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Some Effects of Anaerobic Ageing, Stress Softening and Recovery on the Modulus and Damping of Certain NR Vulcanisates for Anti-seismic Bearings

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Data are shown which quantifies and helps to predict some effects of anaerobic ageing and repeated shear deformations on the modulus and damping properties of selected high damping NR vulcanisates. For such vulcanisates the results show that at a given ageing temperature the increase in modulus associated with anaerobic ageing can be reasonably accurately predicted when plotted as a function of the square root of the ageing time. Measurements showing the influence of different test strains and the number of cyclic deformations at a constant stain on the modulus and damping properties of these vulcanisates are also given. Some cumulative effects of anaerobic ageing, stress softening and recovery on the modulus and damping properties during certain applied strain histories relevant to anti-seismic bearings are also shown. Such results indicate that stress softening can have the dominant effect on the modulus of a vulcanisate at small strains whereas at larger strains anaerobic ageing can be the dominant factor

Much data has been published on the effects of accelerated aerobic ageing, and to a somewhat lesser extent anaerobic ageing, on the properties of NR vulcanisates which are relevant to anti-seismic bearing applications[1–10]. These measurements often include data following a single period of ageing with the vulcanisate in a static unstrained condition. For such a condition a key property like the modulus of the rubber will tend to increase with aerobic and anaerobic ageing. Measurements of the effects of anaerobic ageing on the properties of vulcanisates which have experienced a more complex anaerobic strain history can also be worthwhile since they may produce data more representative of actual service conditions. A difficulty with any such ageing measurements is that in order to obtain dynamic data the material will inevitably be deformed during testing which introduces a further complication, that of stress softening. For gum rubber vulcanisates or lightly filled materials the effect of stress softening can be quite small and may not pose a significant problem when measuring physical properties. For more highly filled vulcanisates, *e.g.* one containing a significant quantity of carbon black filler this is not the case and in such compounds stress softening and recovery can have a considerable effect on properties such as modulus and damping[11–14]. Anaerobic ageing and stress softening normally have opposing effects on the modulus of a vulcanisate where anaerobic ageing will tend to increase the modulus but repeated stressing will tend to

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reduce it. These two opposing effects may result in an overall stiffening or a softening of the material depending upon the particular ageing conditions and strain history applied to the material. The degree of stress softening of a vulcanisate is influenced by many factors which include the vulcanising ingredients, cure conditions and the quantity and type of filler, as well as the strain schedule applied to the material. Because so many factors may influence stress softening it has not been possible to mathematically model stress softening in order to predict its effect on physical properties such as modulus and damping, and accurate data can normally only be obtained by testing particular vulcanisates under specific conditions. Studies of the effects of anaerobic ageing on the properties of certain high damping NR vulcanisates used in anti-seismic bearings have also shown that at slightly elevated temperatures the increase in modulus of the material, associated with anaerobic ageing, does not normally follow an Arrhenius type relationship, and the equivalence between the accelerated data and the changes expected in service could not be made with certainty[15]. Thus accurate long-term predictions of the stiffening of such vulcanisates due to ageing still remains a problem.

High damping NR vulcanisates are used in components such as bearings for the base isolation of structures in order to minimise earthquake damage[16–17]. It is important that in critical dynamic applications such as base isolation bearings the vulcanisate does not stiffen unacceptably with time due to ageing. Base isolation bearings are normally designed to have a non-linear stiffness in the horizontal direction. A high stiffness is desirable at small strains to keep wind sway of a structure to a minimum, while a lower stiffness is required at a larger strain to uncouple the earthquake energy. The non-linearity can be achieved intrinsically *via* the natural rubber formulation. Such a vulcanisate should also possess a relatively high degree of damping so as to avoid any resonance behaviour.

In service an anti-seismic bearing can be expected to experience a number of periods where the vulcanisate is not subjected to any significant cyclic shear strain and during this time predominantly anaerobic ageing will tend to increase the modulus of the vulcanisate and reduce the damping. Such periods are likely to make up a considerable part of the working life of a bearing. Due to wind loadings on the supported structure an isolation bearing may also experience intermittent small strain cyclic deformations where stress softening will take place. Depending on local conditions the number of small strain deformations could be quite large over the working life of a bearing but these periods of cyclic activity will intercalate with quiescent periods when time-dependent recovery of the small strain modulus and damping will occur. In the event of an earthquake an anti-seismic bearing would also be expected to experience small numbers of large cyclic deformations where significant stress softening may occur followed by time-dependent recovery. For anti-seismic bearings it is clearly important to have some understanding of how the opposing effects of anaerobic ageing and stress softening with subsequent recovery will influence the modulus and damping of a vulcanisate which experiences such service conditions. However, little information is currently available. A difficulty with producing any such data is that the exact sequence of events and strain history experience by a bearing will differ for each supported structure which precludes a standardised testing procedure which will accurately simulate service conditions. Data based on a simplified sequence of events can, however, be produced which will at least provide an indication of the effects of such complex strain histories on the physical properties of a vulcanisate and may approximately simulate certain service events Currently there is a lack of such data

This paper reports an investigation on the effect of anaerobic ageing and stress softening on the modulus and damping of two high-damping NR vulcanisates which have been used in anti-seismic bearings Data are given which show the changes and also aids the prediction of the modulus and damping under certain static and dynamic conditions The paper shows data after a single period of anaerobic ageing at a moderately elevated temperature in a static unstrained state and also for more complex situations which include some sequence of events that simulate a simplified strain history akin to that which could occur in an anti-seismic bearing in service

Procedure

All the measurements were carried out on double shear testpieces which consist of two cylindrical layers of rubber 25 mm in diameter and approximately 6 mm in thickness bonded between three mild steel cylinders with a proprietary bonding agent[18] The testpieces were manufactured by hot moulding in a press for 35 min at a temperature of 145°C Two types of testpiece were manufactured, one with a hard (58 IRHD) vulcanisate (Compound 1) the other with a softer (46 IRHD) vulcanisate (Compound 2) Compound 1 contained 60 p p h r and Compound 2, 45 p p h r of N220 carbon black filler Both materials were high-damping natural rubber vulcanisates All the dynamic shear modulus and damping data was obtained at a frequency of 0 5 Hz and at a test temperature of 23°C The testpieces were subjected to different periods of anaerobic ageing in a static unstrained state For dynamic testing the testpieces were removed from the anaerobic ageing state and placed in a suitable test machine under aerobic conditions Test-pieces which were subjected to a further period of anaerobic ageing, following the dynamic testing, therefore experienced a short period (approximately 1 h) in an aerobic environment at a temperature of 23°C The ageing conditions and strain histories applied to each testpiece are described under the following headings for each particular test schedule

