic creep have however shown that there is considerable permanent set, that is set that cannot be recovered even by swelling and deswelling. The presence of this permanent set suggests that there is some form of network breakdown occurring due to the repeated stressing applied to the rubber. Table 3 shows an example of the permanent set on a gum natural rubber worked for twenty cycles as a function of the applied stress. At the lowest strain shown, which is a little below the strain level when strain-induced

crystallisation is known to become significant (approximately 200%), little permanent set occurs, however, as the strain increases the permanent set likewise increases.

Previous work has shown that high stress, even at room temperature can produce rupture of chemical bonds, probably mainly crosslinks, in natural rubber vulcanisates<sup>4</sup>. Some recombination of crosslinks occurs while the rubber is in the strained state, and this leads to changes in the equilibrium swelling of rubber and the observed permanent set. Swelling techniques can in fact be used to detect the breakdown of bonds in a quantitative manner, however, sample weighing and length measurements normally require a high level of accuracy. The question concerning the cyclic creep behaviour is whether cyclic creep is related to the reduction in the number of molecular chains produced by stress-induced bond breakage, or whether the cyclic creep effect is predominantly visco-elastic and hence recoverable with swelling and drying, as in the static creep case, and not determined

TABLE 3. SET OF A GUM NATURAL RUBBER (COMPOUND E)

Number of cycles	Load/unload schedule (s)	Test stress (MPa)	Test strain (%)	Permanent set due to stressing (% of original length)	Total set at end of cycling (%)
20	15/15	1.32	170	0.1	2.2
20	15/15	2.65	360	1.3	7.1
20	15/15	5.30	580	7.4	17.0
20	15/15	6.62	640	11.3	18.2
20	15/15	7.95	750	17.4	24.6
20		6.62	640	10.3	18.1
20		6.62	640	11.3	19.6

by any irreversible effects. Thus to investigate this, a series of measurements were carried out in which strips of gum natural rubber *D* and *E* were repeatedly stressed, the cyclic creep being monitored in the usual way. Portions of the test pieces were cut and their lengths and weights accurately measured and then swollen to equilibrium in n-decane. The changes in equilibrium swelling are normally quite small, of the order of a few percent in the moderately stressed test pieces, so that good accuracy is necessary in these measurements. Generally, there is some extractable material present in the test pieces and therefore each sample was swollen in a container with an unstressed control sample cut adjacently from the same sheet. This procedure also allowed for any inhomogeneity in the rubber sheet. The swollen samples were weighed, and the decane evaporated off, after which the sample dimensions and final weights

were measured and allowances made for any extractable material. These measurements were used to calculate the equilibrium volume fractions of rubber in the control and stressed samples  $V_1$ , and  $V_2$ , and the fractional permanent set  $(\lambda_0 - 1)$ . During the repeated stressing process some recombination of crosslinks takes place which leads to the observed permanent set discussed earlier. This crosslink recombination produces an additional complication when interpreting the swelling measurement results, however, assuming the Tobolsky two-network theory, the fraction of bonds broken and the fractional recombination of crosslinks can be calculated<sup>2,4</sup>.

Assuming there are initially  $N$  network chains per unit volume of rubber of which  $n_1$  break and  $n_2$  recombine during the strained state

at extension ratio  $\lambda'$ ,  $\frac{n_2}{N-n_1}$  ( $= \alpha$ ) is

given by

$$\alpha = \frac{\lambda_0^3 - 1}{\lambda' - \lambda_0^3 \lambda'^{-2}} \quad \dots 1$$

and

$$1 - \frac{n_1}{N} = \left( \frac{v_1}{v_2} \right)^{1/3} \left( \frac{\ln(1-v_2) + v_2 + \chi v_2^2}{\ln(1-v_1) + v_1 + \chi v_1^2} \right)$$

$$\left[ \left( 1 + \alpha \lambda' \right)^2 \left( 1 + \frac{\alpha}{\lambda'^2} \right) \right]^{-1/3} \quad \dots 2$$

These equations make use of the Flory-Huggins relation between modulus and equilibrium swelling using a value of  $\chi = 0.41$ . Figure 9 shows the percentage of broken crosslinks calculated from Equations 1 and 2, plotted against the percentage creep of a natural

rubber gum vulcanisate. The results include data on test pieces repeatedly loaded for twenty cycles at various stress levels, test pieces held at a fixed stress level of 1.77 MPa and loaded over various numbers of cycles, and test pieces which have undergone the various loading schedules shown in Table 4. From Figure 9, the number of broken crosslinks can be seen to correlate with the amount of creep taking place in the rubber. The number of crosslinks which recombine during the strained state has also been calculated for the test conditions discussed and results indicate that between 20% and 30% of these broken crosslinks recombine in the natural rubber vulcanisate studied.

