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On the lubrication and wear of metal by rubber

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A puncture test has been used to investigate the lubrication and wear of metal by rubber. Cylindrical metal indentors are used to puncture a rubber surface under controlled conditions. Several mechanisms are investigated which affect both the lubrication and wear of the metal. A self-lubrication effect is reported whereby there is a drop in puncture load over successive punctures, which correlates with a strong increase in the contact angle of water against the metal surface and is associated with a reduction in metal wear rate. It is suggested that this is due to the formation of a rubbery layer on the metal surface. The wear rates of several different metals are studied and at least one metal showed an enhanced wear rate for its hardness, suggesting some mechanism of polymer radical attack. In most cases, however, metal and rubber hardnesses were the dominant factors in determining wear rates.

1. Introduction

Wear of one material by another is usually considered in terms of the wear of a softer material by a harder one. However, hard materials can be worn by softer materials, as illustrated by the wear of a diamond stylus by repeated use on a gramophone record or the wear of stone steps by pedestrian traffic.

Some recent work has suggested that polymer radicals formed by the rupture or degradation of the polymer surface can react chemically with metal oxide layers and act either to decrease or increase rates of wear in different circumstances. Thus Vinogradov *et al.* [1] found in the course of investigating the sliding of polymer discs against metal surfaces that modification of the contacting surfaces could occur both due to polymer degradation (associated with local frictional heating) and to metallization of the polymer surface. They concluded that in at least one case increasing readiness of polymer oxidation reduced the wear rate against steel. Gorokhovskii *et al.* [2–4] also reported modifications to the structure of steel chips ground together with polymethyl methacrylate (PMMA) associated with progressive mechanical degradation of the polymer. Gorokhovskii *et al.* also found that the addition of a small amount of polymer (~5%) to abrasive particles could increase the rate of wear of metals by a factor of two to three. The phenomenon was however reversed when larger amounts (> 10%) of polymer were present, when some reductions in wear rate were observed. Gent and Pulford [5, 6] have also reported chemical effects. The wear rate of steel razor blades held against rotating solid rubber wheels was, for example, reported to be almost 70 times greater for polyisobutylene-co-isoprene than for ethylene propylene rubber of similar hardness. The authors attributed this effect to the direct attack upon metals of free radical species generated by the mechanical rupture of elastomer molecules.

In the present study surface effects associated with the wear and lubrication of cylindrical metal indentors were studied by using the indentors to puncture the surfaces of solid rubber blocks. In such a case, since wear of the metal cylinder can significantly change the radius at the indentor edge, metal wear can cause a change in the load required to puncture. Wear can thus have important consequences for the interpretation of indentation/puncture tests on rubber. The condition of the metal indentor after rupture has been investigated both by electron microscopy and by use of contact angle measurements which can provide a particularly sensitive indication of changes in the metal surface [7]. Some changes are reported and their correlation with both changes in "puncture

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loads" and with metal wear rates are explored. In the light of this evidence there is some discussion of the likely role of polymer radical interactions.

2. The puncture test

2.1. Experimental method

The basic puncture test consists simply of forcing a metal cylinder (the indentor) into a rubber block until the surface ruptures. The load at rupture is called the "puncture load" and is recorded as the peak on a load/deformation plot. Each puncture test was performed on an Instron testing machine and successive punctures always involved the rupture of new material. The size of the rubber blocks used was 23 cm × 23 cm × 2.5 cm, this providing both sufficient area to allow a large number of successive adjacent punctures without mutual interference and sufficient thickness to eliminate any thickness dependence in the results. The indentors were of 1.5 mm diameter and about 4 cm in length, the minimum required to accommodate the large surface deformation that occurs for soft rubber prior to rupture. Most of the puncture results reported involved the same indentation rate of 0.5 cm min^{-1} although the effect of rate was investigated for the range of 0.5 to 20 cm min^{-1}. The indentor ends were polished to a mirror finish using a Kent lapping machine and various grades of grinding paste. The aim was to produce as sharp 90° corners as possible and subsequent examination of the indentor corners with a scanning electron microscope (SEM) showed that corner radii in the range 1 to 5 μm had been achieved. The harder metals produced the smallest initial corner radii. Fig. 1 shows a typical initial "corner" for tungsten carbide.

