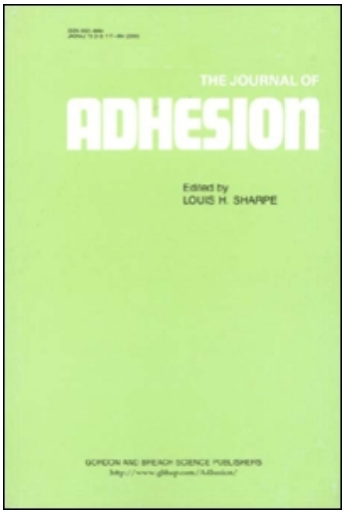

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On The Mechanics of Rubber-to-Metal Bond Failure*

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In many applications rubber is bonded to metal for fixing purposes or to alter the stiffness. Integrity of the bond is often vital to maintain the required stiffness characteristics and ensure adequate life. The mechanics of bond failure is being studied for various types of deformation. Provided tests are carried out under suitable loading conditions, time-dependent failure with a similar locus has been observed in peeling at 90° or 180°, pure shear and various combinations of simple shear and compression. There are indications that an energetics approach can enable results from different geometries to be quantitatively interrelated. Cavitation-like processes observed in the rubber in the bond region are believed to result from the constraint imposed by the metal and may be the cause of the time-dependent failure.

KEY WORDS rubber-to-metal bonding; adhesion; failure rates; geometrical effects; locii of failure; cavitation-like processes; energetics approach to failure.

INTRODUCTION

In applications such as engine mountings, bridge bearings or anti-vibration mounts, rubber is bonded to metal plates to provide a means of attachment or to modify the stiffness. The most common method of bonding is to vulcanize the rubber in contact with the metal which has previously been cleaned, prepared and coated with a suitable bonding agent. Proprietary agents usually consist of a metal primer and a top coat, which provides bonding to the rubber. High strengths can be obtained by this procedure. Nevertheless, bonds sometimes fail. In standard peel or other bond tests, failure quite often occurs well within the rubber if a strong bond has been achieved, whereas, in service, it often takes place much closer to the rubber-metal interface. In the present work, failure of bonded units has been observed to occur in a time-dependent manner under constant load. In the past, such failure has been noted due to attack by ozone or solvent at the bond[1] or to rupture within the adhesive (or at the metal surface in a corrosive environment).[2] Different test geometries were studied in the latter work but no attempt was made quantitatively to interrelate results from them although it was observed that the failure locus could change with change in geometry.

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In the present experiments, although some rupture within the adhesive or at the adhesive-rubber interface cannot be ruled out, the locus of failure is often within the rubber adjacent to the bond. The fracture is too fast to be caused by ozone cracking[1] and might, thus, be ascribed to cohesive failure in the elastomer, although an intriguing feature is that time-dependent failure is observed in strain-crystallizing elastomers which do not normally exhibit time-dependent mechanical crack growth under constant load.

EXPERIMENTAL PROCEDURE

1 Materials and Test Pieces

The materials for which results are reported were conventional accelerated-sulphur vulcanizates of natural rubber (Standard Malaysian Rubber SMR 5L) containing, respectively, no filler, 50 parts per hundred rubber by weight (pphr) medium thermal (MT) carbon black, or 50 pphr high abrasion furnace (HAF) black. In addition to the rubber and black, the other ingredients were zinc oxide 5 pphr, stearic acid 2 pphr, sulphur 2.5 pphr, cyclohexylbenzothiazyl sulphenamide 0.6 pphr, antioxidant 1 pphr.

Test pieces for peel experiments (Fig. 1(a)) in the form of bonded strips, 60 to 100 mm long, 25 mm wide and about 6 mm thick, were made by vulcanizing the compounds for

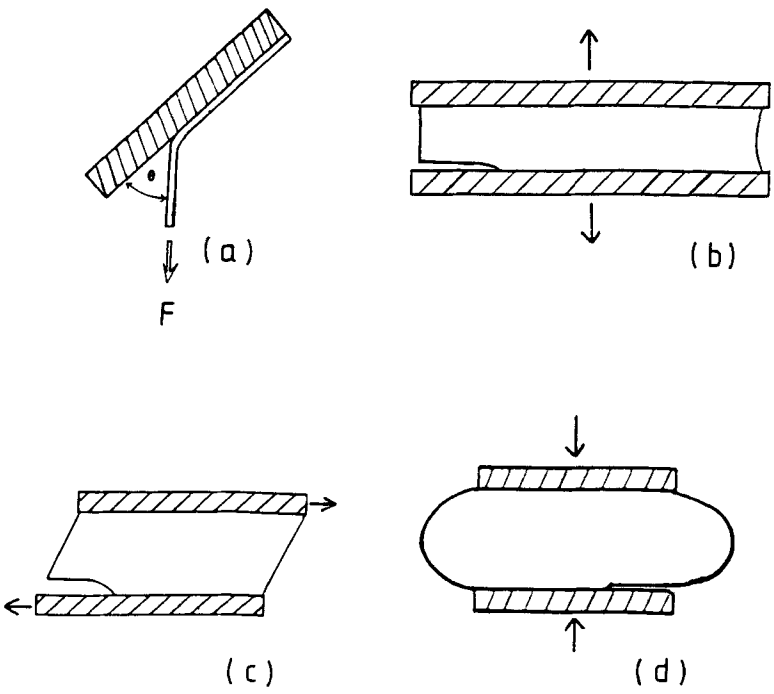

FIGURE 1 Various tests used to assess rubber-to-metal bond failure: (a) peel; (b) pure shear; (c) simple shear; (d) compression.