RESULTS AND DISCUSSION

Anaerobic Ageing in a Static Unstrained State

Single test strain It is important for long life applications such as anti-seismic bearings, anti-vibration mounts and bridge bearings that the modulus of the rubber does not alter by an unacceptable amount as a result of anaerobic ageing For large components anaerobic ageing has the dominant effect on the modulus, with oxidative ageing being confined to a surface layer which at ambient temperature (20°C) is of the order of 25 mm – 30 mm[4,9] Anaerobic ageing increases the modulus of a vulcanisate by post-vulcanisation crosslinking reactions A common method of predicting long-term changes in the properties of rubber is to extra-polate results from short-term tests at elevated temperatures Properties such as tensile strength and surface embrittlement may be predicted by an Arrhenius plot, however, correlations of the modulus can be less satisfactory for anaerobic ageing conditions

Figures 1 and *2* show the increase in the modulus of Compounds 1 and 2, respectively after anaerobic ageing at temperatures of 70°C, 60°C and 50°C for different periods up to 450 days The testpieces were all unaged virgin samples at the beginning of each test and remained in an unstrained static condition during ageing Following each period of ageing

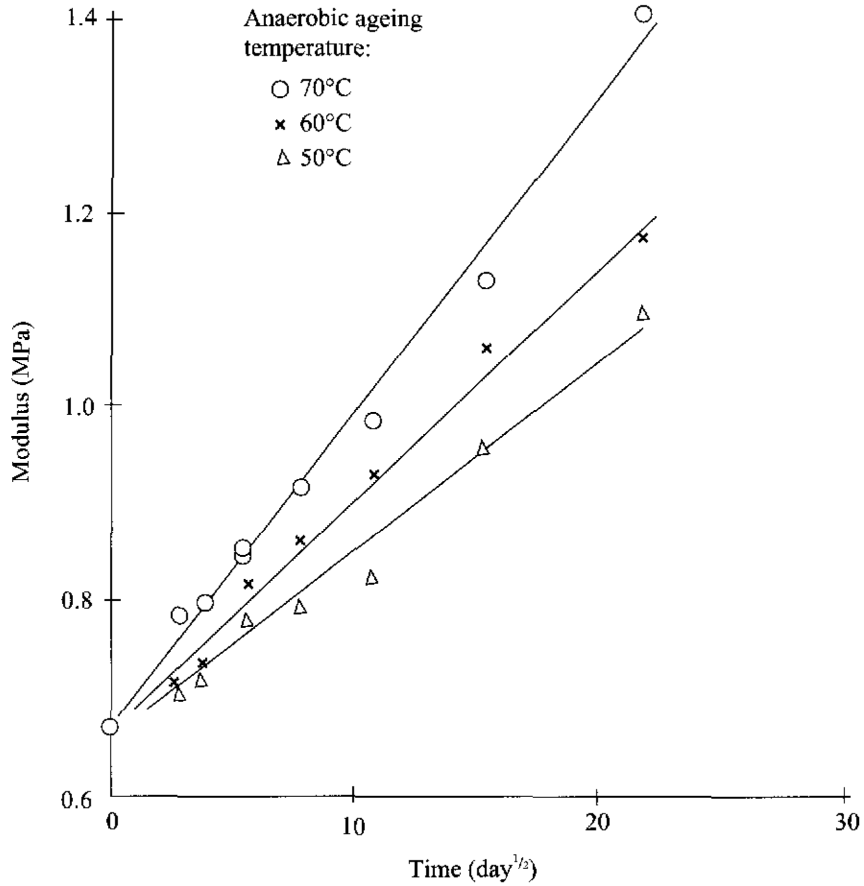

Figure 1. The relationship between the shear modulus and time of anaerobic ageing at temperatures of 50°C, 60°C and 70°C. Compound 1, strain 100%.

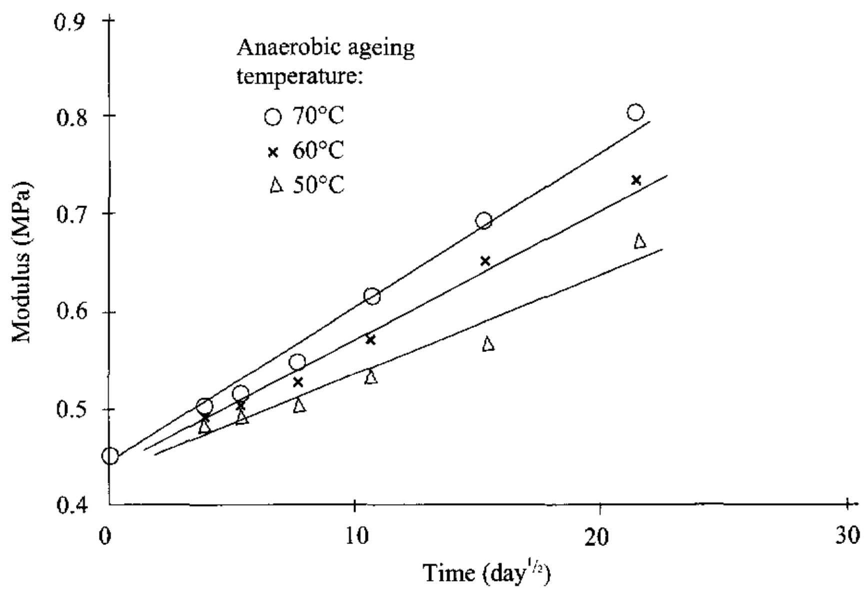

Figure 2. The relationship between the shear modulus and time of anaerobic ageing at temperatures of 50°C, 60°C and 70°C. Compound 2, strain 100%.

modulus data were obtained at a strain of 100% and at a temperature of 23°C. The data shown are for the third cycle. It can be seen that the modulus increases at approximately a constant rate with the square root of the ageing time and that the rate varies with the ageing temperature. Such a plot can provide a reasonably accurate prediction of the increase in modulus caused by anaerobic ageing at the test temperature and may particularly aid tests conducted near ambient temperature, which without a method of extrapolating the data, normally require a period of testing which is impractically long. If the change in the modulus of a vulcanisate caused by ageing is expressed as a percentage increase over that of the unaged modulus per day$^{1/2}$, at an ageing temperature of 70°C, 60°C and 50°C values of 4.93, 3.58 and 2.91 are obtained for Compound 1 and values of 3.55, 2.89 and 2.11 for Compound 2.