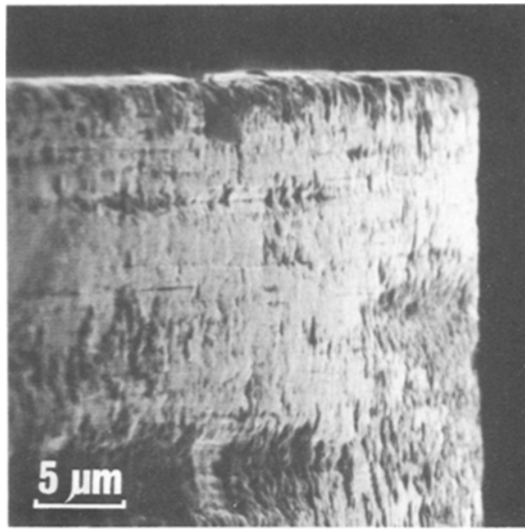

Figure 1 Typical initial indentor corner for tungsten carbide.

Unless otherwise stated, the punctures were carried out in clean dry conditions with no external

TABLE I Vulcanizate formulations

	A	B	C	D	E	F	G	H
Natural rubber, SMR CV	100	100	100	100	100	100	100	100
zinc oxide	3.5	3.5	3.5	3.5	3.5	–	–	3.5
Stearic acid	2	2	2	–	–	–	–	2
Zinc-2-ethylhexanoate	–	–	–	2	2	–	–	–
MOR[1]	–	–	–	1.7	1.7	–	–	–
TBTD[2]	–	–	–	0.7	0.7	–	–	–
Carbon black, SRF (ASTM-N-672)	–	–	20	–	50	–	–	85
Process Oil[3]	–	–	20	–	–	–	–	5
Antioxidant (Nonox ZA)[4]	–	–	–	2	2	–	–	–
Antioxidant/antiozonant, HPPD[5]	3	3	3	–	–	–	–	3
Antiozonant wax[6]	2	2	2	–	–	–	–	2
CBS[7]	0.7	0.7	0.7	–	–	–	–	0.7
Sulphur	2.5	4	2.5	0.7	0.7	–	–	2.5
Dicumyl peroxide	–	–	–	–	–	0.5	5	–
Cure temperature (° C)	141	141	141	140	140	140	140	141
Cure time (h)	1	1	1	1	1	2	2	1

[1] *N*-oxydiethylenebenzothiazole-2-sulphenamide.
[2] Tetrabutylthiuram disulphide.
[3] Fina Process Oil 2059 (Petrofina).
[4] *N*-isopropyl-*N'*-phenyl-*p*-phenylenediamine.
[5] *N*-(1,3-dimethylbutyl)-*N'*-phenyl-*p*-phenylenediamine.
[6] Sunproof Improved (Uniroyal).
[7] *N*-cyclohexylbenzothiazole-2-sulphenamide.