40 minutes at 140°C in contact with steel plates coated with bonding agents. Proprietary bonding systems- Chemlok 205® (primer)/220 or Chemlok 205/252®- referred to, respectively, as System *A* or System *B*, were used (Lord Corp. Erie, PA, USA). Chemlok 220 is based on chlorinated polyisoprene rubber and Chemlok 252 on chlorosulphonated polyethylene. In some cases steel or fabric backing was added to prevent extension of the leg during peeling, the backing also being bonded to the rubber. Similar procedures were used to form bonded pure shear test pieces with rubber about 1mm or 6mm thick (Fig. 1(b)). Bonded blocks, approximately 66 mm × 53 mm × 20 mm thick, were employed to investigate failure under simple shear (Fig. 1(c)) or compression (Fig. 1(d)) or combinations of the two.

2 Test Procedure

Peel tests were carried out at angles of 90° or 180°. All tests were performed at 21 ± 3°C, either at a constant rate of grip separation (in a Universal Instron Testing Machine), when the peel force was measured, or under constant load, when the peel rate was determined. In either case the energy release rate, G, for a crack propagating along or parallel to the interface, was calculated from the peel force per unit width, F, using the relation:

$$G = F(\lambda - \cos\theta) - Wh \tag{1}$$

where λ is the extension ratio of the peeled leg, θ the angle of peel, W the strain energy density in the leg and h the thickness of the rubber in the unstrained state (Fig. 1(a)). When backing is used, this equation simplifies, since $\lambda \to 1$ and $W \to 0$, to $G = F(1 - \cos\theta)$. In constant rate tests, the nominal peel rate calculated from the crosshead speed and the test piece geometry, making allowance for extension of the peeled leg where appropriate, was plotted against the peel energy.

Experiments on bonded pure shear test pieces (Fig. 1(b)) were carried out at fixed length, l, between the grips, with the rate of failure at the bond being measured. For a long crack at the bond (of length greater than l_0) the energy release rate is given by

$$G = Wl_0 \tag{2}$$

where W is the strain energy density in the central region of the rubber (which is in pure shear) and l_0 is the value of l in the unstrained state. Allowance was made for effects of stress relaxation, stress softening and set on W, l_0 and hence G, by carrying out retraction stress-strain curves after times representative of those used in the bond failure tests. The rate of retraction in these tests was (somewhat arbitrarily) fixed at 5 mm/minute.

Tests in simple shear (Fig. 1 (c)) and/or compression (Fig. 1 (d)) were carried out either under constant deflection or constant load. Relations similar to Equation (2) have been found to apply for the maximum energy release rate in either of these deformations (for a crack that is long compared with the height)[3-7] although there may be complications due to frictional effects.[5,15]

RESULTS AND DISCUSSION

1 Preliminary Tests on Bonded Blocks

In service, particularly in engineering applications, rubber is often used in combined compression and shear. Bonded blocks were subjected to similar deformations in the laboratory to check whether a similar locus of failure to that occurring in service could be produced. This was found to be so, failure close to a bonded interface being observed for various combinations of compression and (nominally) simple shear. Furthermore, failure occurred in a time-dependent manner under fixed deformation. For example, for a simple shear of about 150%, failure occurred at an essentially constant rate over a period of three days, after which the block was severed. With the same shear plus a compression of some 50%, severance occurred after 5 days, the longer time (despite higher energy storage) presumably reflecting the influence of friction on the energy release rate.[5] Similar failure was observed in compression alone. A fracture surface is illustrated in Figure 2(a): failure occurred close to the bond but a short distance into the rubber (typically less than 0.1 mm but extending further over some areas). The failure might be ascribed to rubber fatigue, rather than bond failure, but is unexpected since time-dependent mechanical crack growth is not normally observed in natural rubber under constant deformation (the rate of failure appears too high for ozone attack[1]).

2 Peel Tests

(i) Unfilled Rubber

Initially, peel tests were carried out in a machine at constant rate, similarly to standard tests, with a peel angle of 90° and a crosshead speed of 50 mm/minute. For the unfilled material bonded with System *A*, failure occurred well into the rubber and, with unbacked test pieces, the peeled leg subsequently broke. To try to overcome this, two other procedures were investigated. In the first, the use of metal-foil-backed test pieces prevented leg breaks but with System *A* failure still occurred well into the rubber in constant rate tests, with a crack at first growing towards the metal backing and then stopping as the tip had effectively become very blunt. Ultimately, fracture occurred at, or close to, the bond in a very rapid, quasi-catastrophic manner after the nominal energy release rate [calculated according to equation (1)] had reached a high level (Fig. 3). At this stage all of the unfailed portion of the test piece was subject to tensile stress normal to the bond plate and the fracture initiated from the rear end of the bonded section. Cavities (which remained open) were seen in the rubber side of the fracture surface after failure (Fig. 2(b)) and the metal backing was observed to have yielded. The appearance of the cavities is not dissimilar to that reported by Gent and Lindley[8] for internal cracks in bonded cylinders subject to tension, except that in the present case the cavities connect with the bonded interface. Similar behaviour was observed at various crosshead speeds except that at the lowest speed the catastrophic fracture occurred in two steps. The nominal peel energy level was little affected by the crosshead speed (or nominal failure rate) over the range covered, as the results in Figure 3 illustrate. In view of gross departures from the failure locus assumed in