Incremental Test Strain

Following a period of anaerobic ageing in an unstrained state the increase in the modulus and the fall in the damping of a vulcanisate were found to alter with the test strain. *Figures 3* and *4* show the percentage change in the modulus and damping of anaerobically aged vulcanisates compared with that of unaged material *versus* the shear strain. Each virgin testpiece was initially cycled three times at the smallest strain of 2%. The strain was then increased in increments up to 100%. At each strain the testpieces were cycled three times only. The data shown on the graphs are for the third cycle. The behaviour of the curves shown in *Figures 3* and *4* would indicate that two separate mechanisms are taking place one being dominant at small strains, the other at strains greater than approximately 25%.

The increase in the modulus at larger strains is probably associated with additional crosslink formation during anaerobic ageing. Further crosslinks will lead to a reduction in the average length of the polymer chains between crosslinks so that these chain segments will be fully extended at a lower strain than in unaged material, thus increasing the modulus[2]. During anaerobic ageing when a vulcanisate is in a static state further interactions may occur between the carbon black and the rubber and also between the carbon black structure itself. Such interactions may form carbon gel and hydrogen bonds which are relatively weak mechanically when compared to crosslink bonds[1]. The increase in the modulus at small strains shown in *Figures 3* and *4* is attributed to such changes. The application of a successively greater strain on a testpiece would tend to progressively break more and more of these weak bonds which would cause a fall in the modulus of the material. The results indicate that most of these bonds have broken at a strain of about 20%. It is normally desirable to maintain a large stiffness at small strains in an anti-seismic bearing to minimise wind sway of a structure. It is therefore of interest to determine if these bonds may reform and thus produce a recovery in the small strain stiffness of the material if a vulcanisate remains in a static state for a sufficient period of time. Some further measurements to investigate this aspect are discussed later in this paper.

Stress Softening

Constant strain. As mentioned earlier stress softening can have a significant effect on the modulus and damping of a vulcanisate particularly if the material contains a substantial quantity of carbon black filler. *Figures 5* and *6* show how stress softening alters the modulus and damping of compounds 1 and 2, respectively when cycled at a constant strain of 100%. The testpieces were virgin unaged samples at the beginning of each test. Initially

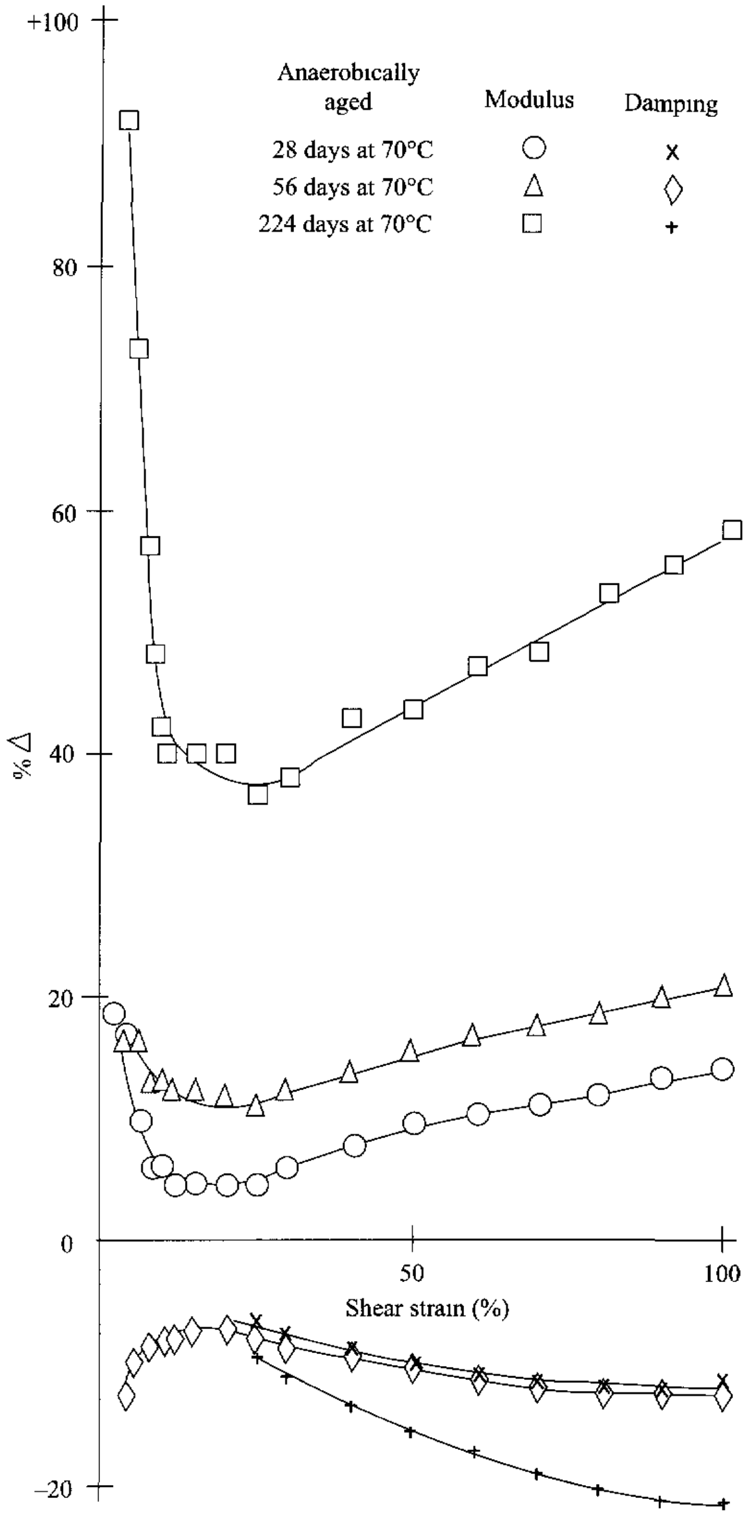

Figure 3 The change (%) in the modulus and damping versus *shear strain of testpieces aged for different times at a temperature of 70°C when compared to an unaged testpiece Compound 1, strain 2% – 100%*

