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Elastomer · Swelling · Fatigue · Bubble inflation

This paper describes the behaviour of rubber in multi-axial fatigue, with respect to oil resistance. A series of tests were carried out on EPDM specimens subjected to varying degrees of swelling. The control methodology and the loading mechanism employed in the testing of elastomers using dynamic multi-axial bubble inflation are outlined. Swollen specimens were cycled to failure and the results were analysed. The number of cycles to failure for a given engineering stress amplitude was plotted for both unswollen and swollen specimens. Fracture surfaces for each set of specimens were analysed. The effect of swelling on the stress-strain behaviour and the change in complex modulus for each specimen set is discussed.

Der Einfluss der Ölquellung auf die Lebensdauer von Elastomeren im zyklischen Aufblasversuch**Elastomer · Quellung · Ermüdung · Aufblasversuch**

In der Arbeit wird das Verhalten von Elastomeren im multiaxialen Ermüdungsversuch im Hinblick auf die Ölbeständigkeit eingehender beschrieben. Eine Reihe von Versuchen wurden mit unterschiedlich gequollenen EPDM Proben durchgeführt. Versuchsbedingungen und Auswertung wurden beschrieben. Gequollene Proben wurden bis zum Versagen zyklisch belastet. Die Anzahl der Zyklen bei konstanter Spannungsamplitude wurde für nichtgequollene wie für gequollene Proben aufgetragen. Die Bruchflächen wurden für jede Serie von Proben untersucht. Der Einfluss der Quellung auf das Zug-Dehnungsverhalten und die Änderungen des komplexen Moduls wird für jede Probe beschrieben.

The Effect of Oil Swelling on the Fatigue Life of Elastomers Subjected to Cyclic Bubble Inflation¹

Most rubber components are subjected to fatigue conditions. In addition, there are cases where such components may be at risk from oil contamination. In such cases performance of the part in question may be severely compromised if the rubber and the fluid are compatible in terms of swelling potential. EPDM components are commonly used in automotive applications, where they have the advantage of having more functionality at higher temperatures than SBR or natural rubber. However, these advantages are offset by their poor resistance to numerous fluids used in vehicles. By using reference oils, their properties are comparable with such fluids and the parameters of the swelling liquid can be reported with the test results. While there have been previous studies into the fatigue behaviour of swollen rubber, none of these have considered repeated multi-axial loading. Few components are loaded uniaxially and rubbers are quite different materials when loaded multi-axially, due to the orientation of long chain molecules. The fundamental question this study addresses is whether altering the rubber filler matrix by the introduction of a swelling agent affects the multi-axial fatigue behaviour of the rubber. These tests allow quantification of the damage and the effect that swelling has on the fatigue life of EPDM. In order to fully evaluate the problems presented, parallel investigations were carried out to investigate the influence of swelling in multi-axial fatigue. Firstly, it was proposed to conduct fatigue tests on EPDM test-pieces, with one specimen set untreated and the other subjected to swelling before testing. These fatigue tests were carried out on both sets of test-pieces using equal pressure and subsequently equal stress amplitudes. Secondly,

the structures of both sets of specimens at failure were studied to determine the effects of swelling on the fracture surfaces.

Theory

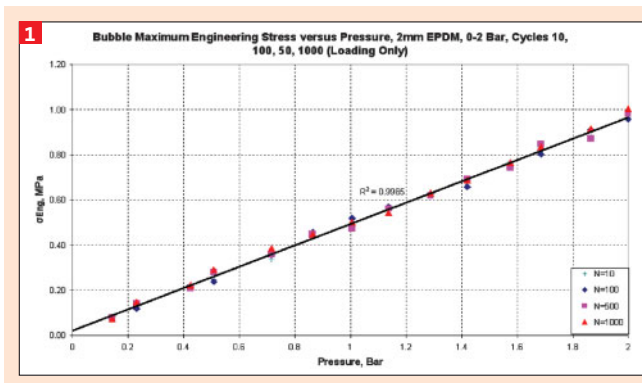
In terms of oil and rubber compatibility, it is important to consider that in any practical application both the oil and the rubber are made up of several different components. The compatibility can be made complex by such variables as fillers and plasticisers, rubber molecular structure, selective swelling action by fluid components, time-temperature effects on swelling rates and chemical degradation of the elastomer or fluid. All rubber will swell to some extent in oil [1], but the degree of swelling can be estimated for a particular oil-rubber combination, if the solubility parameters δ , for both substances are known. If the square root of the difference between the solubility parameters of the rubber and the oil is less than one, then the rubber will swell appreciably in the oil. It is clear that there can be difficulties in accurately determining the solubility parameter for a fluid when it consists of two or more fractions. Previous research [2] found that the solubility parameter for a hydraulic fluid may be estimated from known physical properties which are readily available. The experiments allowed the relationship between the solubility parameter and the ani-