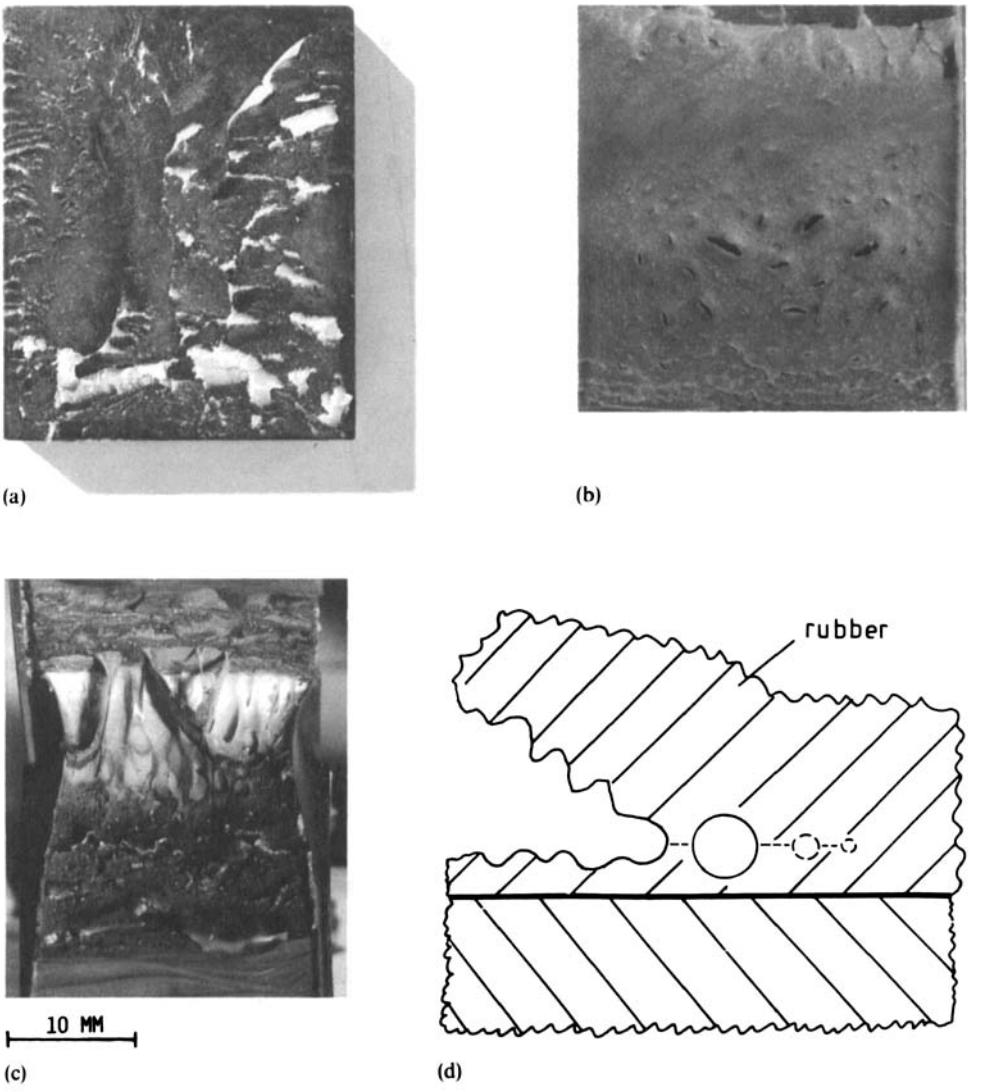

FIGURE 2 Various fracture surfaces (unfilled vulcanizate): (a) steel plate from a simple shear failure; (b) rubber surface from a constant rate test on a backed test piece showing cavities; (c) peel front region from a constant load test on a backed test piece, showing large cavities; (d) schematic diagram showing cavitation in the crack tip region that may lead to near-bond or "thin-rubber" failure.

deriving Equation (1), large fluctuations in the rate of peeling and yielding of the metal backing, little significance should be attached to the quantitative peel rates and peel energy levels given in Figure 3.

The second alternative procedure involved using unbacked test pieces under constant load (below the level at which leg breaks occurred). In this case, with System *A*, failure proceeded in a time-dependent manner along a locus close to the bond, similar to that observed in the shear and compression tests. The rate of failure increased rapidly

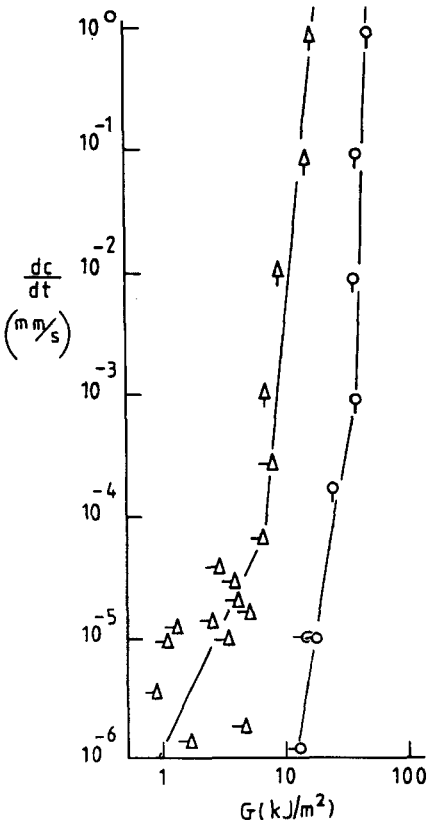

FIGURE 3 Time-dependent peel rate, dc/dt, *versus* strain energy release rate, G, from 90° peel tests for unfilled natural rubber bonded to steel with System *A*: constant rate tests ♀, constant load tests –○; System *B*: constant rate tests △, constant load tests –△ (backed test pieces were used throughout; logarithmic scales).

with energy level, the results in Figure 4 showing about a sixth-power dependence. As can be seen, the peel energy levels for the constant load tests on the unbacked test pieces are well below the nominal values for the constant rate tests on the backed test pieces. As noted above, the latter are very uncertain but to investigate further this discrepancy, constant load tests were carried out on backed test pieces. Time-dependent failure was again observed but the average rates (plotted in Fig. 3) were much below those for the unbacked test pieces (at the same peel energy level) and, surprisingly (and presumably coincidentally) appear consistent with the nominal constant rate test results (Fig. 3). The locus of failure in the constant load tests on the backed test pieces was approximately parallel to the bond but observation of the peel front zone showed large cavities to be present over an extensive region (Fig. 2(c)). It is evident that the foil backing not only prevents extension of the peeled leg but also increases the bending stiffness of the leg substantially, so that the extent of the high-stress region at the peel front (for a given peel energy) is greatly increased compared with that for an unbacked test piece. The differences in peel energy level seen in Figure 4 presumably also reflect this. A further feature of the time-dependent failure in the backed test pieces was that failure