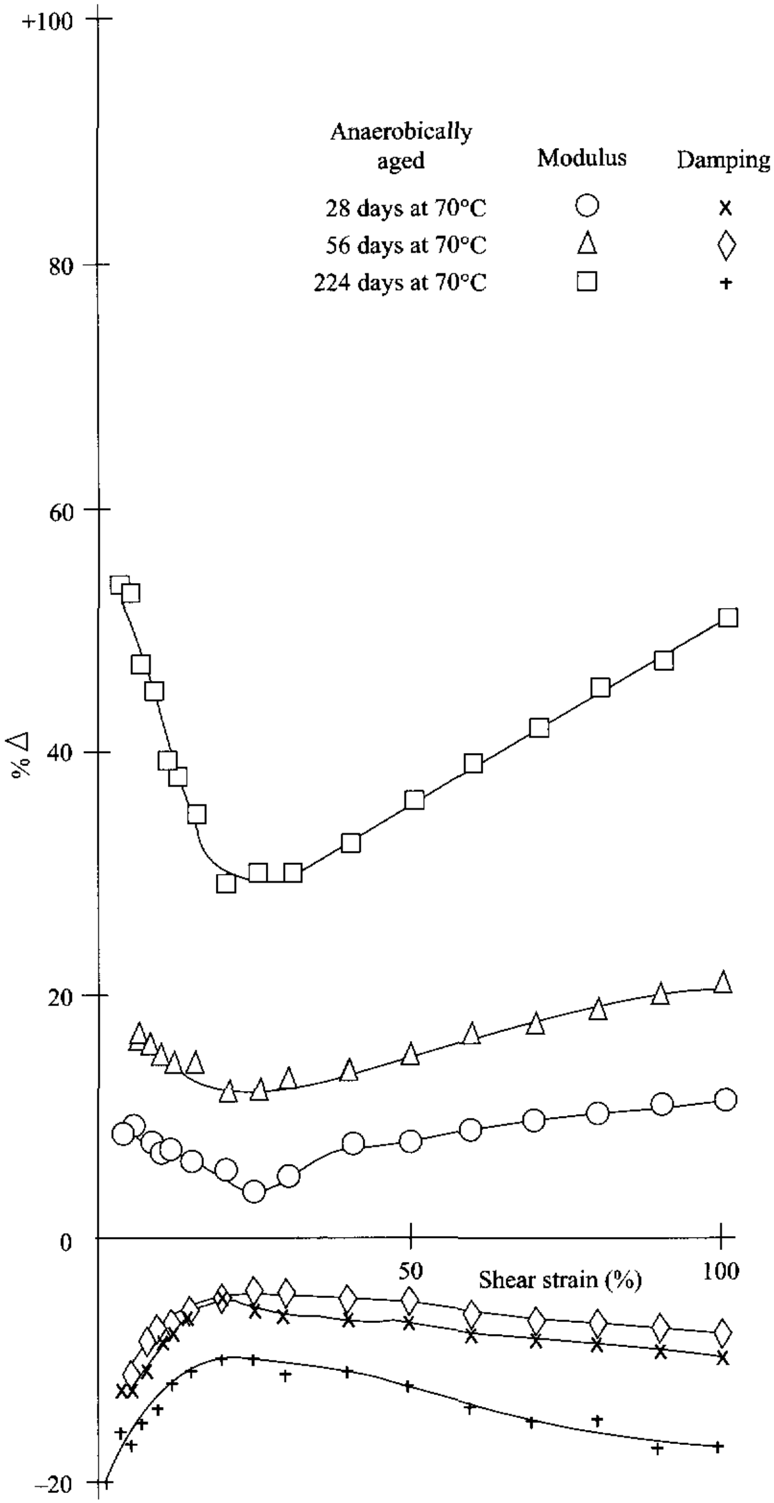

Figure 4. The change (%) in the modulus and damping versus *shear strain of testpieces aged for different times at a temperature of 70°C when compared to an unaged testpiece. Compound 2, strain 2% – 100%*

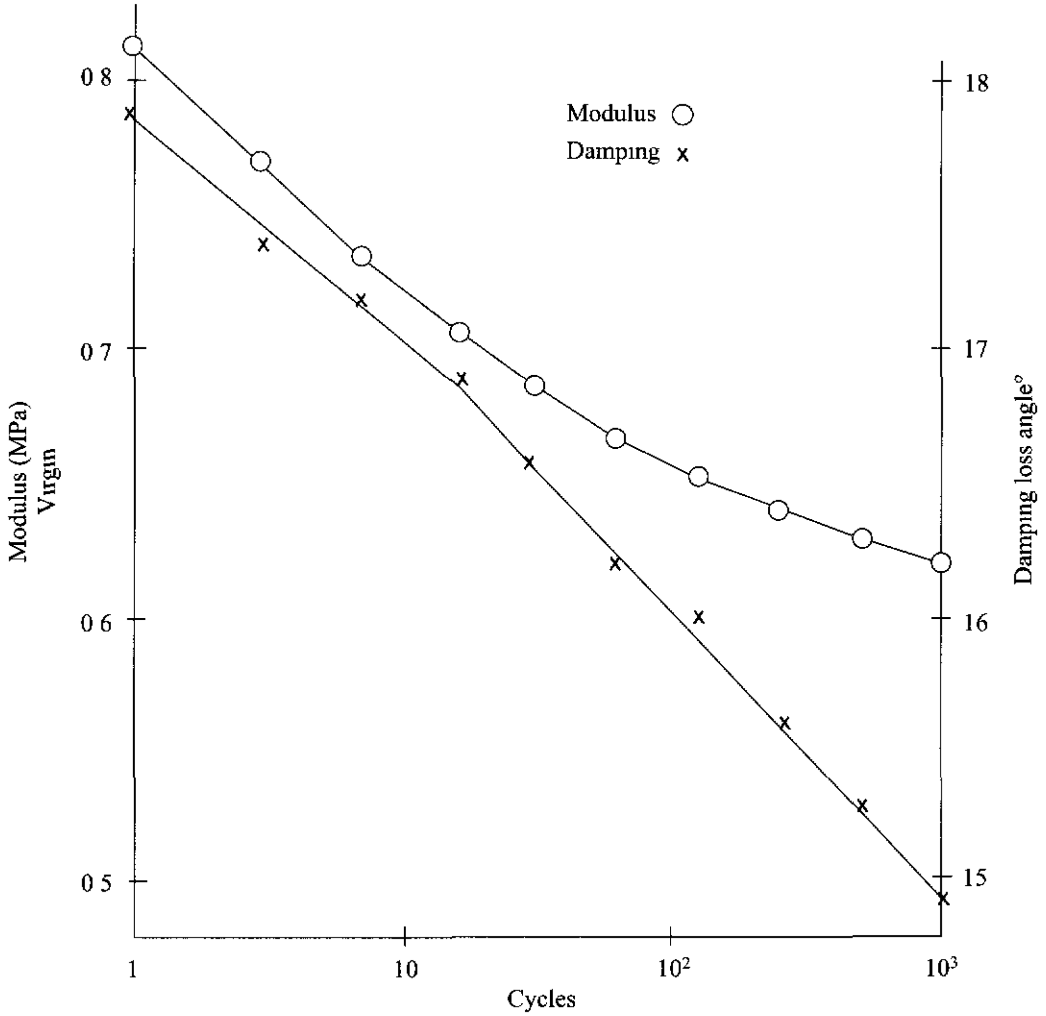

Figure 5 The values of the modulus and damping versus *the number of sinusoidal cyclic deformations Compound 1 strain 100%*

