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Failure of Bonded Natural Rubber Cylinders in Tension

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This study describes modes of failure which may occur in bonded rubber blocks in tension. Aspects of cavitation failure and interfacial bond strength are discussed in vulcanisates containing different carbon loadings. The relationship between cavitation and bond failure on the ultimate failure stress is examined. Results indicate that the mechanism responsible for ultimate failure tends to change from one of debonding to cavitation as the carbon content is increased. The conditions required to initiate surface crack propagation and the effect of such cracks on breaking stress are also described.

Under some conditions, cracks can propagate from cavities in the interior of rubber. Busse[1] and Yerzley[2] originally reported this phenomenon and later other workers[3,4], concluded that for a neo-Hookean material, a critical inflating pressure of 5E/6 was required to initiate rupture of these cavities. Subsequently this criterion was found to be correct only for cavities with a limited range of initial sizes[5,6]. For rubber bonded between rigid plates and subjected to a tensile force the resulting triaxial tension in the rubber can lead to negative hydrostatic forces sufficient to cause rupture of cavities in a similar manner to a positive internal pressure[3,7,8]. Although natural rubber is not normally used in tension, and for most practical purposes, the rupture of cavities will not occur, localised tensile forces can exist in laminated blocks in shear. The propagation of cracks from cavities in bonded natural rubber blocks under tension does not, however, always lead to an immediate catastrophic failure of the specimen, but rather to a progressive accumulation of damage which will reduce the component's performance. As well as failure by cavitation, bonded rubber blocks subjected to tensile forces can experience failure at the rubber-bonding adhesive interface. Thus it is of interest to determine whether cavitation or bond performance will normally lead to the ultimate failure of a rubber component. The work reported here has been conducted in an attempt to study further cavitation and bond failure in a range of natural rubber vulcanisates bonded with a proprietary adhesive. Measurements of the effect of reinforcing plate misalignment on the ultimate failure of a bonded rubber layer are also discussed.

The experiments examine the failure under tension of single-layer natural rubber bonded cylinders covering a range of shape factors. Test pieces moulded from vulcanisates containing various carbon black loadings were investigated. The types of failure occurring and their effects on stress-strain behaviour are discussed.

EXPERIMENTAL

Test Pieces

Seven natural rubber compounds covering the normal hardness range (about 43–83 IRHD) were investigated. A conventional sulphur cured gum vulcanisate was used as the base material with carbon black loadings (N330) of between 1.0 p.p.h.r. and 80 p.p.h.r. added. The rubber compound formulations, with relevant physical properties, are given in *Table 1*. The load-deformation and failure studies were all conducted on single-layer natural rubber

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TABLE 1. RUBBER COMPOUND FORMULATIONS AND PHYSICAL PROPERTIES

Formulation (p.p.h.r.)	Compound No.						
	0	1	2	3	4	5	6
SMRL 100	100	100	100	100	100	100	100
Zinc oxide	5	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2	2
Sulphur	2.5	2.5	2.5	2.5	2.5	2.5	2.5
CBS	0.6	0.6	0.6	0.6	0.6	0.6	0.6
Antioxidant 2246	2	2	2	2	2	2	2
Carbon black (N330)	–	0.1	2	20	80	80	50
Dutrex 729						25	
All cures to t_{95}							
Physical properties							
Young's modulus, E_o[a] (MN/m^2)	1.6	1.6	1.7	2.6	9.3	5.6	6.3
Tear strength[b] (kN/m)	8.0	8.1	8.3	12.0	5.2	26.3	24.0
Hardness (IRHD)[c]	43	43	44	55	83	67	69
Tensile elongation at break[d], e_b, ISO 37 (%)	770	760	755	660	206	421	545
Average bonded breaking strain (e_b) as function of e_b	0.46	0.47	0.48	0.44	0.29	0.49	0.21

[a] E_o to ± 0.1 MN/m^2

[b] Tear strength, trouser, to *ISO 34*

[c] Hardness to *ISO 48*

[d] Break to *ISO 37*

bonded discs (diameter 50 mm) pulled in tension using the proprietary bonding adhesive system[9], Chemlok 205/220.

Test pieces were manufactured by cutting unvulcanised rubber discs from calendered sheet and placing them between two mild steel discs to form a sandwich unit. Each metal disc was 20 mm in thickness, sufficient to ensure no significant bending occurred during testing. Before applying the bonding adhesive, the metal discs were degreased by cleaning in trichloroethane and shot blasted to ensure good bond strength. Each metal disc had a central threaded hole for attachment to a tensile test machine. The assembled units were hot cured in a mould to 95% of full cure.

Method

The thickness of the rubber discs was varied to give a range of shape factor, S[= radius/(2 × thickness)] between about one and eleven. The test pieces were given a single pull on a test machine at a crosshead speed of 2 mm/min unless otherwise stated. Load-extension graphs were obtained in each case. Internal rubber cracking could be detected by deviations in the load-extension data and in some cases by associated audible cracking sounds.

The extent and distribution of cracks were investigated by cutting open the test pieces after pulling to known stress values, either to

break, to a load just before break or in some cases to slightly in excess of the cavitation stress. Thirty samples were sectioned.

RESULTS

Internal Rubber Failure, the Cracking (or Cavitation) Stress

Internal rubber cracking was identified by fluctuations in a test piece's load-deformation behaviour. Some examples of such behaviour for different compounds of similar shape factor are shown in *Figure 1*. Well defined cracking stresses can be identified for gum and lightly filled carbon black compounds. The smallest stress, when the first fluctuation occurs, is given as the initial cracking stress. Typically, and particularly for unfilled and lightly filled rubbers, the majority of cracking takes place over a narrow stress band. Such

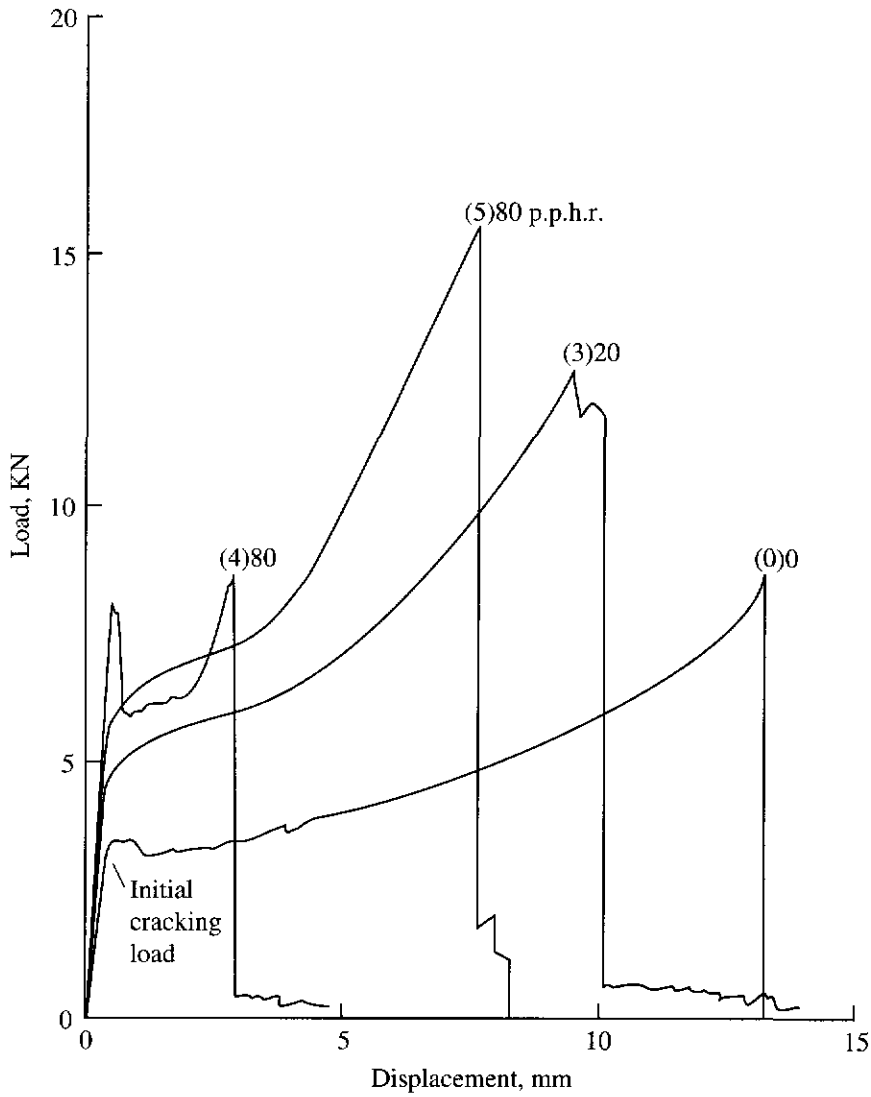

Figure 1. Load-displacement plots for four rubber discs pulled in tension to failure. The numbers in brackets indicate the vulcanisate tested and the carbon content (see Table 1*). Shape factor 3.5.*