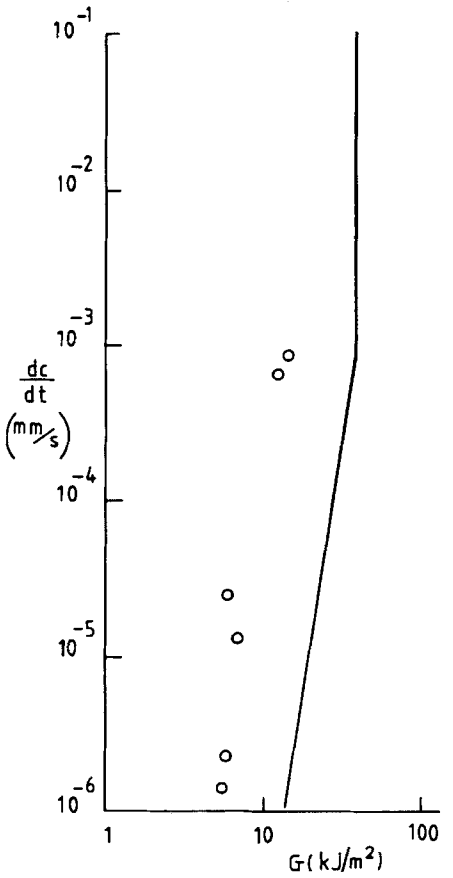

FIGURE 4 Peel rate, dc/dt, *versus* strain energy release rate, *G*, from 90° peel tests for unfilled natural rubber bonded to steel with System *A*: constant load tests on unbacked test pieces ○, line for backed test pieces (from Fig. 3)—.

occurred in a stick-slip manner on a large scale (steps of the order of several mm), the rate during the slip stages being some two orders of magnitude higher than the average. This appeared to be associated with partial failure in the region immediately ahead of the tip (*cf.* Fig. 2(c)), many cavities being present with unbroken strands of rubber in between (the foil backing provides additional constraint for the rubber in its vicinity). In the unbacked test pieces, the scale of the high stress region is less but strands are also seen at the crack tip and some cavities are observed. The critical dilatational stress required to produce cavitation would be expected to increase when the high-stress region is small.[9,10] Whether time-dependent failure proceeds in a stick-slip manner *via* smaller-scale cavitation-like processes for unbacked test pieces is not known at present but this appears to be a distinct possibility. Delayed cavitation at stresses below the critical value has been reported.[9] The type of cavitation process envisaged is illustrated schematically in Figure 2(d). Propagation of failure

through cavities formed near the metal interface would leave a thin coating of rubber on the metal.[11] The process might resemble that observed in glassy materials at elevated temperatures.[12]

For backed test pieces of the unfilled rubber bonded with System *B*, failure in constant rate peel tests was different from that with System *A* in that steady failure was observed. Although some excursions well into the rubber occurred, mostly the locus of failure was close to the interface within an apparently modified rubber layer. Peel energy levels with System *B* were lower than the nominal values with System *A* but the peel rate *vs* energy release rate relationship (Fig. 3) is presumably more meaningful. Constant load tests gave a similar locus of failure, with lower energies and rates consistent with essentially the same relationship (Fig. 3). The apparently modified layer for the unfilled natural rubber showed as a distinctive orange colour which was generally present on both rubber and metal sides of failure, suggesting that failure was often within it. The large variability of the results in the constant load tests reflects weaker bonding in some regions than others (the crack front was seen to run ahead in weak regions). A not dissimilar variability was observed in constant rate tests (*cf.* Fig. 5) although this is not evident in Figure 3 where average peel energy levels over the entire failure region are plotted for these tests. Whether the occurrence of weakly-bonded regions was due to faulty preparation technique or is inherent for the unfilled rubber with System *B* is not clear at present.

(ii) Carbon-Black-Filled Rubber

Results from constant rate tests for a vulcanizate containing 50 pphr of a large particle black (MT) with both bonding systems are shown in Figure 6. As with the unfilled rubber, leg breaks again occurred in this type of test if backed test pieces were not used. Differences between System *A* and System *B* are less with the MT-filled rubber although it appears that System *B* still tends to give somewhat lower bond strengths. The loci of failure were similar for the two systems, tending to be in the rubber, to an extent that decreased with decreasing rate, in constant rate tests. In constant load tests with System *A*, time-dependent failure was again observed with the locus still apparently being mainly within the rubber although close to the bond. The constant load results

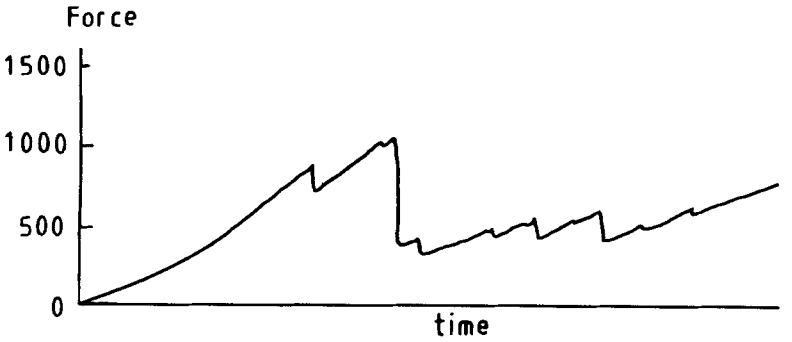

FIGURE 5 Typical force-time trace for a constant rate peel test.