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1 Plot of bubble pole stress versus inflation pressure for dry EPDM, selected cycles, 1-1000

line point of the oils being tested to be plotted as an empirical linear relationship. In order to best demonstrate the effects of swelling in typical hydraulic oils it is therefore necessary to use hydraulic fluids where the properties have been determined and can be used as a reference. From the ASTM standards for rubber liquid compatibility, the reference oil IRM903 is the most appropriate choice for use as a high swelling oil for the physical testing of swollen rubbers. Its aniline point has been determined and can be compared with the solubility parameter for the test material to allow rubber-liquid compatibility to be gauged. Moreover, it is generally comparable with typical hydraulic oils in terms of liquid properties.

In terms of fatigue of swollen elastomers, previous research [3] investigated the phenomenon of fatigue resistance for constant 'stress-work' amplitudes with a range of degrees of swelling for a given rubber-solvent combination, using rubber plasticisers as the solvents. The results of these uniaxial tests showed that the relative fatigue resistance of the natural rubber decreased with an increase in the degree of swelling, regardless of the rubber-solvent combination used. Further work on fatigue of swollen rubber [4], investigated fatigue crack growth of elastomers on two types of SBR swollen in mineral oil and fatigue tested under conditions which replicated pure shear with sharp stress raisers (pre-cracks) and strains applied cyclically at magnitudes of 10-35%. Tearing energy and crack growth rate were measured and compared against specimens that were not swollen. The study found that the fatigue resistance of the elastomers was decreased in the presence of low-viscosity fluid in proportion to the degree of swelling. This variation of the fatigue resistance of an elastomer in the swollen state was due to the reduction in viscoelastic energy loss and sharpening of the crack tip when the rubber was swollen. The tests presented in this work differ from those referenced in a number of

respects. Firstly, test frequencies were maintained at 1Hz, as opposed to frequencies in excess of 4 Hz, which would have led to heat build-up and consequent thermal degradation [5]. Secondly, virtually all components are complexly loaded in service, so constant stress-work or simulated pure shear do not give a complete picture of the fatigue behaviour of swollen elastomers.

Test procedure

Materials

EPDM rubber of 70 Shore A hardness, cross-linked with sulphur and containing low activity carbon black was first chosen for the investigation. Initially dry (unswollen) specimens were fatigued to failure with a zero minimum load. Dynamic multi-axial tests were carried out by clamping a circular specimen having a thickness of 2 mm and a clamped diameter of 35 mm around its edges and applying pressure to one face of the disc causing it to inflate in a balloon like manner.

Swelling tests

When inflating rubbers using liquids, the swelling potential of the inflation media must be taken into account. Silicone based fluids will not swell EPDM appreciably [6]. Following swelling tests to confirm this, such a fluid was chosen as the inflation medium and all fatigue tests were carried out at temperatures of 15 °C. Firstly, the unswollen test-pieces were fatigued to failure under multi-axial loading conditions. Stress-strain relations were recorded at different cycles and the cycles to failure were noted for each specimen.

Swelling experiments were carried out by immersing the specimens in reference oil IRM903, at an elevated temperature of 100 °C for a period of one hour. The specimens were removed from the hot oil and cooled in oil at ambient temperature for a short period, before being wiped dry and

weighed [7]. An average swelling ratio of 1.10 (10% increase in mass) was calculated for the samples, where the swelling ratio Q can be expressed as,

$$Q = W_s / W_d \quad (1)$$

where W_s is the weight of the swollen elastomer sample and W_d is the weight of dry elastomer before swelling. Following swelling calculations, the swollen test-pieces were inflated to failure under multi-axial fatigue loading, again using silicone fluid to inflate the test-pieces.

Multi-axial fatigue tests – Development of test procedure

Bubble inflation is considered to comply with theory for application of pressure to thin shell structures possessing negligible bending stiffness, alternatively described as membrane theory. Stresses at the bubble pole can be determined from the local radius and applied pressure using equation [2]:

$$\sigma = PR/2t \quad (2)$$

Where P is the pressure, R is the radius at the bubble pole and t is the original sheet thickness.