DISCUSSION

Evidence suggests that the presence of strain-induced crystallisation within a rubber can produce an increase in creep under cyclic loading conditions.

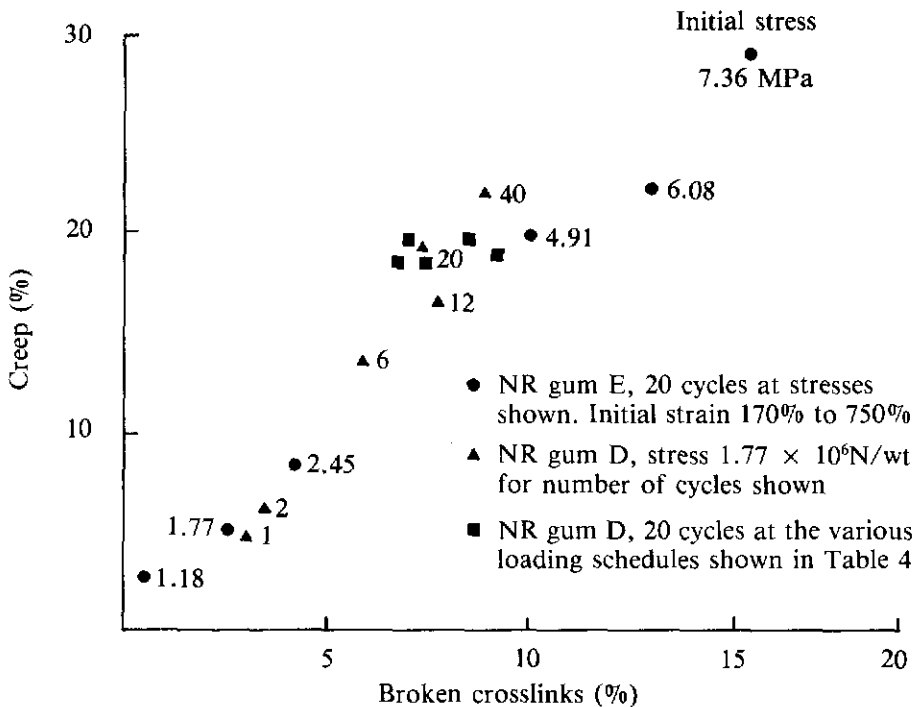


Figure 9. Percent creep variation with percent broken crosslinks deduced from set and swelling measurements of two natural rubber gum vulcanisates (E and D).

TABLE 4. NATURAL RUBBER GUM (COMPOUND D), INITIAL STRAIN 550%

Time under load (s)	Time unloaded (s)	Cyclic creep rate (% per decade)
30	15	12.3
5	5	13.4
5	20	12.2
15	15	12.4
20	5	12.5

The varying quantities of crystallisation present in the three crystallising rubbers discussed, natural rubber and polychloroprene types *W* and *WRT*, appear to influence the degree by which the cyclic creep rate behaviour is enhanced over that of the static creep rates. The greater degree of strain-induced crystallisation present in the rubber, the greater becomes the cyclic creep rate. The much smaller cyclic creep shown by the normally non-crystallising SBR, EPDM and isomerised natural rubber suggests that the absence of significant strain-induced crystallisation produces cyclic creep rates of a magnitude similar to those of the static creep condition. The SBR and EPDM rubbers still, however, show cyclic creep rates somewhat greater than in the static condition. This may indicate that there is something akin to crystallisation taking place even in these rubbers to a limited extent. Some evidence of this has been found in stretched SBR using X-ray technique<sup>5</sup>. The cut growth behaviour of SBR can also show a dependence with the number of cycles of deformation rather than the time under load<sup>6</sup>, somewhat like a strain crystallising rubber but much less pronounced. This behaviour also further suggests that there may be a modest amount of crystallisation or crystal-like development taking place during the stressing of SBR.