the modulus falls at a constant rate with the logarithm of the number of cycles in both compounds However, this relationship breaks down after about ten cycles in the case of the stiffer Compound 1 and after about thirty cycles for Compound 2 The progressive reduction in the rate of fall of the modulus with further cycling suggests that eventually a constant value of modulus may be achieved but it is likely that to reach such a state will require many tens of thousands of cycles Although Compounds 1 and 2 initially exhibit a reduction in the rate of fall of the damping per cycle further cycling can cause the rate of fall of the damping to become almost constant with the logarithm of the number of cycles The

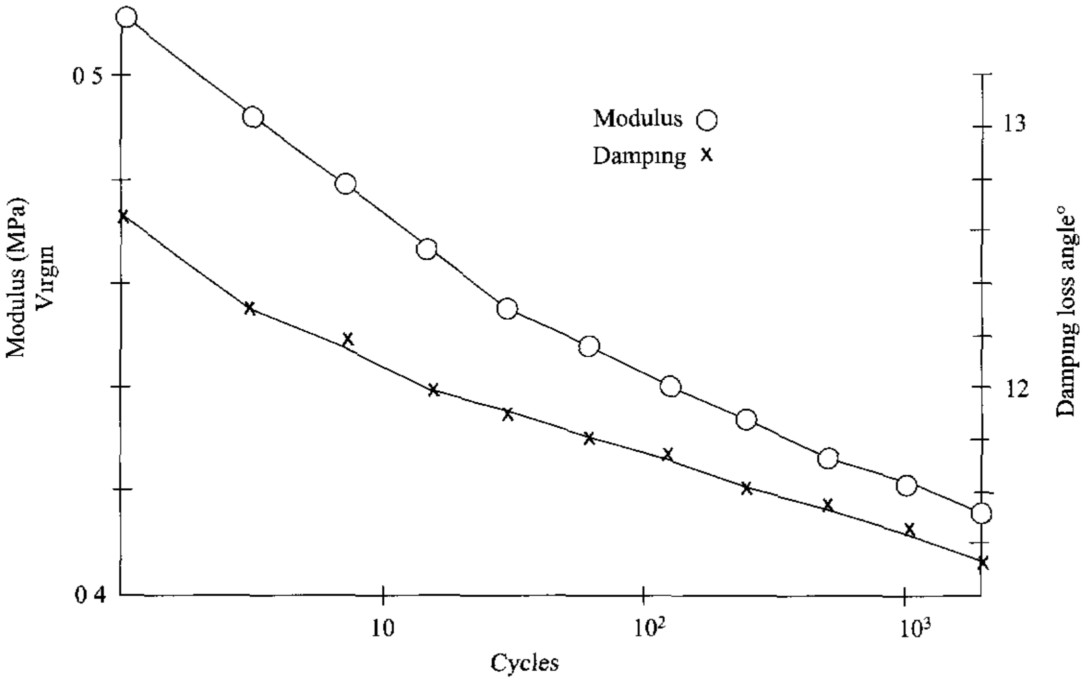

Fıgure 6 The values of the modulus and dampıng versus *the number of sınusoıdal cyclıc deformatıons Compound 2, straın 100%*

constant fall ın the dampıng wıth cyclıng may be expressed as a reductıon ın the loss angle (degrees) per decade of cycles and such an expressıon gıves a value of 1 06 degrees/decade for Compound 1 and 0 3 degrees/decade for Compound 2 The fall ın the dampıng of a vulcanısate wıth cyclıng may, therefore, be reasonably accurately predıcted over the longer term Thıs ıs not the case for the modulus where a more complex behavıour leads to greater uncertaınty

Anaerobic Ageing, Stress Softening and Recovery

Followıng a lengthy perıod of anaerobıc ageıng where the modulus of a materıal can sıgnıficantly ıncrease, as shown ın *Fıgures 3* and *4*, ıt ıs of ınterest to determıne the degree by whıch further cyclıng wıll reduce the modulus and the extent of the recovery ın hıgh-dampıng NR vulcanısates *Fıgures 7* and *8* show the percentage change ın the modulus of Compounds 1 and 2, respectıvely *versus* the shear straın when compared to unaged materıal for the partıcular straın hıstory shown The data for the unaged materıal were obtaıned usıng the same test straın schedule as shown ın *Fıgures 3* and *4*

It can be seen that the ıncrease ın the modulus at small straıns caused by anaerobıc ageıng for 224 days at 70°C ıs eradıcated ın the softer Compound 2 followıng the cyclıng at