typical cracking behaviour for lightly carbon black loaded vulcanisates of different shape factors is shown in *Figure 2*. The cracking stress became less well defined as filler content increased because of a decrease in the magnitude of the stress fluctuations upon cavitation and also a decrease in the number of cavities present (*Figure 1*). When cracking occurred, the fluctuations remained sufficient, however, for the cracking stress to be identified even for high filler loadings.

The initial cracking stress as a function of shape factor for the seven compounds is shown in *Figure 3*. Generally, the stress increases with increasing shape factor. For gum and lightly filled compounds, the cracking stress increased by about a factor of two between a shape factor of about 1 and 10; however, as filler content increased, this factor became somewhat less, reaching about 1.3 at high filler loadings. The initial cracking stress values obtained in this study are in reasonably good agreement with the values found by Gent and Lindley[3].

The inclusion of small quantities of carbon black filler (N330) appears to increase the cracking stress significantly, for example, 2 p.p.h.r. added to a gum compound increases the cracking stress by about 20%. As carbon black content increases, however, this improvement in cracking stress becomes proportionately less. The observed initial cracking stress falls between about 0.9 MPa for low shape factor test pieces moulded from gum vulcanisates to about 4.4 MPa for high shape factor test pieces moulded from highly filled vulcanisates. The stress is shown as a function of Young's modulus (E_O) for each vulcanisate at three different shape factors in *Figure 4*. The stress-strain behaviour of filled rubbers particularly those containing a large proportion of filler, is non-linear. The E_O values quoted here are for 0%–2% strain. The initial cracking stress is seen to be proportional to E_O, as originally found by Gent and Lindley[3] for gum and intermediately loaded carbon black vulcanisates (*Figure 4*). For the highly filled vulcanisates ($E_O > 4$ MPa), however, a departure from this linear behaviour occurs and the stress passes through a maximum value. Comparison of the performance of *Compounds 4* and *5* shows that adding process oil (25 p.p.h.r. Dutrex 729) reduces the cracking stress by about 20%.

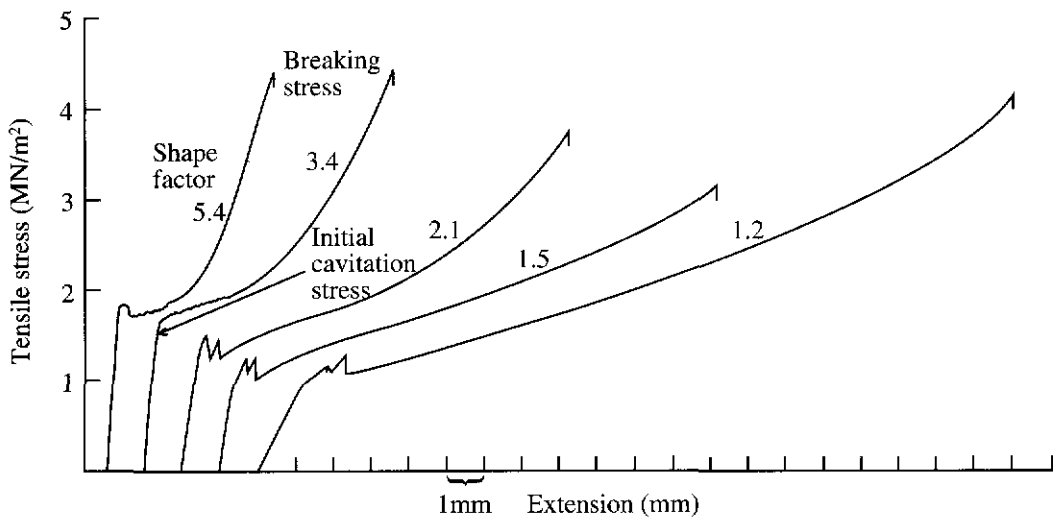

Figure 2. Tensile stress versus *extension behaviour of a lightly carbon black filled vulcanisate* (Compound 2) *for test pieces of different shape factors. Cavitation is indicated by deviations in the curves.*

Internal Rubber Failure, the Initial Cracking Strain

Generally, for elastomers, the initial cracking strain was found to be relatively small. Typical variations of cracking strain with shape factor are shown in *Figure 5*. The increase in cracking strain at small shape factor (approximately 2) is attributed to test pieces approaching a large length to breadth ratio, *i.e.* normal tensile type dimensions.