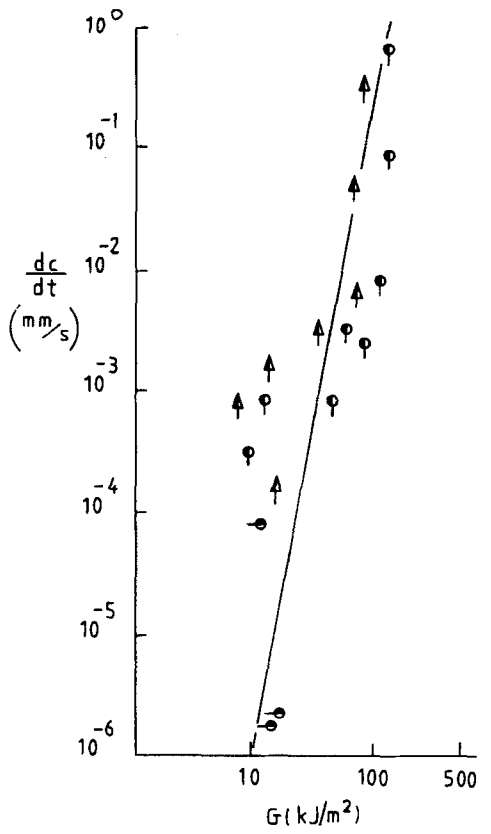

FIGURE 6 Peel rate *versus* energy relationships from 90° peel tests for the natural rubber vulcanizate containing 50 pphr of medium thermal black bonded to steel with System *A*: constant rate tests ●, constant load tests ⊖, or System *B*: constant rate tests ▲ (steel foil backed test pieces were used throughout).

appear consistent with the constant rate ones. Considerable variability is shown with both bonding systems for this material.

Results for a vulcanizate containing 50 pphr of a fine particle (HAF) black with the two bonding systems are shown in Figure 7. With this material, leg breaks again occurred with unbacked test pieces in constant rate tests but very little difference between the two bonding systems was observed for backed test pieces. In these tests, the locus of failure is well into the rubber with both bonding systems. In a constant load test (with System *B*), time-dependent failure, with a locus closer to the bond, was again observed.

(iii) Effects of Peel Angle

All of the peel tests described above were carried out with a nominal peel angle of 90° (with the normal procedure, small changes in angle occur during a test, but these would affect the peel energy level only slightly). The marked changes in the locus of failure that

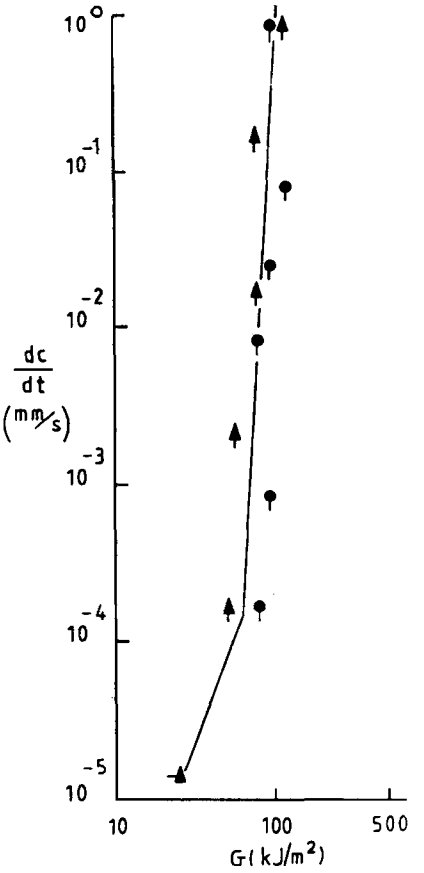

FIGURE 7 Peel rate *versus* energy relationships from 90° peel tests for the natural rubber vulcanizate containing 50 pphr of HAF black bonded to steel with System *A*: constant rate tests ●, or System *B*: constant rate tests ▲, constant load test ⊣▲(backed test pieces throughout).

have been observed under different test conditions undoubtedly have several causes. One factor that has been suggested previously[11,13] is that a transition in the failure locus occurs for peel angles in the vicinity of 90°. Thus, experiments nominally at this angle might be rather sensitive to small variations. Constant rate experiments over a range of peel angles from 30° to 180° lend some support to this view. Figure 8 shows the peel energy plotted against peel angle for an approximately constant peel rate for fabric- backed test pieces of the unfilled vulcanizate bonded with System *A* (the crosshead speed was adjusted with peel angle in these experiments so that the peel rate would have been within the range 4–5 mm/s irrespective of angle for steady-failure along a locus parallel to the bond). The peel energy is not constant, as might have been expected, but increases progressively with increasing peel angle. Variations in failure locus also occurred. For angles up to 90°, fracture occurred fairly close to the bond, with progressively increasing amounts of rubber being left on the metal with increasing angle, whereas, for 120° and above, the locus of failure moved well into the

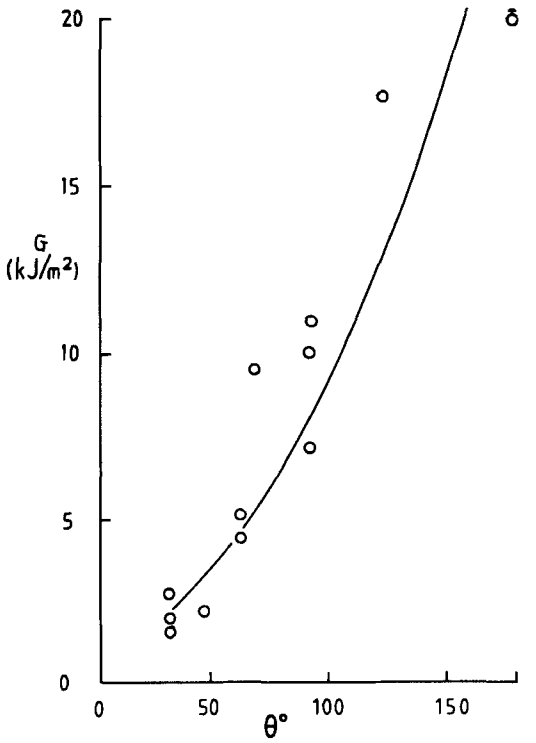

FIGURE 8 Peel energy (G) from constant rate tests at various peel angles (θ) for the unfilled natural rubber vulcanizate bonded to steel with System *A* (fabric backed test pieces, peel rate about 0.1 mm/s, linear scales); at 180° the backing detached before G had stabilized.