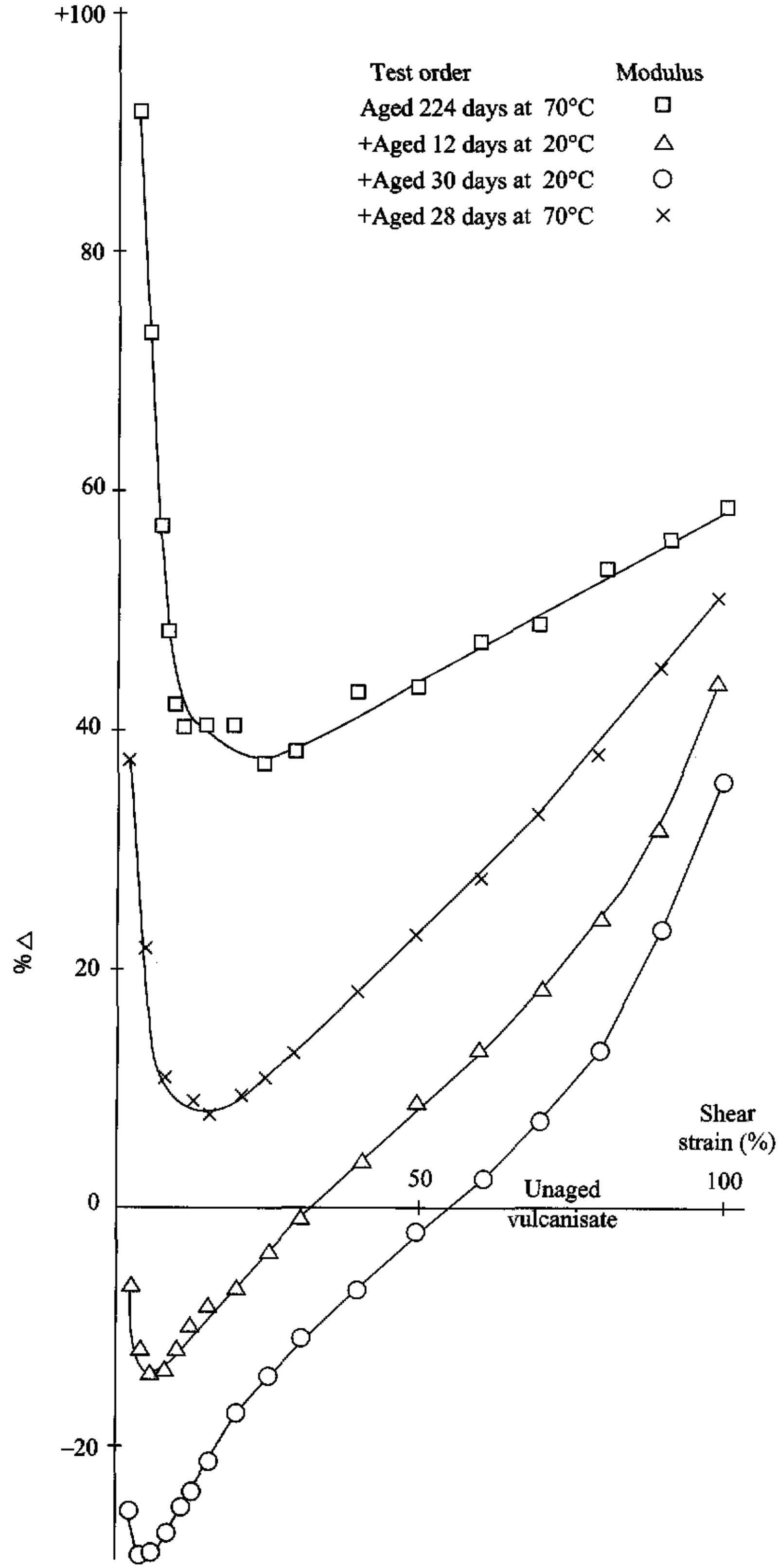

Figure 7. The change (%) in the modulus versus *shear strain of a testpieces subjected to the particular strain history shown (see test order) when compared to an unaged testpiece. Compound 1, strain 2% – 100%.*

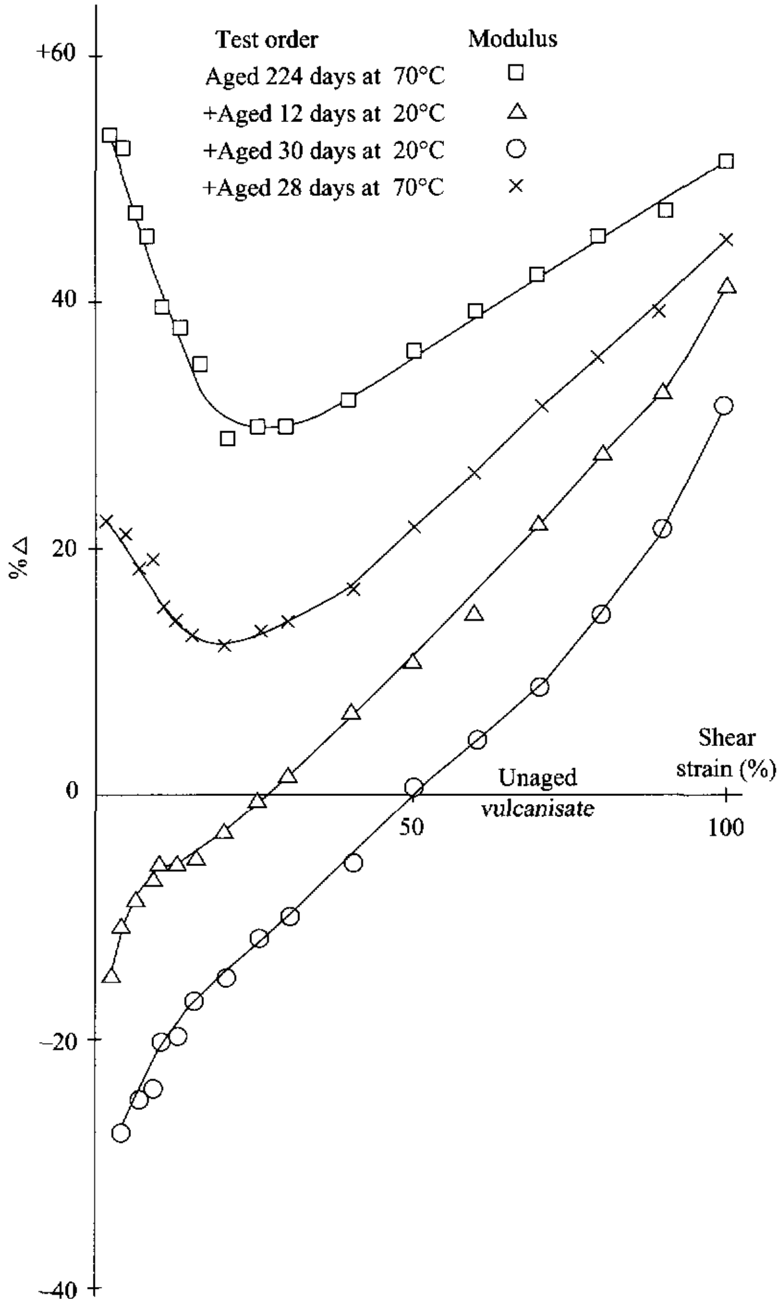

Figure 8. The change (%) in the modulus versus *shear strain of a testpieces subjected to the particular strain history shown (see test order) when compared to an unaged testpiece. Compound 2, strain 2% – 100%.*

greater strains, with the modulus in fact falling to approximately 15% below that of the unaged material after twelve days recovery in an unstrained state at ambient temperature. A somewhat similar result can also be seen for the stiffer Compound 1 although a small upturn in the modulus remains at strains of less than approximately 5%. Such results indicate that further bonds are formed during anaerobic ageing and that the bonds which are responsible for the increase in the modulus at small strains are relatively weak and may be almost completely broken by the application of a moderate strain. The breakage of such bonds can produce a significant reduction in the small strain modulus which can fall to below that of the modulus of unaged material. There is no evidence of any significant short-term (30 mins at 20°C) reformation of these weak bonds when the vulcanisate is allowed to rest in an unstrained state and following the same rest period the prior deformations have produced an almost equal fall in the modulus at all the subsequent strains probably due to further crosslink breakage.