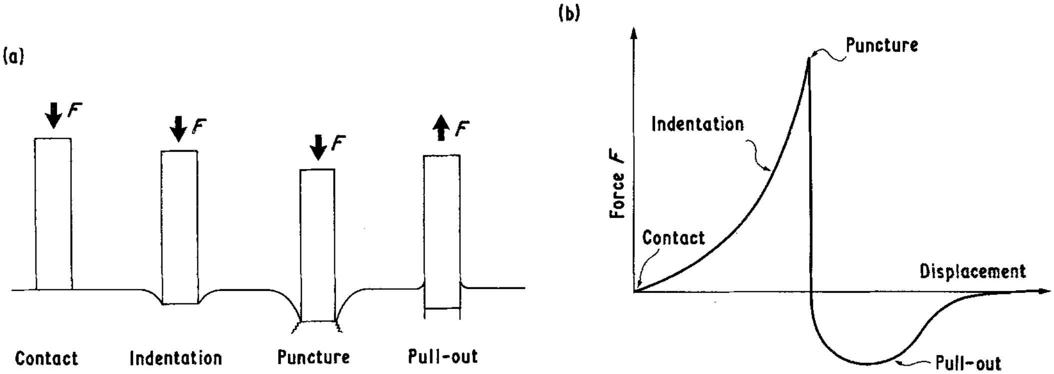

Figure 2 (a) Schematic view of puncture test stages. (b) Typical force/deformation plot for puncture test.

lubrication. The type of rubber used initially (vulcanizate A) was natural rubber cross-linked (or vulcanized) with 2.5 parts of sulphur per hundred of rubber (pphr) and containing the usual ingredients for a "conventional" vulcanizate use in engineering applications, but without carbon black. This produces a vulcanized network with polysulphidic cross-links. Other cure systems with less sulphur (monosulphidic cross-links) and with dicumyl peroxide (carbon–carbon cross-links) were also investigated. Full details of the vulcanizate formulations are given in Table I.

2.2. Results

A typical force/deformation plot, obtained as the cylindrical indentor is forced into the rubber surface, is shown in Fig. 2. At the point of puncture (or rupture) there is a sudden drop in load, and as the indentor is withdrawn some force in the opposite direction occurs due to friction between rubber and metal. The recorded value of puncture load could be highly reproducible for given conditions of indentor and rubber vulcanizate type. Vulcanizate A, for example, when punctured with a sharp-cornered 1.5 mm titanium indentor showed a coefficient of variation of only 2% after the initial ten events.

Over the first few punctures however a surprising drop in puncture load was observed of about 15% (see Fig. 3). This is qualitatively similar to the reduction in puncture load observed when the indentor is lubricated by, for example, the application of silicone oil.

The indentor used in the test was fabricated from commercially pure titanium (99% Ti). Repeating the experiment with indentors made from several different metals, namely a titanium alloy (Ti–89%, Al–6%, V–4%), tool steel (Fe–70%, C–0.75%, W–18%, Cr–4.7%, Co–5.5%, V–1.5%, Mo–0.4%, Si–0.2%, Mn–0.2%) silver steel (Fe–99.25%, C–0.75%) and tungsten carbide produced the same phenomenon. All compositions are by mass. It was found however that if the indentor was recleaned with acetone between successive indentations, the phenomenon did not occur and the puncture load remained at the initial higher level.

The experiment was also repeated with a series of different vulcanizates (A to D) cured with different amounts of sulphur. The results (shown in Fig. 4) demonstrate that increasing the level of sulphur to 4 pphr (vulcanizate B) increased the effect whereas reducing it to 0.7 pphr (vulcanizate D) eliminated it altogether. Incorporating 40 pphr

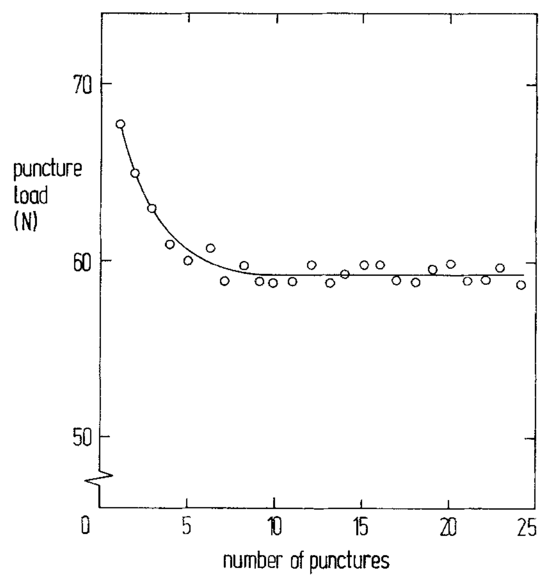

Figure 3 Reduction in puncture load over the first few events. ○ vulcanizate A, 1.5 mm titanium indentor at 0.5 cm min^{-1}.