rubber (Fig. 9). Some evidence of a cavitation-like process, with strands or webs of intact material remaining well behind the fracture front, was also seen. [NOTE: The results at 90° in these tests differ from those discussed earlier for the unfilled elastomer with the same bonding system (the only material difference being the use of fabric rather than steel foil as the backing material). The difference in the results seems to be mainly associated with a change in the locus of failure, which moved into the bulk rubber at a peel angle between 90° and 120° in the present tests (*cf.* Fig. 9).]

As noted above, constant load experiments suggest that changes in the locus of failure are not simply a matter of the peel angle. Further evidence of this is provided in Figure 10 which compares constant load test results over a range of rates and peel energy levels for peel angles of 90° and 180° for the vulcanizate containing 50 pphr MT black bonded with System *A*. The peel energy levels are well below those for backed test pieces (*cf.* Fig. 6), for the same reason as discussed earlier for the unfilled rubber, but are very similar for the two angles, in contrast to the constant rate test results of Figure 8. Fracture surfaces for the two peel angles are shown in Figure 11 and can be seen to be very similar, again in contrast to those from the constant rate tests (*cf.* Fig. 9). Thus, when the locus of failure remains the same, results for different peel angles can be correlated by means of an energetics approach.

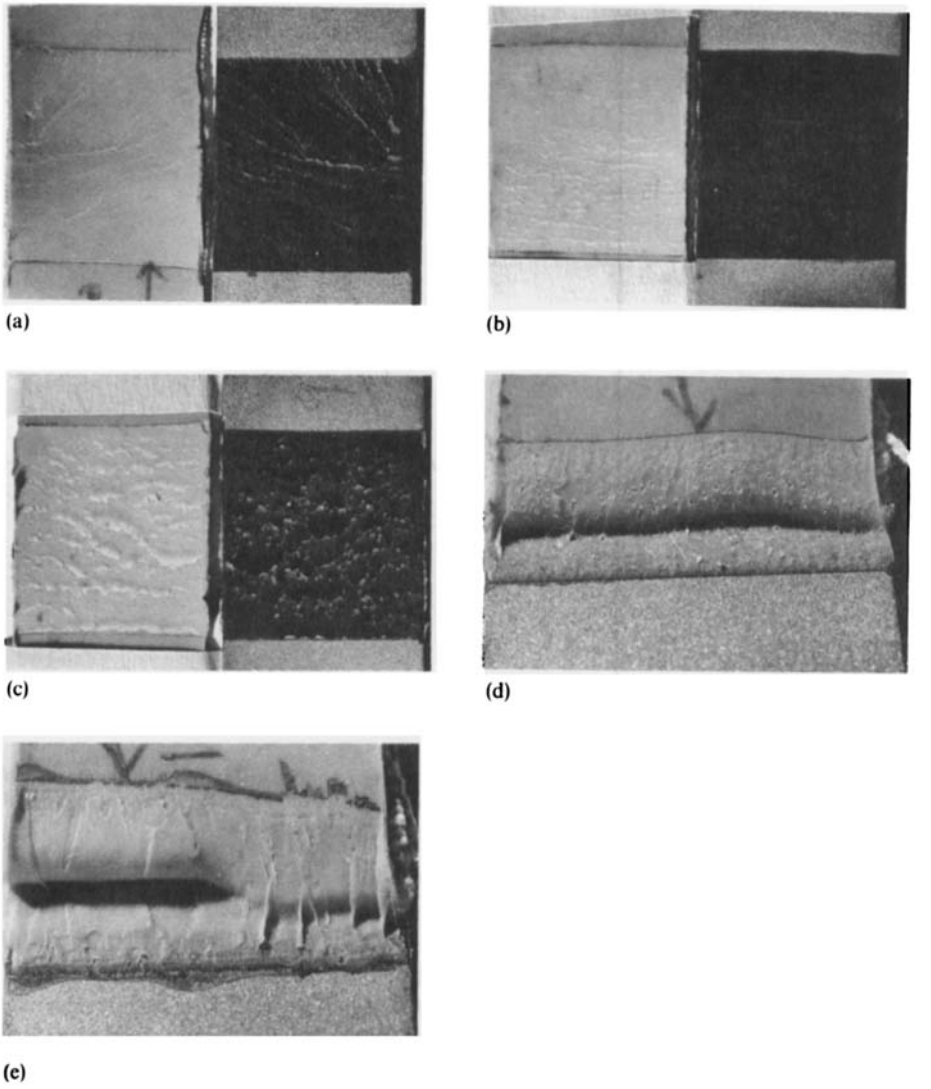

FIGURE 9 Fracture surfaces (both bulk rubber and steel plate sides) from constant rate peel tests at various angles: (a) 30°, (b) 60°, (c) 90°, (d) 120°, (e) 180°.