Given a longer period of recovery in a static unstrained state (28 days at 70°C) the weak bonds can reform and again produce a significant increase in the modulus of a vulcanisate at small strains. Over the same period of time recovery of the modulus at larger strains can occur probably as a result of reformation of crosslinks following the breakage due to repeated stressing and also formation of additional crosslinks as a result of anaerobic ageing. It can be seen that such recovery can result in an overall increase in the modulus at all strains when compared to unaged material but particularly at small and large strains.

Further Strain History Measurements

In the event of a significant earthquake the rubber in an anti-seismic bearing will normally experience a small number of cyclic deformations of large strain. Following an earthquake the rubber may experience a period of relative inactivity mainly in a static unstrained state but perhaps with intermittent periods of small strain deformations caused by wind loadings. Small strain deformations alone of the order of a few percent do not significantly alter the stiffness of a vulcanisate as shown in *Figures 3*, *4* and *9*. However, the small strain stiffnesses can be altered considerably following a larger strain as shown in *Figures 7* and *8*, and as shown elsewhere[12,13]. A typical strain history of such a vulcanisate may therefore include a significant fall in the small strain modulus of the material immediately after an earthquake followed by a degree of recovery of the modulus in the short term. The modulus may also be expected to show a longer term increase as a result of anaerobic ageing before a further fall in the event of another earthquake.

It is therefore of practical importance to determine the extent such a strain history will have on the modulus and damping of an anti-seismic vulcanisate and some results from specific strain histories are shown in *Figure 9* and *Table 1*. The change in the modulus of a vulcanisate *versus* the number of cyclic shear deformations is shown in *Figure 9*. The testpiece which was initially in a virgin state was cycled three times at a strain of 5% followed by three cycles at 100% and subsequently one thousand cycles at a strain of 5%. The vulcanisate was next allowed to rest in an unstrained state for one day at a temperature of 70°C in an anaerobic condition before retesting in the same manner as before. The testpiece was now rested again in an unstrained state this time for seven days at a temperature of 70°C and then retested as previously. *Table 1* shows modulus and damping results from eight testpieces which have undergone a similar cyclic history except

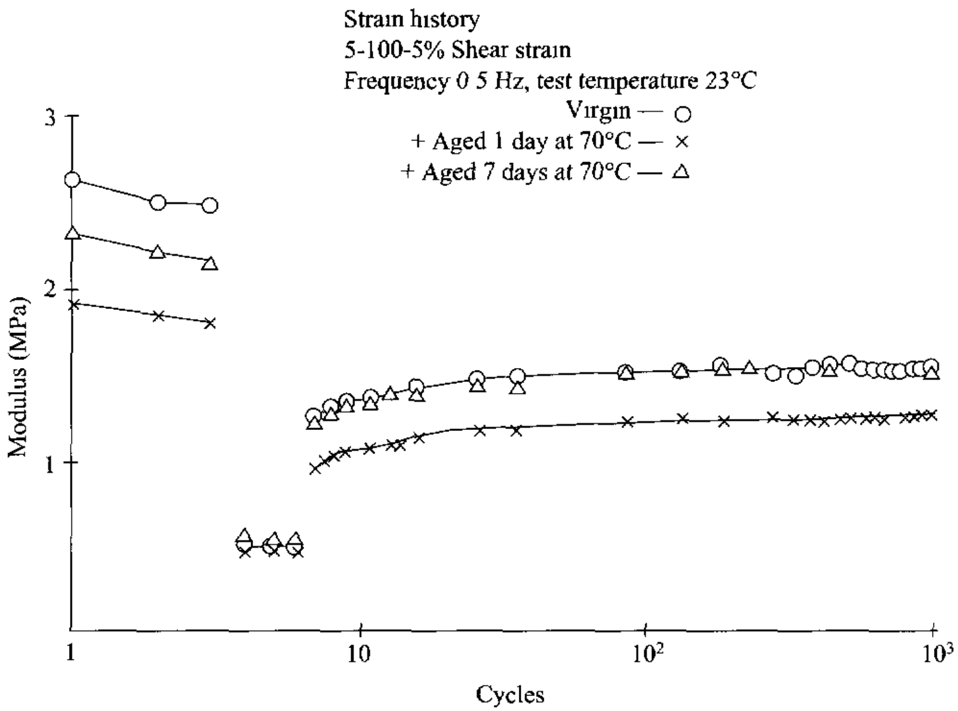

Fıgure 9 The values of the modulus versus *the number of cylıc deformatıons of a testpıece subjected to the straın hıstory shown Compound 2, straın 5% 100% and 5%*

the three large deformatıons have been varıed between 20% and 200% straın Further data followıng addıtıonal perıods of anaerobıc ageıng for 14 and 28 days ın an unstraıned state are also shown for testpıeces 5 and 6 Wıth contınuous constant small straın cyclıc deformatıons whıch follow a larger straın some recovery of the modulus takes place as shown ın *Fıgure 9* Such short-term recovery was found to be nearly complete after about one hundred cycles or approxımately three mınutes and a steady state was achıeved after approxımately one thousand cycles or 33 mın for all the test condıtıons shown ın *Table 1* The small straın modulus ın such a steady state can be seen to have fallen to between 38% and 64% of the value of the vırgın modulus from *Table 1*, the exact value beıng dependent upon the magnıtude of the prıor larger straın and the compound ıngredıents Interestıngly, the values of the modulus ın thıs steady state are only altered slıghtly by further perıods of anaerobıc ageıng and subsequent testıng, thus anaerobıc ageıng and stress softenıng appear to have nearly equal and opposıte effects on the modulus of the vulcanısates for such condıtıons