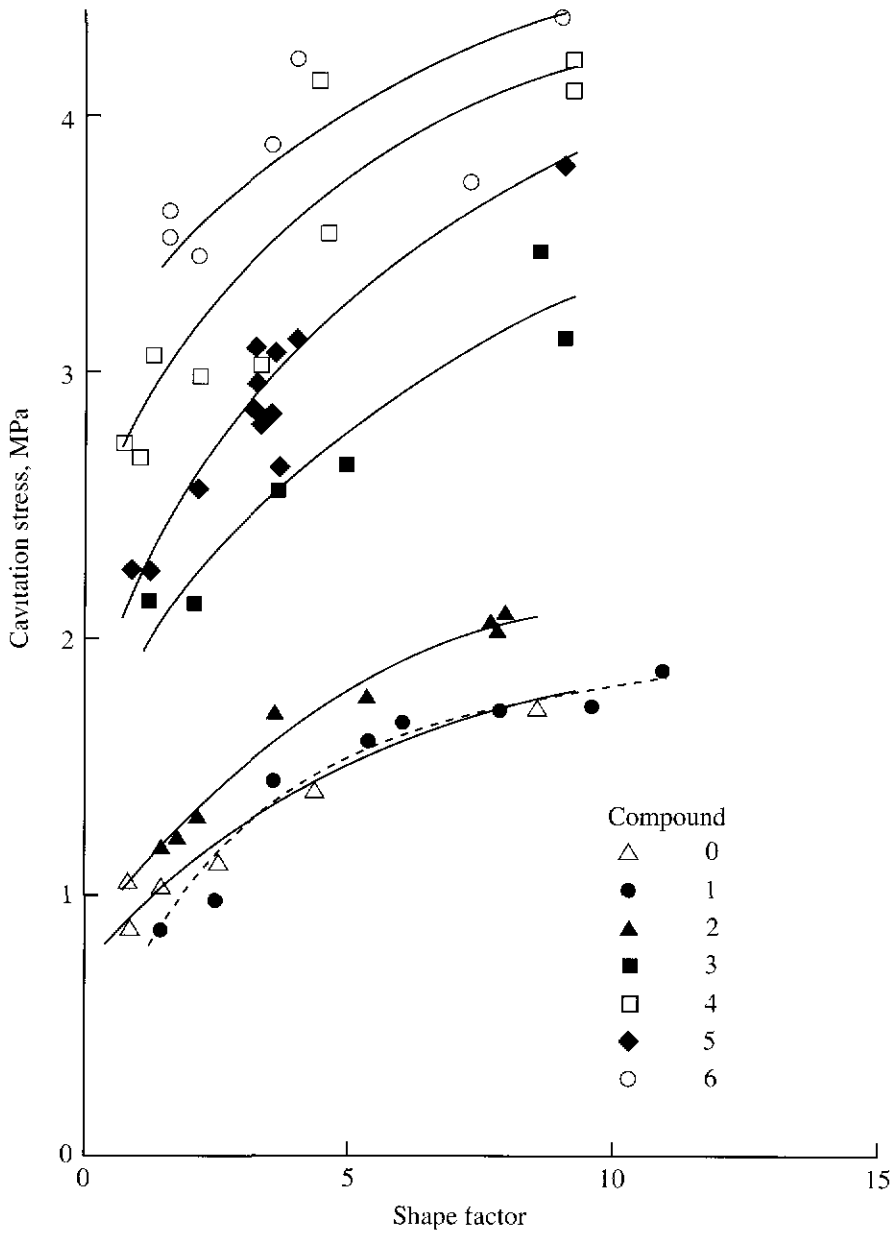

Figure 3. Cavitation stress plotted against shape factor for the seven vulcanisates in Table 1.

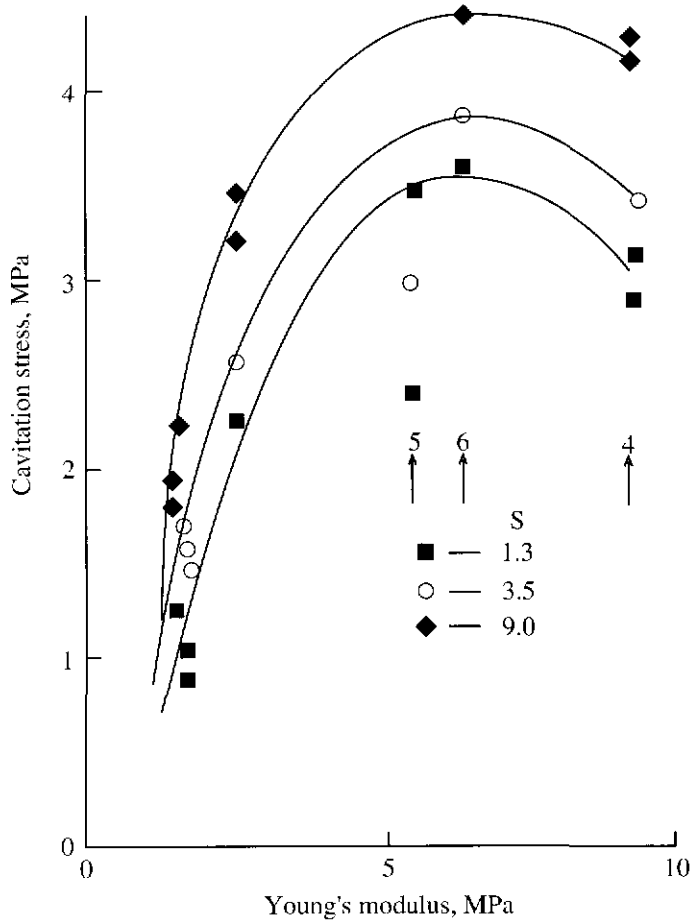

Figure 4. Cavitation stress plotted as a function of Young's modulus (at 2% strain) for test pieces of three different shape factors. Arrows indicate results for the particular compounds (given by the numbers) discussed in the text.

Effect of Internal Rubber Cracking on Stress Retention

Although internal cracking within rubber can occur at stresses much less than those required to break a test piece under most conditions, the presence of these internal cracks does not have a catastrophic effect on the stress retention of the rubber. Internal cracking will, however, alter the force *versus* deflection behaviour, and for purposes of component design, the onset of cavitation may be regarded as the normal maximum working stress. An indication of the magnitude of the effect of internal cracking on the stress-extension behaviour of a very lightly carbon black loaded vulcanisate (*Compound 2*) given a single pull, is shown in *Figure 2* for a range of shape factors between 1.2 and 5.4. In unfilled and lightly filled vulcanisates of large shape factor, the onset of cracking is typically followed by a number of small stress fluctuations because of continued crack propagation; such an example is seen at a shape factor of 3.4. In some cases,

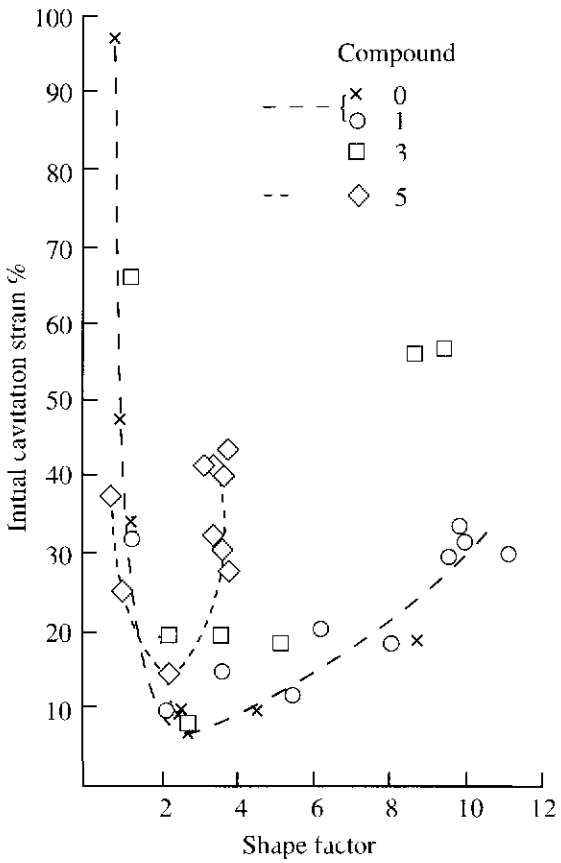

Figure 5 Typical initial cracking strain behaviour plotted against shape factor for four vulcanisates in Table 1

a small reduction in stress occurs following the onset of cracking as seen in the test piece of shape factor 5 4

The magnitude of the stress fluctuations caused by cracks is dependent on the shape factor of the test piece For large shape factors a large number of small cracks form The magnitude of individual stress fluctuations is small, though these fluctuations can be large in number, and approximately correspond to the number of cracks observed in sectioned samples Individual cracks in large shape factor test pieces typically reduce the stress by about 1% of the initial cracking stress as indicated in *Figure 2* As the shape factor decreases, the number of internal cracks reduces and they tend to become larger in size The number of internal cracks present in low shape factor test pieces can be small, as indicated in *Figure 2* where for a shape factor of 1 2 only one major and one minor crack are present The presence of such a single large crack can result in a significant reduction in stress typically of the order of 25% of the initial cracking stress

For test pieces of similar shape factor, the magnitude of the stress fluctuations due to internal cracking is found to depend on the quantity of carbon black present in a vulcanisate Unfilled vulcanisates produce the largest stress fluctuations, as filler content is increased, the magnitude of the stress fluctuations and thus damage by internal cracking tends to diminish (*Figure 1*) Few large cavities were detected in vulcanisates with high filler content In intermediate and highly filled vulcanisates, although most stress fluctuations occur just after the onset of cracking, they continue to occur at intervals between the initial cracking stress region and the breaking stress, whereas in unfilled and lightly filled vulcanisates, they are limited to the initial cracking stress region The effect of a crack on the stress retention of a highly filled vulcanisate (*Compound 4*) is seen in *Figure 1*, a crack has partially propagated through a test piece leaving about 70% stress retention In moderate and highly filled vulcanisates where internal crack propagation leads to 'break', some rubber normally near the free surface, remains undamaged and provides a small amount of continued stress retention In unfilled and lightly filled vulcanisates failure is by separation at the rubber-adhesive interface and the bond is completely severed