3 Results in Shear

(i) Pure Shear

As discussed above, there is evidence that a type of cavitation process, probably associated with dilatational stresses arising from the constraint applied by the metal, may be involved in time-dependent bond failure. The dilatational component of the stress will be affected by the thickness of the rubber and, to investigate its possible effect on failure, experiments have been carried out using pure shear test pieces (Fig. 1(b)) of

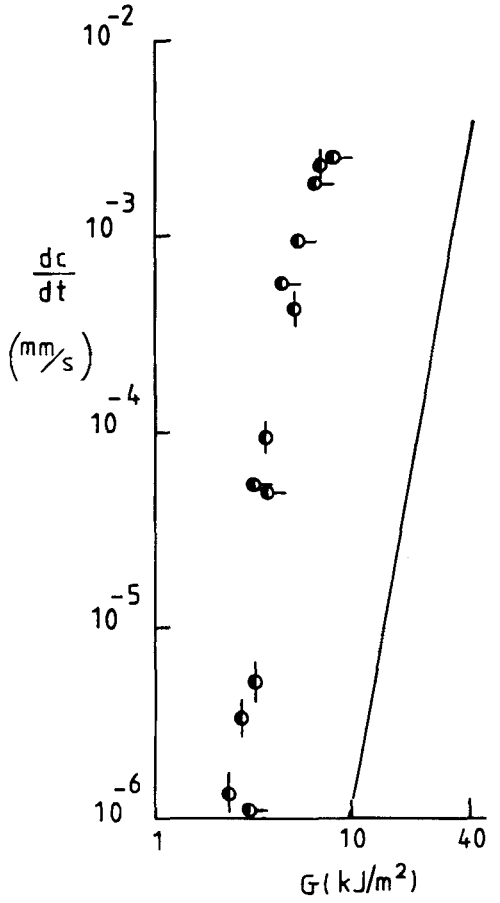

FIGURE 10 Peel rate *versus* peel energy from constant load tests on unbacked test pieces of the MT-black filled vulcanizate bonded to steel with System *A*: peel angle 90° ◐, 180° ◐; line for backed test pieces from Figure 6 —— (logarithmic scales).

different thicknesses. With 1 mm-thick test pieces, whether unfilled or filled, in most experiments either no failure was observed (at lower energies) or failure was cohesive, through tearing in the rubber. In two cases of relatively long-term experiments, very slow failure in the vicinity of the bond was observed; this failure was attributable entirely to ozone attack.[1]

With 6 mm-thick pure shear test pieces, time-dependent near-bond failure was observed with all three materials used. Results for the unfilled material, using energy release rates calculated from Equation (2), are shown in Figure 12 where they are compared with the 90° peel test results for unbacked test pieces from Figure 4. As can be seen, the two sets of results are very similar, although the pure shear results tend to be displaced slightly to the right. This may simply be because the thickness of the pure shear test pieces was less than the width of the peel test pieces (*ca.* 25 mm). In calculating the peel energy levels from Equation (1) [for unbacked peel test pieces] or Equation (2),

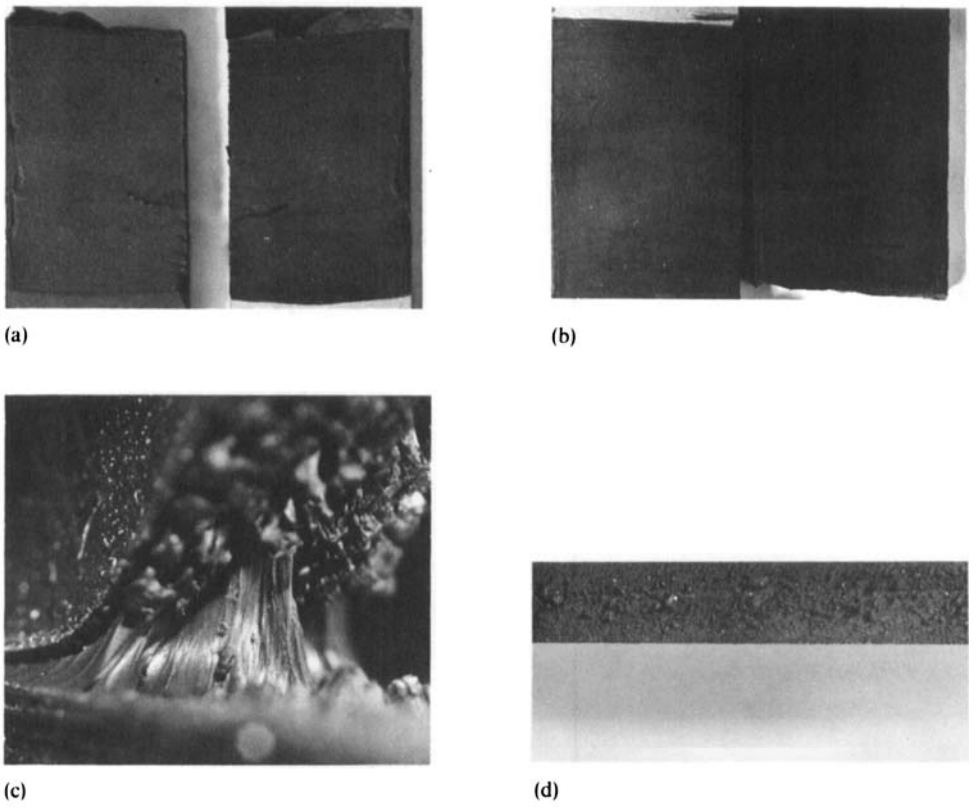

FIGURE 11 Fractography: fracture surfaces from constant load peel tests on the MT black-filled vulcanizate (unbacked test pieces): (a) 90°, (b) 180°; constant extension pure shear test on a 6 mm thick test piece of the HAF black-filled vulcanizate: (c) crack tip region ($G \simeq 8\ \mathrm{kJ/m^2}$); (d) fracture surface.

it is necessary to make allowance for effects of stress relaxation and set occurring during the bond failure tests, as noted earlier. This has been done for the results for the unfilled elastomer shown in Figure 12, but is considerably more difficult for the filled materials owing to the greater complexities of their stress-strain behaviour. As a result, it has not yet been possible to make accurate comparison of results for different geometries for the filled materials, but the nature and rates of failure observed in pure shear tests were very similar to those for the unfilled material. Figure 11 (c) shows the region near the tip of the failure front for a filled pure shear test piece, with much strand formation and cavitation, while Figure 11(d) shows a fracture surface.