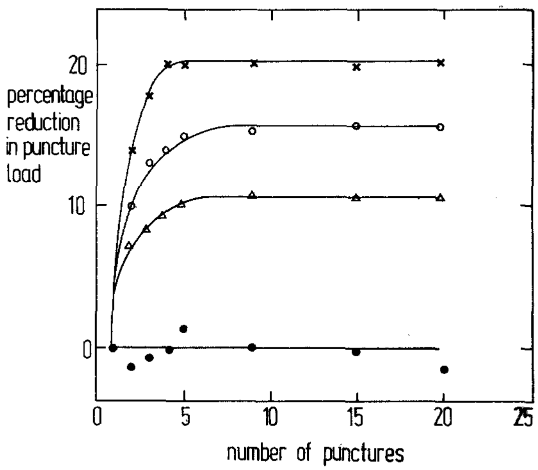

Figure 4 Percentage initial puncture load reduction for several natural rubber vulcanizates. ○ vulcanizate A; × vulcanizate B; △ vulcanizate C; ● vulcanizate D. Titanium indentor at 0.5 cm min^{-1}.

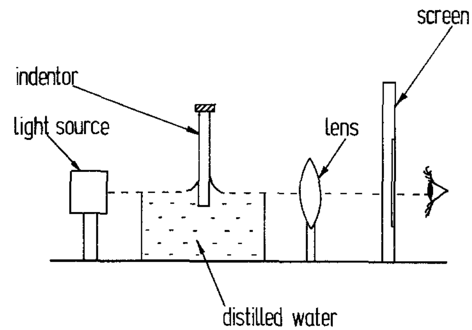

Figure 5 Experimental arrangement for contact angle measurements.

of carbon black into a vulcanizate (C) otherwise identical to vulcanizate A had the effect of substantially reducing the extent of the puncture load decrease. It was however still present.

These observations suggested that in some cases a form of "self-lubrication" occurs whereby a layer of material from the rubber forms on, or reacts with, the metal surface immediately after rubber rupture, and this then reduces the puncture load. The metal surface of an indentor which had shown the effect was examined by scanning electron microscopy with some care immediately after indentation but this failed to reveal any difference in appearance to that of a new, clean indentor. Chemical analysis of the constituents of the solvent wash used to clean the indentors similarly failed to detect any material clearly attributable to the vulcanized rubber. This result suggests that if there is a surface layer it must be extremely thin, i.e. less than 10 nm. At the next stage in investigating this effect, it was decided to make use of contact angle measurements as a sensitive method for detecting small changes in surface layers.

3. Contact angle measurements

3.1. Experimental details

An indentor was immersed in distilled water and then carefully raised through the liquid surface. The region of contact between the water and the metal indentor was then enlarged by projecting its silhouette on to a translucent screen, as shown in Fig. 5. The screen was marked with angular gradations, which enabled the angle of contact to be measured with reasonable accuracy. The reliability of this technique was demonstrated by using it to measure the contact angles of nylon and polytetrafluoroethylene (PTFE) rods. These were found to be 10° and 104°, respectively, in reasonably good agreement with literature values of 0° and 108° [8] obtained by conventional contact angle tests on flat plates. The technique adopted here effectively measure retarding contact angles, which were found to be more reproducible than advancing angles. Since each of the metals tested had shown the same puncture load reduction, most of these experiments were with titanium indentors. This avoided the complication (with the steels) of forming corrosion products in water and dramatically changing the contact angle. Confirmatory experiments showed that provided the latter was avoided, the choice of metal was irrelevant.