Effect of Displacement Rate on Cracking Stress

Since natural rubber is a viscoelastic material, it is of interest to determine whether the cracking stress is significantly affected by the rate of loading of a test piece The stress was found to increase approximately linearly, with the log of loading rate The results obtained for a high damping vulcanisate (*Compound 5*) are shown in *Figure 6* for a range of cross head

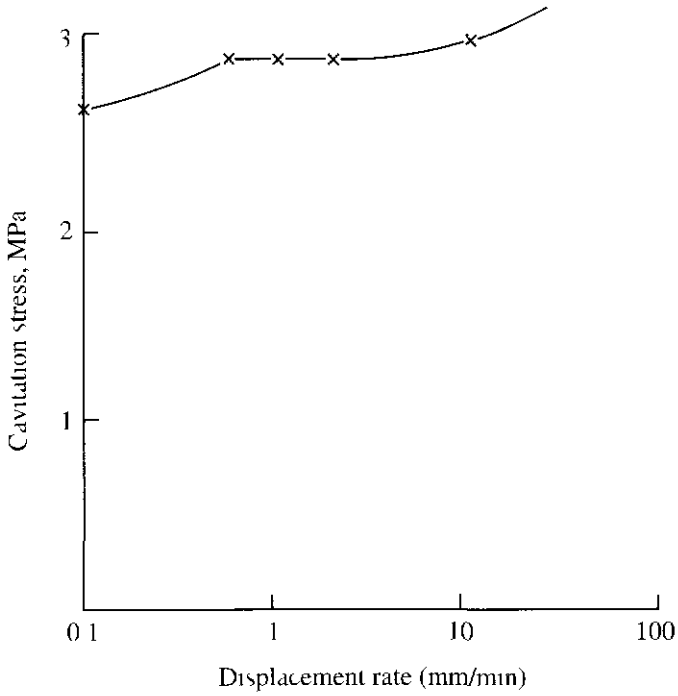

Figure 6 Initial cavitation stress plotted against displacement rate for a highly carbon black filled vulcanisate (Compound 5) *Shape factor 3 5*

speeds of 0 1–25 mm/min Although changes in loading rate have some influence on cracking stress, the effect is not large, *Compound 5* for example shows an 8 5% increase in the stress per factor of ten increase in loading rate

Internal Crack Propagation and Distribution

For unfilled and lightly filled vulcanisates, a typical crack takes the form shown in *Figure 7* The two crack faces are for a test piece moulded from *Compound 1* The crack length is approximately 3 mm the right hand face shows the initial cavity site at the intersection of *A* and *B* Crack propagation is indicated by concentric contour lines in the rubber emanating from the initial site

Crack number and length are strongly dependent on the shape factor of a test piece in the manner originally described by Gent and Lindley[3], and correspond to the influence of shape factor on the magnitude of stress fluctuations discussed above For unfilled and lightly filled vulcanisates, an increase in shape factor produces increasing numbers of cracks albeit of reduced length An example of cracking in a lightly filled vulcanisate (*Compound 1*) is shown in *Figure 8* for shape factors between 1 2 and 11 All the test pieces were loaded until break, which was by separation at the rubber bonding adhesive interface shown Large numbers of small length cracks are present in the largest shape factor specimen with crack numbers reducing, but increasing in length as the shape factor is reduced The major cracks in each test piece were formed when the applied stress reached or just exceeded the initial cracking stress, as confirmed by separate measurements on similar vulcanisates Most of the major cracks have propagated from one bonded surface to the other The secondary cracks or in some cases cavities appear to develop just prior to break, and are normally only found close to the rubber-bond

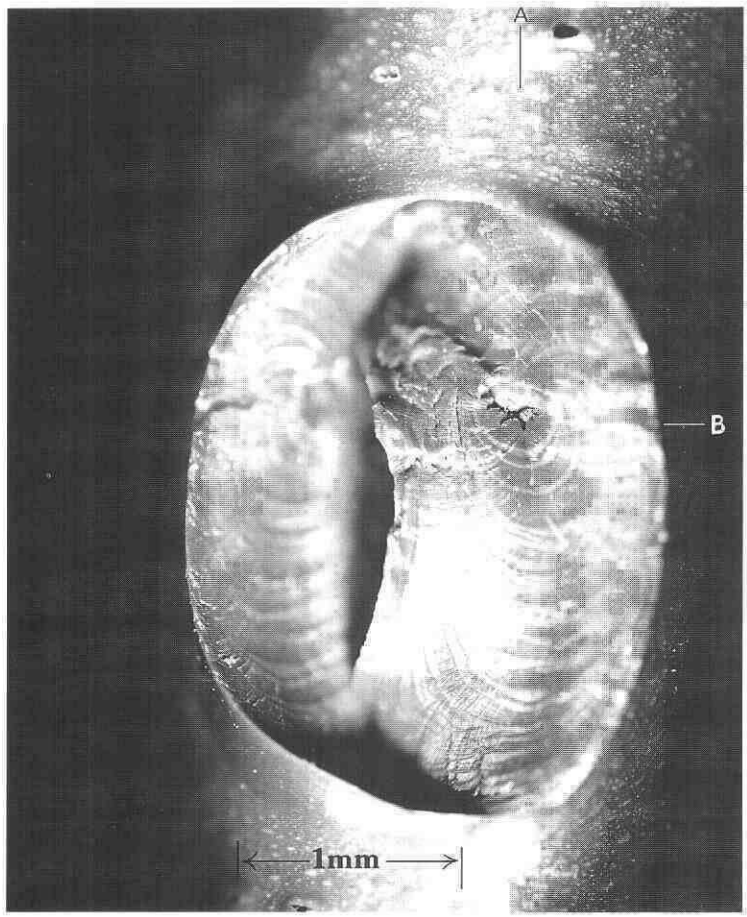

Figure 7. A typical cavitation crack in a lightly carbon black filled vulcanisate (Compound 1) *pulled to slightly in excess of the cavitation stress. The test piece is shown solid to reveal the two crack faces.*

adhesive interface. These secondary cracks are not seen close to major cracks, where the stress has been relieved.

Sectioning of test pieces stressed almost to break indicated that, in some cases, further cracks develop from the major cracks. The additional cracks are normally regularly spaced, and tend to propagate from alternate sides of the primary crack at an angle of approximately 90°. An example of such crack development in a test piece moulded from *Compound 1* is shown in *Figure 9.*

The crack surface in vulcanisates with large filler loadings (80 p.p.h.r. N330) is quite different in appearance from those for unfilled or lightly filled compounds. Crack surfaces in highly filled vulcanisates (*Compounds 4* and *5*) do not show the characteristic contour lines found in unfilled and lightly filled compounds but rather, a comparatively featureless smooth surface. Such a surface is shown in *Figure 10* for *Compound 5*, with one of the few cavities observed in this vulcanisate. The often reported feature of 'knotty' tearing in natural rubber vulcanisates containing intermediate