From *Table 1* ıt can also be seen that a perıod of anaerobıc ageıng ın an unstraıned condıtıon followıng the steady state condıtıon above results ın sıgnıficant further recovery of the small straın modulus of a vulcanısate For the soft vulcanısate subjected to a smaller straın (20%) almost full recovery (93%) can

TABLE 1. MODULUS AND DAMPING RESULTS FROM 8 TESTPIECES

Testpiece no.	Testpiece conditions	Strain schedule (%)	1st cycle		% of 1st cycle (Unaged)		4th cycle		7th cycle		% of 1st cycle (Unaged)		1000th cycle		% of 1st cycle (Unaged)	
			G	δ°			G	δ°	G	δ°	G	δ°	G	δ°	G	δ°
1	Unaged	5-20-5	5.93	22		100	2.17	21	3.24	26	55	118	3.35	23	56	105
	Aged 1 d at 70°C		4.93	22	83	100	2.11	20	3.12	27	53	123	3.26	23	55	106
	Aged 7 d at 70°C		5.12	21	86	95	2.17	19	3.15	25	53	114	3.35	22	56	100
2	Unaged		2.73	17			1.17	16	1.70	21	62	124	1.75	18	64	106
	Aged 1 d at 70°C		2.45	18	90	106	1.14	16	1.56	21	57	124	1.69	19	62	112
	Aged 7 d at 70°C		2.53	17	93	100	1.20	15	1.68	20	62	118	1.77	18	65	106
3	Unaged	5-50-5	5.34	22			1.19	19	2.36	29	44	132	2.92	24	55	109
	Aged 1 d at 70°C		4.44	22	83	100	1.15	18	2.37	26	44	118	2.80	23	52	105
	Aged 7 d at 70°C		4.75	20	89	91	1.25	17	2.50	27	47	123	2.94	22	55	100
4	Unaged		2.79	18			0.73	15	1.46	23	52	128	1.67	20	60	111
	Aged 1 d at 70°C		2.28	17	82	94	0.70	15	1.37	22	49	122	1.59	19	57	106
	Aged 7 d at 70°C		2.29	18	82	100	0.75	14	1.34	26	48	111	1.62	19	58	106
5	Unaged	5-100-5	6.00	21			0.86	18	2.29	27	38	129	2.86	21	48	100
	Aged 1 d at 70°C		4.48	21	75	100	0.83	18	2.01	26	34	124	2.52	21	42	100
	Aged 7 d at 70°C		4.32	21	72	100	0.91	16	2.17	26	36	124	2.69	22	45	105
	Aged 14 d at 70°C		4.22	20	70	95	0.96	16	2.08	26	35	124	2.61	21	44	100
	Aged 28 d at 70°C		4.05	19	68	90	1.04	15	1.90	24	32	114	2.55	20	43	95
6	Unaged		2.64	17			0.51	13	1.25	22	47	129	1.53	19	58	112
	Aged 1 d at 70°C		1.91	16	72	94	0.50	13	0.96	20	36	118	1.25	17	47	100
	Aged 7 d at 70°C		2.34	17	89	100	0.56	12	1.23	21	47	124	1.49	18	56	106
	Aged 14 d at 70°C		2.29	17	87	100	0.58	12	1.17	21	44	124	1.44	18	55	106
	Aged 28 d at 70°C		2.16	16	82	94	0.63	11	1.05	19	40	112	1.36	17	52	100
7	Unaged	5-200-5	5.67	22			0.81	15	1.91	27	32	123	2.48	21	42	95
	Aged 1 d at 70°C		3.53	22	59	100	0.77	15	1.80	27	30	123	2.30	21	38	95
	Aged 7 d at 70°C		3.75	22	63	100	0.84	13	1.76	26	29	118	2.30	21	38	95
8	Unaged		2.62	16			0.44	12	0.93	22	35	138	1.26	17	48	106
	Aged 1 d at 70°C		1.69	18	65	113	0.43	11	0.92	22	35	138	1.19	18	45	113
	Aged 7 d at 70°C		2.09	18	80	113	0.47	10	1.02	21	39	131	1.29	17	49	106

take place, but, this is not the case for the stiffer vulcanisate experiencing greater strains where recovery values have fallen to as little as 59% of the unworked modulus. The damping of both vulcanisates at a small strain is significantly increased immediately following a larger strain, an increase of between 11% and 30% compared with virgin material, but during the recovery the damping tends to fall and typically returns to a value not very different from virgin material when a steady state has been reached. Only a relatively small change occurs in the value of the steady state damping throughout all the strain histories employed here despite the variations in the period of anaerobic ageing and larger applied strain. The overall effect of the strain – anaerobic ageing histories shown in *Table 1*, is to decrease the small strain modulus (5%) of the materials in all cases when compared to virgin material but damping may increase or decrease slightly depending upon the particular condition. With such a fall in the modulus throughout all the strain histories, even following a period of 28 days ageing at a temperature of 70°C (which alone will normally produce a significant increase in the modulus of the vulcanisate), stress softening evidently has a greater effect on the modulus than anaerobic ageing at small strains under these conditions.

At the maximum strain applied to a testpiece (20%–200%, *Table 1*) a sufficiently long period of anaerobic ageing (≥7 days at 70°C) produces an increase in the modulus of the vulcanisates when compared to virgin material. Here the effect of anaerobic ageing on the modulus is dominant over that of stress softening. Anaerobic ageing will produce further crosslinking as a result of the cure maturation processes occurring in the vulcanisates which will tend to reduce the damping of the material. Such a fall can be seen to occur at all the strains between 20% and 200% shown in *Table 1* (4th cycle data).

CONCLUSIONS

Anaerobic ageing tends to increase the modulus but decreases the damping of a vulcanisate. The modulus is found to increase at approximately a constant rate with the square root of the ageing time. Such behaviour aids the long-term prediction of time-dependent change in the modulus of a vulcanisate. The increase in modulus due to anaerobic ageing is found to be dependent upon the applied strain with the largest changes in modulus occurring at small and large strains. The combined effect of anaerobic ageing and stress softening in the strain histories described here tends to reduce the small strain modulus (5%) of a vulcanisate when compared to virgin material, but following a sufficiently long period of anaerobic ageing, the modulus at the largest strain (≥20%) increases. The different strain histories have little long-term effect on the damping of a vulcanisate which when in a steady state remains similar to virgin material although immediately following a larger strain the damping at a smaller strain can show a significant short-term increase.

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