3.2. Results

Vulcanizate A showed a substantial and progressive increase in contact angle from 10° up to an "equilibrium" value of about 75°. This increase was quite distinct and, in spite of the inherent experimental difficulties with contact angle measurements, it was well outside experimental error. Experiments with the other vulcanizates showed the phenomenon to varying degrees as shown in Fig. 6. It was found that those vulcanizates which showed the largest increase in contact angle were also those which showed the largest drop in puncture load. This correlation of contact angle increase with puncture load decrease, illustrated more clearly in Fig. 7, provides further evidence for the formation of a lubricating layer of rubbery material on the indentor surfaces. Separate experiments with metal rods coated with a thin layer of rubber latex also yielded contact angle of about 80°. Drops of the same distilled

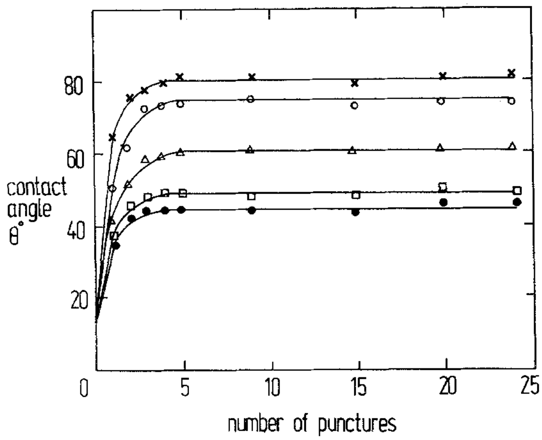

Figure 6 Contact angle increases for initial puncture events. ○ vulcanizate A; × vulcanizate B; △ vulcanizate C; ● vulcanizate D; □ vulcanizate E. Indentation rate 0.5 cm min^{-1}, titanium indentor.

water on flat moulded surfaces of vulcanizates A to D formed contact angles consistent with those shown in Figs. 6 and 7 as the equilibrium values for indentors used to puncture the same vulcanizates.

In the experiments described so far the duration of contact between the indentor and ruptured rubber surfaces was approximately constant, and

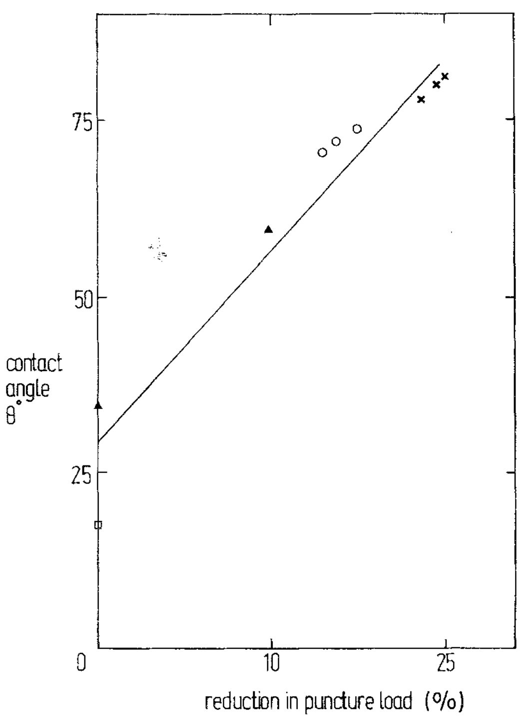

Figure 7 Reduction in puncture load plotted against contact angle. ○ vulcanizate A; × vulcanizate B; □ vulcanizate E; ▲ vulcanizate H.