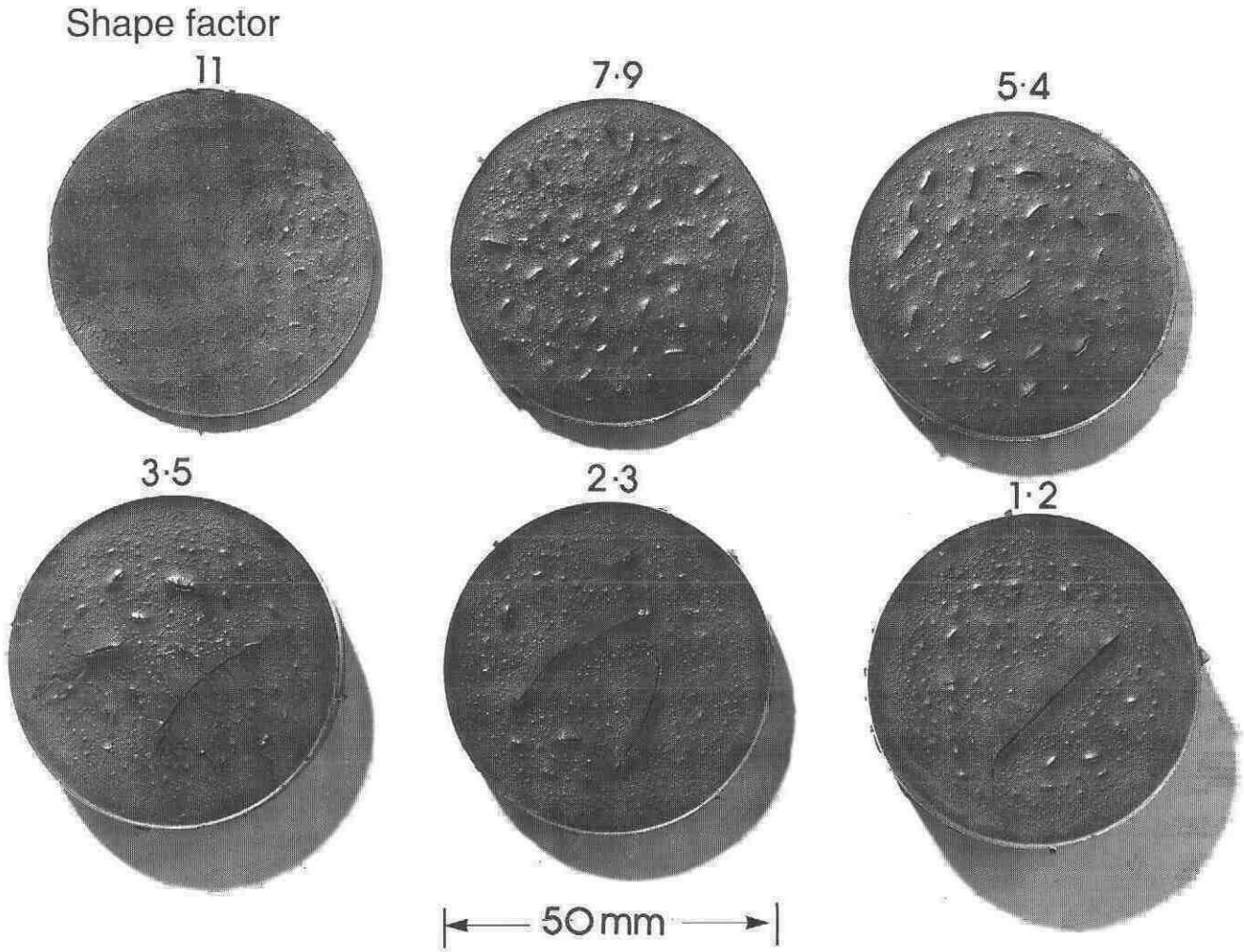

Figure 8. Crack distribution in a lightly carbon black filled vulcanisate (Compound 1) *pulled to break for test pieces of different shape factors.*

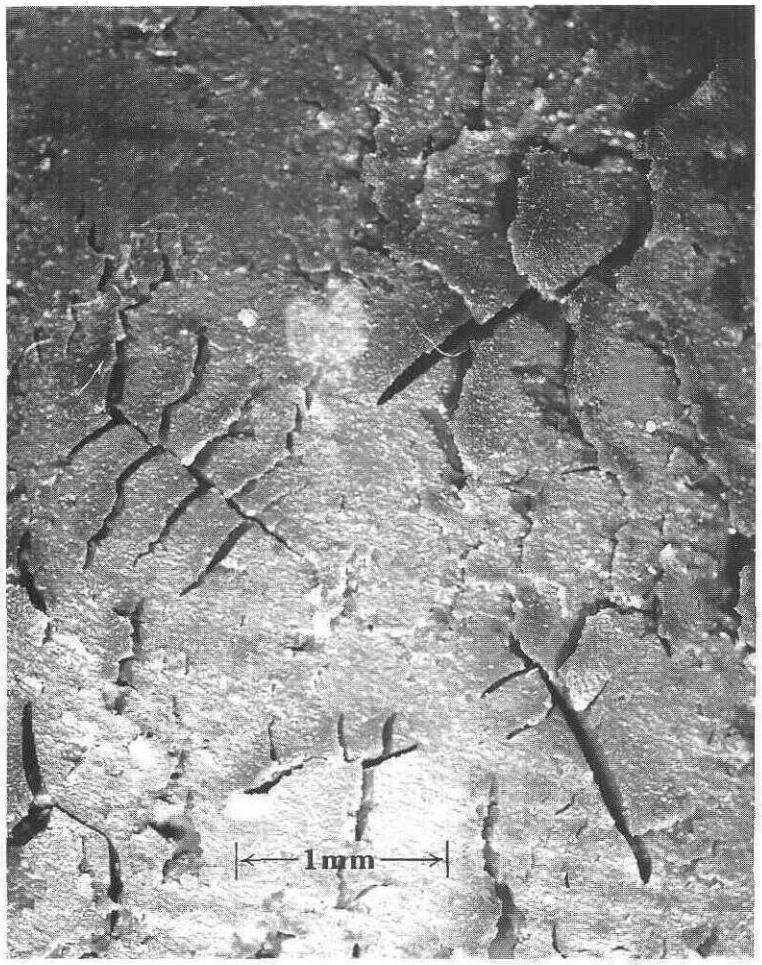

Figure 9. A close-up view of cavitation cracks in a lightly carbon black filled vulcanisate (Compound 1). *At a stress approaching break, secondary cracks can propagate from the primary cracks normally at an angle of approximately 90°.*

quantities of carbon black filler appears to be largely absent despite *Compound 5* being highly filled.

Cavity size and distribution in vulcanisates containing intermediate carbon black loadings are influenced by shape factor in a broadly similar manner to the variation observed for unfilled and lightly filled vulcanisates. For small shape factors, of the order of one, cavity size is typically at a maximum, and tends to reduce as shape factor increases. The number of cavities also tends to increase with increase in shape factor. An example of cavity distribution in a vulcanisate with an intermediate carbon black loading (*Compound 3*) is shown in *Figure 11*. The test pieces have been pulled to break and subsequently cut open for inspection. At low shape factors, the tendency is to produce a single cavity of large diameter, the example shown having a 5 mm diameter cavity near the centre. As the shape factor