The results of the swelling and set measurements indicate that part of the cyclic creep phenomenon, due to repeated stressing, can be ascribed to the breakage of crosslinks or equivalent primary bonds. It can be seen from Figure 9 that the percentage creep is numerically

greater than the percentage of broken crosslinks, in fact it is about double. If broken crosslinks were the only mechanism causing cyclic creep, it could be expected that the percentage broken crosslinks, and the percentage creep would be approximately equal. The values obtained in Figure 9 suggest that about half of the creep can be explained in terms of bond scission. The set measurements also confirm this conclusion where of the set at the end of a period of cycling, about half can be recovered by swelling and deswelling. The cyclic creep behaviour, therefore, appears to have two components. One is a non-recoverable creep which is responsible for a little over half the total creep and crosslink breakage is probably the main contributor. The second creep component is recoverable creep, this is probably associated with the normal rearrangement of the rubber network associated with the slippage of chain entanglements *etc.*, during stressing.

Although the results suggest that the mechanism causing the cyclic creep behaviour involves crystallisation, the exact mechanism is not known. A possible explanation of this cyclic creep mechanism is that rubber chains held around the perimeter of the crystallite sites are severely strained when a load is applied, and some failure may occur. Then as further regions of the rubber become crystalline and the crystallites melt and reform in different positions, due to the repeated loading process, further failure occurs around the new crystallite sites leading to the progressive creep behaviour taking place during each loading cycle.

The results indicate that for a gum rubber that does not significantly strain crystallise during deformation, which for a natural rubber under a tensile deformation is normally at strains less than approximately 200%, the cyclic creep rate will be smaller than the static rate and the creep is almost entirely visco-elastic and thus recoverable in time. At strains greater than about 200%, the non-recoverable creep component, caused by bond breakage, becomes increasingly important. In the gum natural rubber vulcanisate (Compound E), for example, approximately half of the total set is non-recoverable at a strain of 600%.

The inclusion of carbon black filler in a rubber has the effect of increasing the cyclic creep rate compared with the gum material when the rubber phase of the vulcanisate is cycled at a similar strain. Thus, the rubber-filler or the filler-filler interaction plays an important role in determining creep rates during repeated deformations. The repeatedly loaded carbon black filled non-crystallising rubbers (*Compounds F and G*) show approximately 50% increase in creep, due to cycling, even at modest strains. This observation suggests that some bond breakage is occurring possibly due to local stress concentrations caused by the introduction of filler. The still larger increase in creep, due to cycling, shown by the filled strain crystallising rubbers suggests that further enhancement of stress concentrations are occurring. This may be because of strain-induced crystallisation developing at relatively modest bulk strains, due to the stress concentration effect of filler, thus causing bond breakage by the mechanism already described.

Although the majority of the cyclic creep work described here has been confined to tensile deformation, the stress relaxation study in compression does indicate that the enhanced cyclic creep can still be present in the compression mode although to a lesser extent. The effect will almost certainly be present in shear deformation, probably to an extent somewhere between that of the tension and compression modes of deformation. Since many engineering components are subjected to cyclic loadings it is clearly important to be able to predict the creep that will occur under these conditions. The large amounts of creep occurring in some of the tension results reported here, will not normally be found in the majority of practical applications of rubber components because stress/strain levels are normally very much lower than those used here, and tensile deformations are normally avoided by design. In bridge bearings, for example, tensile strains occurring during shearing of a bearing, do not normally exceed 50% and often are considerably smaller. Thus the practical range of strains is generally small and the reported creep phenomena would not normally be a practical problem. However, the work indicates the increase in creep that can

occur if rubbers are used beyond normal operating stress and strain levels.

The presence of strain-induced crystallisation in a rubber, although producing increased cyclic creep rates at high strains, imparts other beneficial properties. In particular, these include excellent strength and cut growth characteristics. As is often the case, a good balance of properties is of more value to the engineer than outstanding performance in one particular aspect.

The phenomenon of the enhanced cyclic creep, and subsequent failure, in repeatedly loaded crystallising rubbers may occur in practice, however, in certain specific applications. An example of one important application may be in motor vehicle tyres, where the rubber can be locally repeatedly deformed to considerable strains upon contact with road asperities during each rotation of the wheel. The crosslink breakage and subsequent cyclic creep and failure which may take place under this repeated loading condition could partly account for the wear characteristics of crystallising rubbers not being correspondingly related to their strength properties.

#### CONCLUSIONS

The repeated loading of rubber samples can produce an increase in creep when compared to the creep occurring under a static load. The presence of strain-induced crystallisation is thought to produce stress concentrations in the rubber leading to breaking of crosslinks during repeated loading. While the general phenomenon of enhanced creep due to repeated loading has been known for sometime, the present investigation has led to new results suggesting that at high strains crosslink breakage is responsible for approximately half of the total creep with visco-elastic effects accounting for the remainder.

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