(ii) Simple Shear Result

For a crack growing parallel to the bonded surfaces in a simple shear block that is long compared with the height (l_0) of the rubber, the energy release rate may approach the value Wl_0 given by Equation (2), as noted earlier. Estimates of the energy release rates for shorter cracks in rectangular blocks were made by Lindley and Teo,[3] who found

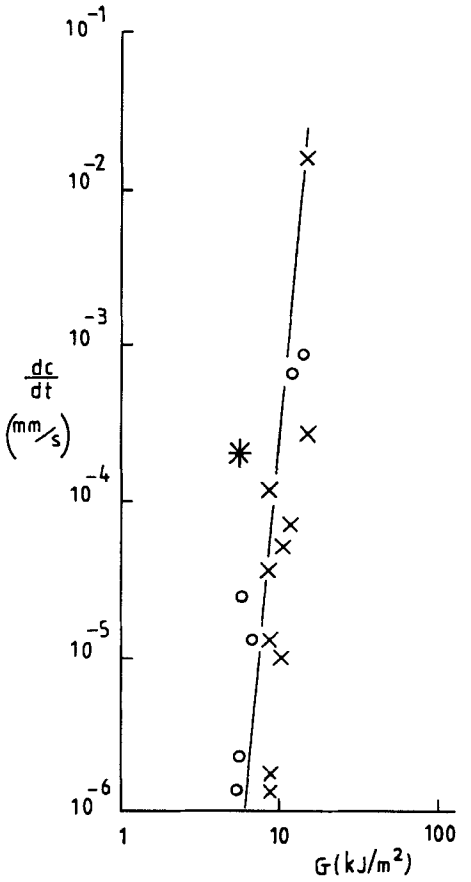

FIGURE 12 Failure rate, dc/dt, *versus* strain energy release rate (G) for the unfilled vulcanizate bonded with System *A* for various test geometries: 90° peel tests under constant load (unbacked test pieces, from Fig. 4) ○; constant extension pure shear results (6 mm thick test pieces) ×; approximate result from simple shear ✳.

quite a complex relationship using finite element methods. More recently, Gent and Wang[15] have computed broadly similar results for coaxial cylinders, although they found that under some circumstances the energy release could be less than Wl_0 even for a long crack. Two-fifths of this value (the result obtained by Lindley and Teo for very short cracks) is taken as representative for the failure of the blocks used in the present investigation. The average rate of growth for the preliminary experiment in simple shear described earlier is plotted against this energy release rate in Figure 12. As can be seen, the simple shear result lies close to the other results. Although no great significance can be attached to this because of the quantitative uncertainties, it is of interest that a result from a quite different geometry again, appears to lie in the same region of the rate-energy relationship. [NOTE: Although the application of a small simple shear does not introduce dilatational stresses in the bulk (even near the bond) such stresses will arise in the crack tip region.]

CONCLUSIONS

In peel testing of rubber-to-metal bonds, the locus of failure has been found to vary according to the method of loading. Whereas failure often runs into the bulk of the rubber under the most widely-used loading method, in which the test piece is pulled apart at a constant rate, time-dependent mechanical failure near to the bond is observed if a constant force of sufficient magnitude is applied. The difference is probably associated with the rate of failure rather than the loading method as such. Similar behaviour is observed with both unfilled and filled vulcanizates.

Although the time-dependent failure occurs near to the bond, the failure locus generally appears to lie slightly within the rubber (typically, at a depth of a few tenths of a millimetre). Under suitable loading conditions a similar locus is observed for various geometries: peel at various angles, pure shear (if the test piece is sufficiently thick) and various combinations of simple shear and/or compression.

The observed time-dependence of the failure is surprising in that the rubber used is strain-crystallizing natural rubber which normally does not show such cohesive failure under constant load. A possible explanation is that a modified layer of rubber is formed due to interaction with the bonding agents. Some evidence of a modified layer has been observed with one of the bonding systems used. The relative performance of the two bonding systems studied appears to vary with the type and amount of filler incorporated in the rubber.

Use of a metal-foil backing on peel test pieces to obviate failure in the rubber is found to give increased peel energies, perhaps mainly because of the increase in bending stiffness. Large-scale cavitation-like processes are observed in the peel front region. The dilatational stress would also be increased by the constraint applied by the backing.

It appears that cavitation-like processes may also occur in the peel front region on a much smaller scale, during near-bond failure for unbacked peel test pieces. There is some visual evidence of this. In addition, with pure shear test pieces, time-dependent failure of this type is observed with relatively thick test pieces but not with thin ones (for which the dilatational stresses would be much less). If the cavitation-like process can occur in a time-dependent manner, this could account for the occurrence of time-dependent failure in a strain-crystallizing elastomer.

Results for different test geometries can be interrelated by means of the strain energy release rate when near-bond failure occurs. Thus, time-dependent failure results from peel tests at 90° or 180° or pure shear tests (with 6 mm thick test pieces) are very similar when plotted in this way. An approximate result from a simple shear test is also not far removed. Thus, when the failure locus does not change, it appears that the relationship between the rate of failure and the strain energy release rate may be a characteristic, geometry-independent property for a particular rubber-metal joint system; the present results thus parallel, with strongly-bonded systems, earlier observations with weaker systems for which failure was interfacial.[5, 14] The dependence of failure rate on energy release rate is very strong for the systems studied.

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