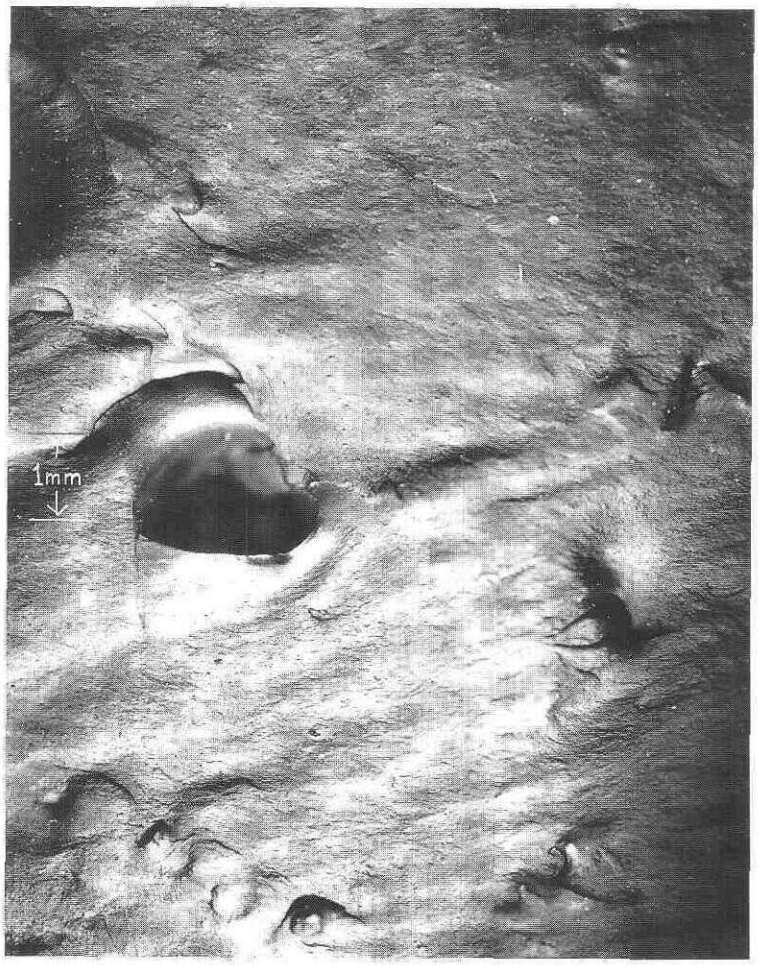

Figure 10. A typical crack face and cavity in a highly carbon black filled vulcanisate (Compound 5) *pulled to break. The crack face is relatively featureless when compared to an intermediate filled vulcanisate.*

increases, cavities are distributed at fairly regular intervals across the width of the test piece but are not present close to the rubber's free outer edge, because of the lower hydrostatic tension there. Cavities are also found close to the rubber-bond interface, but their size is largely independent of shape factor unlike the cavities formed within the bulk of the rubber. Close proximity to the rigid bond surface appears to limit cavity size. Natural rubber vulcanisates containing intermediate amounts of filler (some 20–50 p.p.h.r. N330) produce cavities consisting of a very complex structure. An example of such a cavity is shown in *Figure 12* (*Compound 3*) where the inner surface of the near spherical void consists of a multitude of crack faces. This type of crack structure was originally observed by Gent and Lindley[3] and appears to be a three-dimensional form of knotty tearing. A hemispherical crack can also be seen around the smaller of the two cavities.

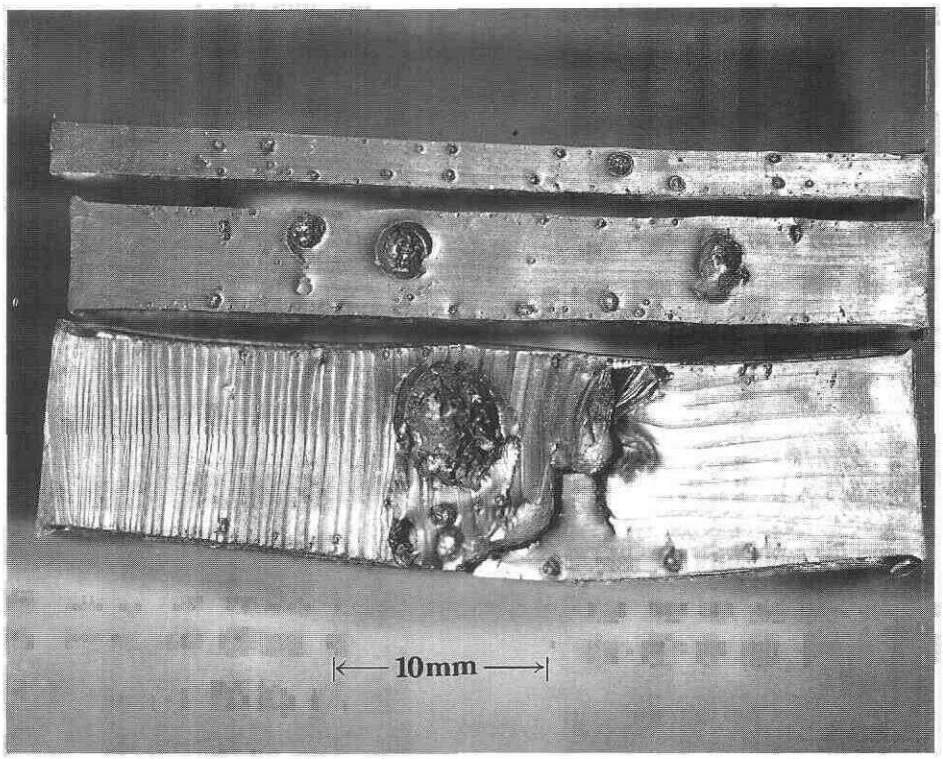

Figure 11. Cavity distribution in an intermediate carbon black filled vulcanisate (Compound 3) *for test pieces of shape factors 4.8, 2.1 and 1.1 pulled to break.*

Ultimate Failure — The Failure Modes

Three types of failure mechanism were identified:

a) Internal cracking within the rubber. This failure initiated from cavities in the rubber produced by negative hydrostatic pressure.

b) Separation at the rubber adhesive interface.

c) Propagation of cracks originating at the outer edge of the rubber.

The unfilled and lightly filled vulcanisates all catastrophically failed by mechanism b). This failure mode resulted in rubber-adhesive separation across almost the entire cross-section of the test piece with no significant remaining stress retention. In some cases, small rubber tendrils remained adhered to the metal close to the outer edge of the rubber. The entire failure process occurred very rapidly with no prior indications, and was accompanied by a loud popping sound. The factor limiting the applied stress in the case of gum and lightly filled vulcanisates therefore appears to be the strength of the rubber-bond interface (Chemlok 205/220) and not the tear strength of the rubber, or internal crack propagation through cavitation.

Test pieces containing an intermediate amount of carbon black filler, such as *Compound 3* (20 p.p.h.r. N330) also mainly failed by mechanism b). Internal crack propagation

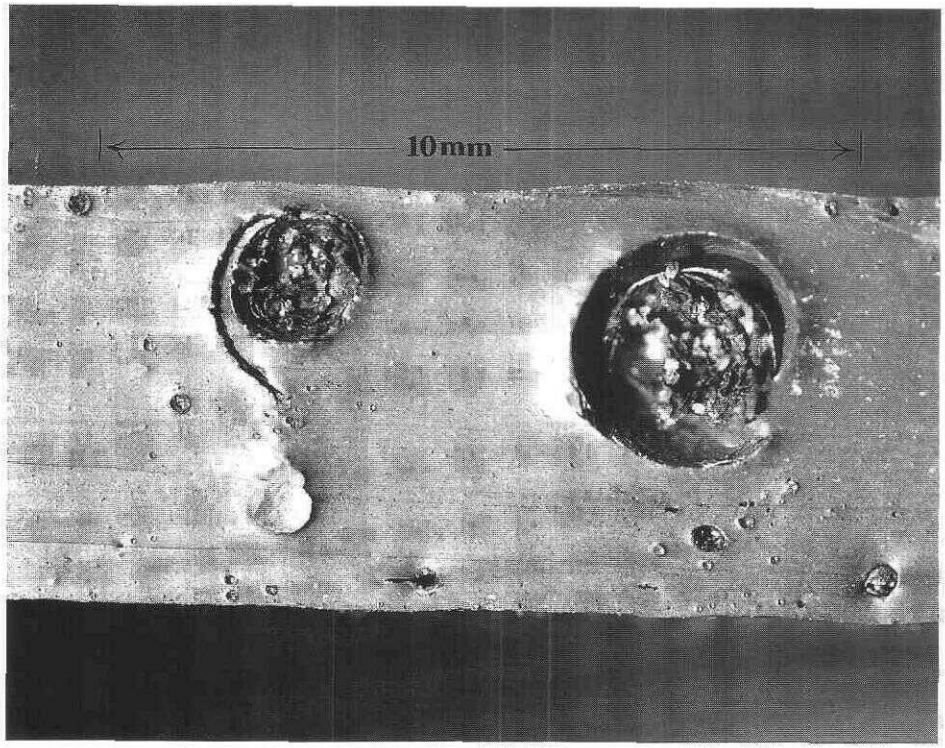

Figure 12. Typical cavities in an intermediate carbon black filled vulcanisate (Compound 3), *pulled to break, showing the complex crack structure within each void. Shape factor 2.1.*

did, however, typically occur in the rubber across a fraction of the cross section. Such compounds appear to correspond to a transition stage in the failure mechanism where both modes a) and b) can occur. Internal crack propagation by mode a), typically took place close to, but not into, the rubber-adhesive interface.