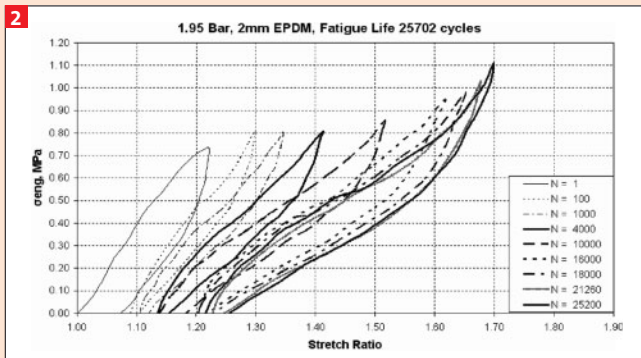
Local stretch ratios at the pole can be determined using equation [3]:

$$\lambda = ((x_{cir} - x_{orig})/x_{orig}) + 1 \quad (3)$$

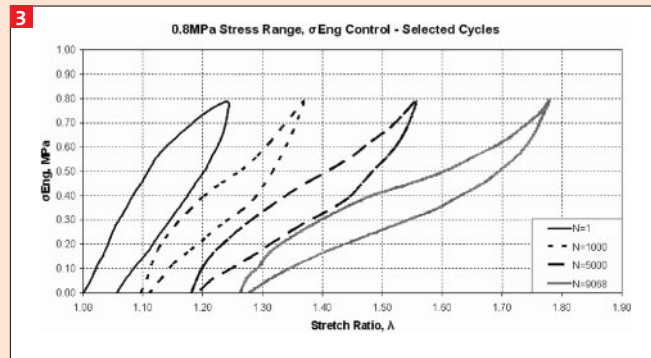
Where λ is the principal stretch ratio, x_{cir} is the circumferential point spacing at the bubble pole and x_{orig} is the original point spacing.

Prior to fatigue testing, pre-defined pressure limits were selected. Thereafter, the specimen was inflated and deflated between these limits. During these deformations, the movements of markings on the surface of the sheet were recorded and stress and strain values were calculated from the applied pressure and measured bubble geometry. Initial tests were carried out using constant pressure as a control parameter for the fatigue tests. This was based on calculating the engineering stress from plots of pressure versus stress for selected loading cycles, where the relationship was found to be approximately linear up to 1000 cycles, as illustrated in Figure 1.

Subsequent analysis of the stress-strain behaviour of the fatigued samples showed that when constant pressure was used as a control mechanism during dynamic cycling of the EPDM test-pieces, engineering stress increased throughout the test for a given applied pressure. This is illustrated in Figure 2, where a sample loaded to an initial first cycle stress of 0.7 MPa showed a continued



2 Stress-strain behaviour of EPDM for constant pressure controlled test, selected cycles, 1-25200



3 Stress-strain behaviour of EPDM for constant engineering stress controlled test, selected cycles, 1-9068

increase in maximum stress in subsequent cycles. The peak stress remained relatively constant between cycles 10-1000, but thereafter continued to increase until failure occurred. This was found to be the case for all the ranges tested.

To allow the peak engineering stress in a cycle to be maintained at the same value for each and every cycle throughout the test, the pressure set-point was adjusted continually throughout the test. By using this method of control, practical S-N curves could be obtained to compare the fatigue lives of the dry and swollen rubber. Figure 3 shows typical specimen stress-strain behaviour for selected cycles of an EPDM sample when the maximum engineering stress is controlled for a zero minimum stress and hence a constant stress range.

Results

Multi-axial fatigue tests – S-N curves

Plots were generated of stress-amplitude versus cycles to failure for both the swollen and unswollen test-pieces and these are shown in Figure 4.

Unsurprisingly, the unswollen specimens exhibited greater fatigue resistance than the swollen test-pieces. It can be seen that

the S-N curve for the swollen material is shifted downwards from that of the unswollen material. In order to study this effect further, the complex modulus E^* of both specimen sets was analysed for several stress ranges as shown in Figure 5.

E^* was approximated by calculating the slope of the loading curve from zero stress and minimum strain to peak engineering stress and maximum strain for the cycle in question. It can be seen clearly that there is an offset in initial modulus for the swollen EPDM. The rate of decrease of E^* in the dry rubber is more rapid E^* values at failure appear to lie on a single line when plotted against the log of cycles to failure (Reference X-X on Fig. 5). The decrease in E^* is less severe in the swollen specimens but the relationship between E^* and the log of cycles to failure no longer appears to be linear (Reference Y-Y on Fig. 5).

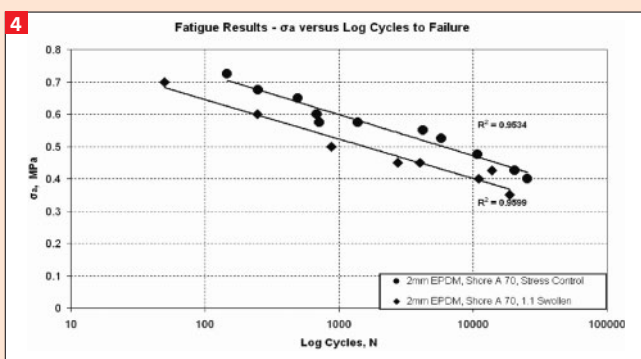
Multi-axial fatigue tests - Sample fracture surfaces