For vulcanisates with large filler loadings (*Compounds 4, 5* and *6*), the failure mode was almost always of type a). Internal cracks typically propagated, at least initially, close to the bond, but in some cases would subsequently deviate into the bulk of the rubber. The cracks appeared to initiate from the relatively small cavities formed close to the bond, such as those shown in *Figure 11*. The one exception to this was when the very highly filled vulcanisate (*Compound 4*) was tested at high shape factor; the failure then was typically of type b).

Failure mode c) appeared mainly to be associated with non-parallel metal reinforcing plates. Undulations in the thickness of the adhesive coating could also be expected to have some influence on mode c) failure at high shape factors.

Ultimate Failure — The Breaking Stress

Each vulcanisate was subjected to an increasingly large stress to induce break or catastrophic failure of the test piece. The breaking stress (σ_B) is shown as a function of shape factor in *Figure 13*. Unlike the cracking

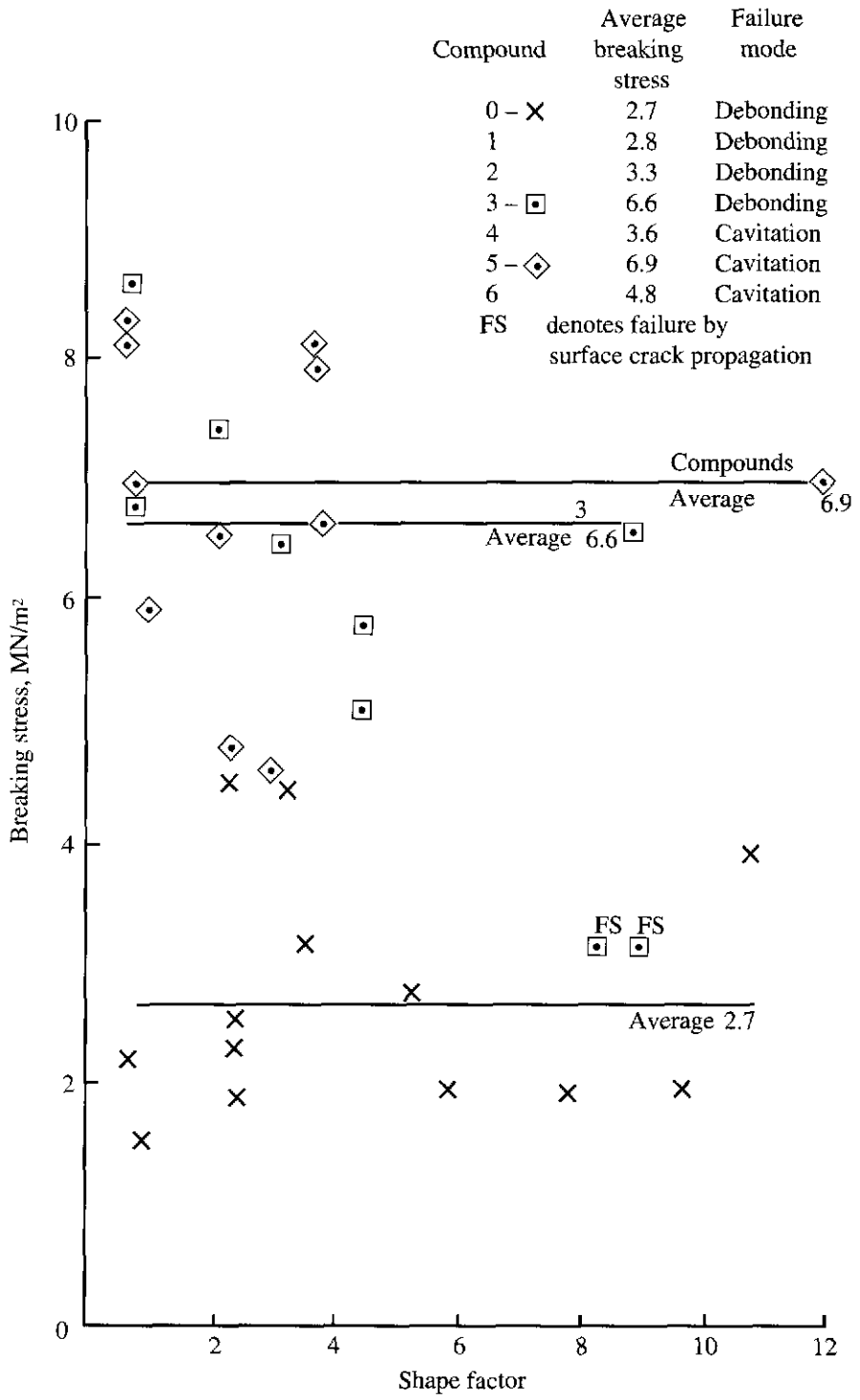

Figure 13. Breaking stress plotted against shape factor for the vulcanisate in Table 1.

stress which is relatively reproducible, breaking stress shows more variability, with σ_B values typically varying by some factor of 2, with failure by both internal rubber cracking and debonding. Average breaking stress values are also given in *Figure 13*. For the vulcanisates which failed by debonding, the breaking stress increased with filler loading. Thus it appears that an improvement in the bond between NR and the adhesive is obtained when the amount of carbon black filler (N330) is increased from about 0–20 p.p.h.r. For test pieces which ultimately failed by surface crack propagation [mode c)], the failure stress was less than that in modes a) and b). *Compounds 1, 2 and 3 with a mode c) type failure* produced an average failure stress of 2–3 MPa.

The average breaking stress is found to be approximately three times that of the cracking stress at small shape factors (about 1.5) for unfilled and lightly filled compounds.

Catastrophic Failure — The Breaking Strain

The breaking strain was investigated for each vulcanisate and results are given in *Figure 14* as a function of shape factor. Average breaking strain values are indicated by the lines. Breaking strain appears independent of shape factor, except where a type c) failure occurred in which case the breaking strain is reduced by approximately one half. Average breaking strain values for unfilled and lightly filled vulcanisates [mode b) failure] are some 360% and for highly filled vulcanisates [*Compound 4*, mode a) failure] some 60%. The values are therefore considerably less than the tensile elongation to break (e_b). Values for the average bonded breaking strain (e_c) relative to (e_b) are given in *Table 1*.

The inclusion of process oil (25 p.p.h.r. Dutrex 729) in a highly carbon black filled compound appears to increase the tear strength and breaking strain appreciably, as indicated by comparing *Compounds 4* and *5*. The tear strength and breaking strain increase from about 5.2 kN/m to 26.3 kN/m and from 59% to 208% respectively on inclusion of the oil.

Effect of Non-parallel Reinforcing Plates on Breaking Stress

Inevitably some degree of non-parallelism will occur between the metal plates during moulding. Plate misalignment leads to a non-uniform stress distribution across the rubber, with a stress concentration at the outer edge of the rubber in the region of the smallest rubber thickness. A stress concentration caused by plate misalignment together with necking of the rubber can lead to premature failure of a test piece by cracks propagating into the rubber from a surface crack *i.e.* mode c) failure. To investigate the misalignment required to initiate surface cracking, test pieces were manufactured with known plate misalignment angles and different shape factors, and pulled apart until failure. Surface cracks were normally found to propagate through the rubber close to the rubber-bond interface. With sufficiently large misalignment of plates, failure by surface crack propagation occurs, and this, not internal rubber cracking due to negative hydrostatic pressures or debonding, will normally determine the ultimate failure stress of a test piece. The degree of plate misalignment normally required to initiate failure by surface cracking was found empirically from test data to be approximately given by:

$$x \geq 0.15\, h \qquad \ldots 1$$

where h is the minimum rubber thickness and x the difference between the maximum and minimum thickness as indicated in *Figure 15*. Thus for a constant plate misalignment angle θ, surface cracking can occur when h becomes sufficiently small *i.e.* when the shape factor is sufficiently large.

CONCLUSION

This investigation has dealt with aspects of failure of bonded test pieces in tension. An attempt has been made to study the relation between internal cracking and bond strength, together with the appearance of surface cracks in order to obtain bond strength data for a particular adhesive (Chemlok 205/220) in combination with differently compounded NR vulcanisates. A principal finding is that the

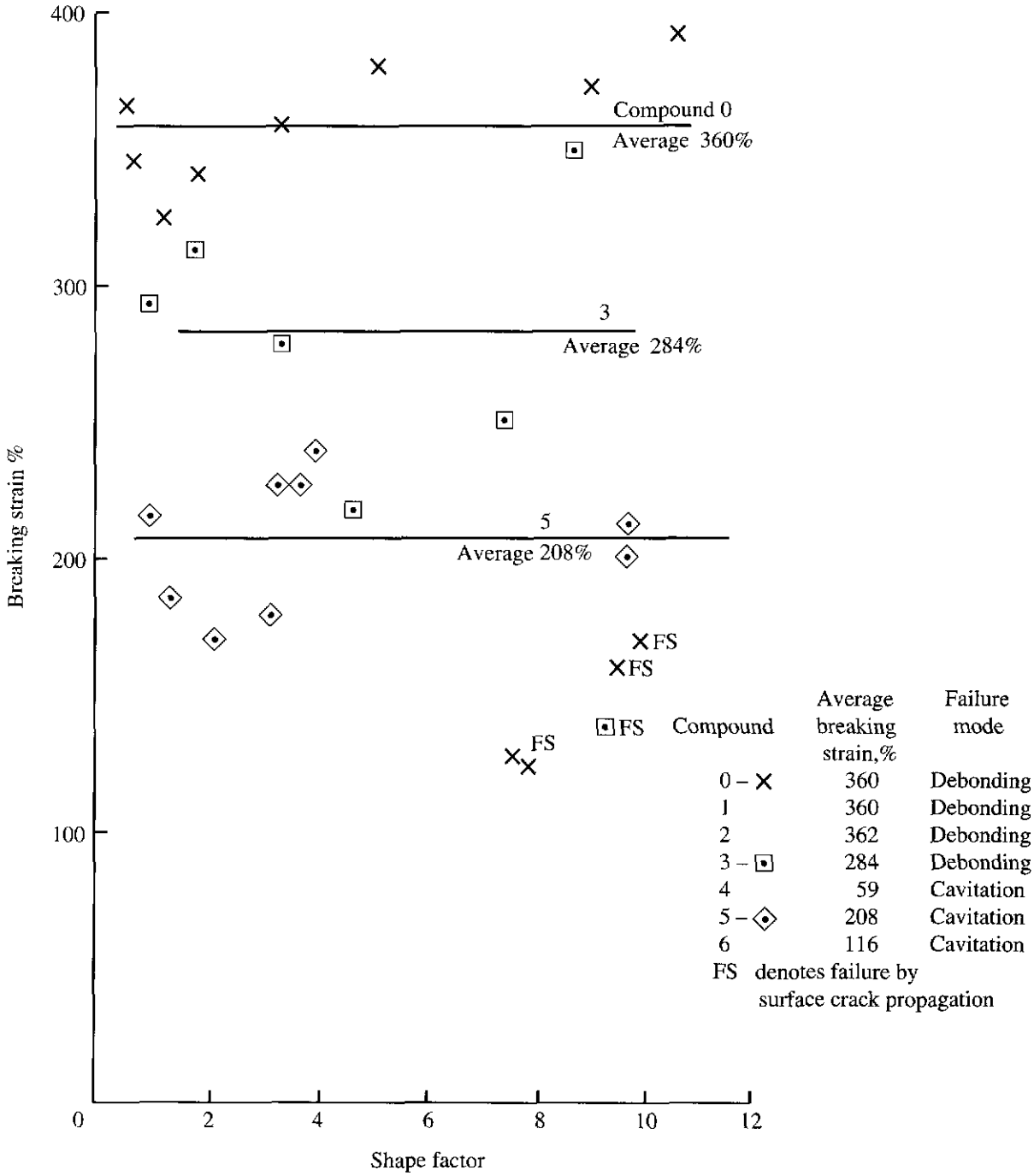

Figure 14. Breaking strain plotted against shape factor for three of the vulcanisates in Table 1.

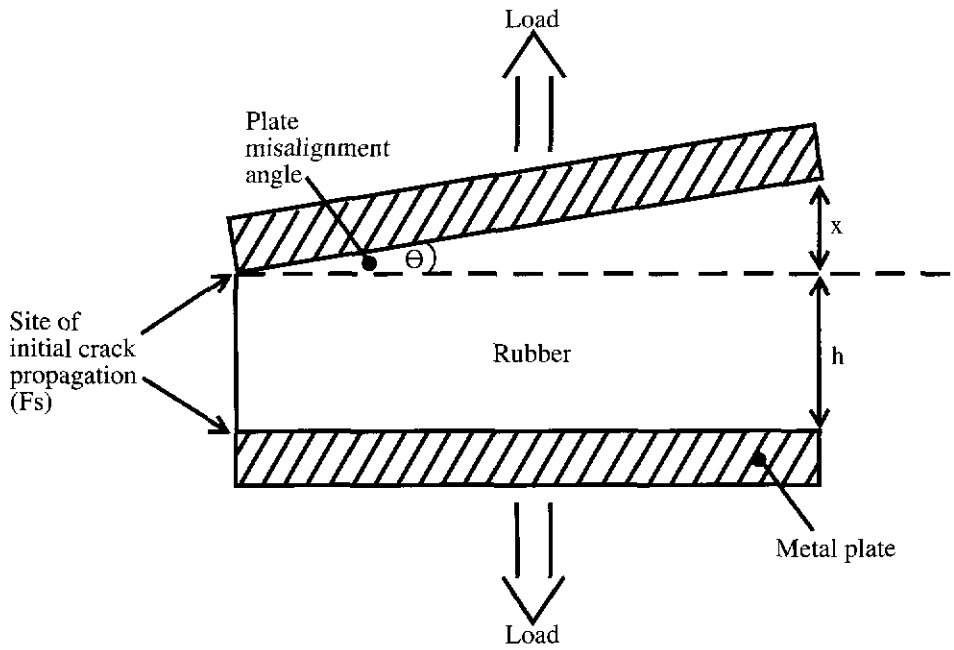

Figure 15. Diagram indicating the degree of plate misalignment normally required to initiate surface cracking (i.e. *when* $x \geq 0.15h$).

bond strength increases with the amount of carbon filler present, and with that increase the ultimate failure mechanism changes from one of interfacial debonding to internal cavitation. In this study, the change-over in mechanism was found to occur at about 20 p.p.h.r. of carbon black type N330, but it is likely to vary according to black type. If the rigid reinforcing plates in a rubber laminate are misaligned, when this component is pulled, surface cracks may appear. A criterion for their appearance is given. If cracks appear they will cause failure at a lower breaking stress well before the occurrence of interfacial debonding or internal cavitation.

The inclusion of process oil (25 p.p.h.r. Dutrex 729) to a highly carbon black filled vulcanisate was found to substantially improve tear strength and breaking strain but reduce internal cracking stress.

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