The fracture surfaces of the dry samples were analysed using a digital imaging microscope. A magnification factor of X200 was used to view failure surfaces. For low cycle lives of less than 100 the type of failure is more akin

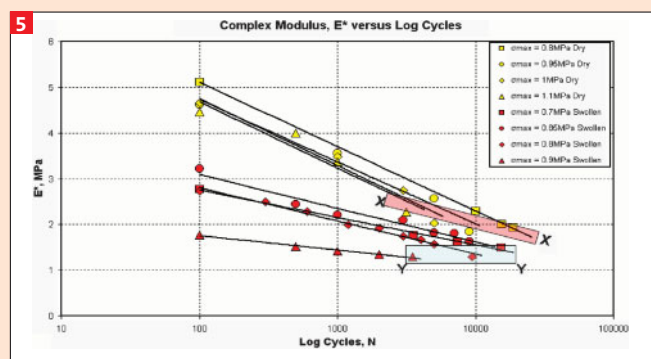
to that of a single cycle test to destruction, with the surface morphology fibrous in nature and in some instances showing delamination at the failure surface. Failures at cycles greater than this show clear evidence of crack propagation and subsequent rupture. In most fatigue failures, the cracks appear on the surface of the bubble and continue to grow until failure. This behaviour was common to both the swollen and unswollen specimens. Surface morphologies for both dry and swollen fatigued specimens are shown in Figure 6. In the dry samples there is a coarser failure surface at lower cycles than for failures at higher cycles. The roughness of the failure surfaces of the swollen EPDM samples is much greater than that of the dry test-pieces for all stress ranges. These results correlate with those carried out by Cho *et al* [4] in shear tests on specimens which were fatigued to failure with a pre-crack. Their study reported that the smoother surface of the dry samples indicated blunt tearing, while the rough surfaces of the swollen test-pieces indicated sharp tearing.

Conclusions

The fatigue life of an EPDM sample under dynamic multi-axial loading can be greatly



4 Plot of stress amplitude versus cycles to failure for the dry and swollen specimens



5 Log plot of complex modulus E^* versus cycles for swollen and dry samples

reduced in the presence of the oil, even for relatively small amounts of swelling. The lower fatigue lives of the swollen specimens can be attributed to a number of factors, both physical and chemical in nature. Physical factors include a lower initial complex modulus following swelling, breakdown of filler-filler bonds and the presence of larger voids in the network due to the swelling action. Chemical factors include changes in the network structure due to swelling, where there is a reduction in the number of chains resisting the tensile force, while the reformation of polysulphidic linkages during a loading cycle can be inhibited in the presence of swelling fluid. The general fatigue behaviour of swollen elastomers loaded multi-axially is in close agreement with the experiments carried out in the uniaxial and shear loading cases.

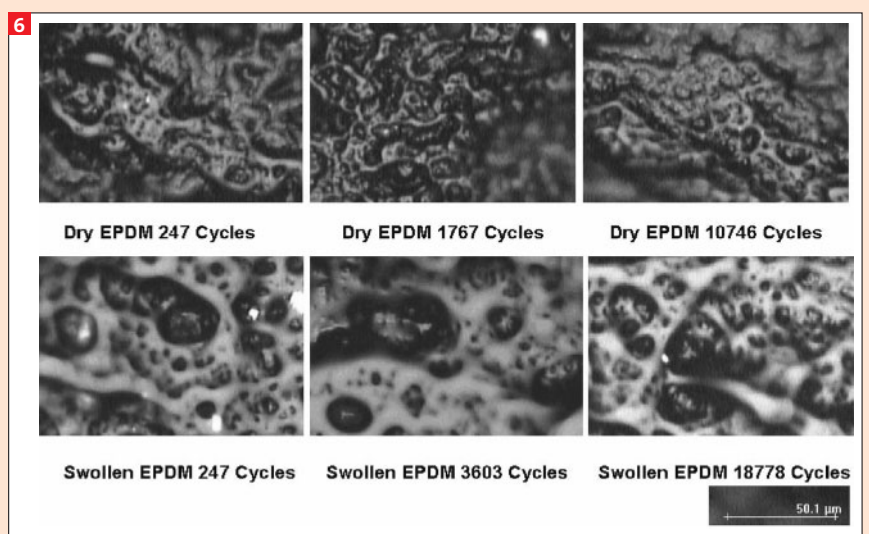
Further testing is planned where longer fatigue tests will be performed on dry and swollen samples, with additional tests being carried out on EPDM swollen using a medium swelling reference oil (IRM 902). The stress-strain behaviour of each set of samples will be analysed in an attempt to determine if the stored energy in a cycle or a limiting value of E^* can be used as a predictor of useful component life. Further imaging of the specimen fracture surfaces will also be carried out using scanning electron microscopy (SEM).

Acknowledgements

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6 Fracture surface of dry and swollen specimens, magnification factor X200

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