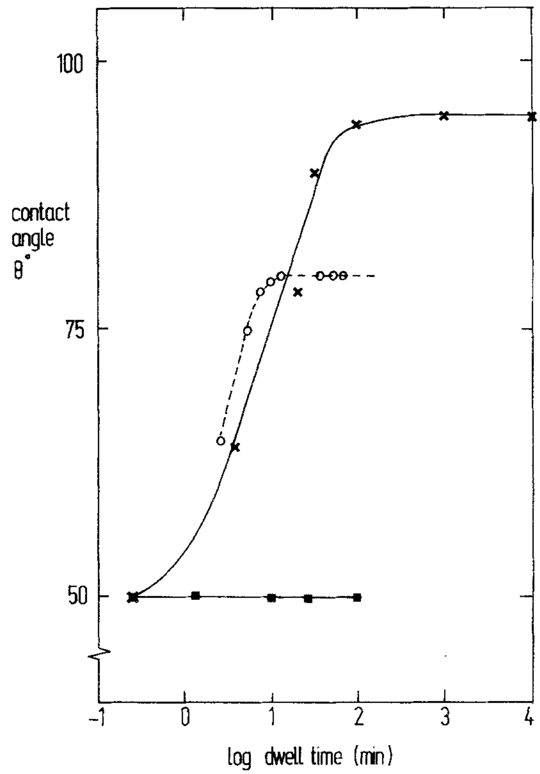

Figure 8 Effect of rubber/metal contact time on contact angle using vulcanizate B and a titanium indentor. ■ repeated puncture at 5 cm min^{-1}, ○ repeated puncture at 0.5 cm min^{-1}; × single puncture indentor remains in rubber for dwell time indicated.

was 3 min. A series of experiments with vulcanizate B showed that reducing the dwell time by increasing the rate of indentation from 0.5 to 5.0 cm min^{-1} significantly reduced the final contact angle from 80° to 50°, as Fig. 8 shows. This suggested that time of contact between rubber and metal may be an important factor. It was found subsequently that if the indentor was not withdrawn after the first indentation but was left in contact with the freshly ruptured rubber for different periods of time up to 7 days, even higher contact angles, approaching 95°, were now recorded (see Fig. 8). In a control experiment the same total rubber/metal contact times were accumulated by means of repeated indentations at 5 cm min^{-1}. This however produced no further increase in the effect beyond that recorded for the first five indentations. Repeated exposure to the air therefore appears to halt further contact angle increases. This suggests that some oxidation of the rubbery layer may occur during or immediately after its formation, producing a product with a lower contact angle in water.

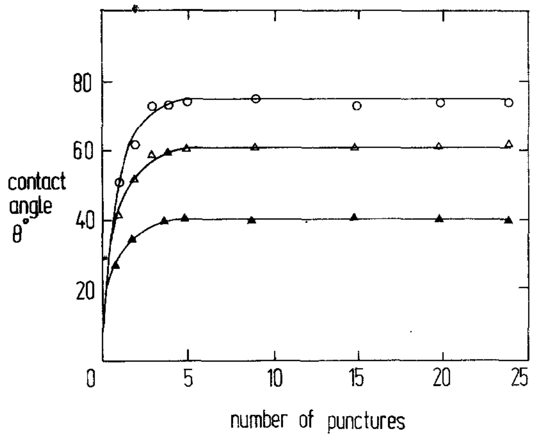

Figure 9 Effect of increasing carbon black content on contact angle effect. ○ vulcanizate A; △ vulcanizate C; ▲ vulcanizate H.

Some further experiments were undertaken to attempt to identify the nature of the rubber ingredients responsible for the self-lubricating layer. With sulphur-cured vulcanizates it has already been observed that the effect increases with the amount of sulphur used in vulcanizing the rubber. A rubber cured with low level of added sulphur (vulcanizate D) did not show the same effect. Omitting the antioxidants and antiozonants from vulcanizate type A did not alter the phenomenon, nor did substituting the stearic acid for zinc-2-ethylhexanoate. This traces the origin of the rubbery layer to the nature of the cross-linked rubber network. Experiments with a lightly cross-linked peroxide-cured natural rubber (vulcanizate F) did show a small effect, especially in contact angle changes, but this was not increased by increasing the cross-link density tenfold (vulcanizate G).

Contact angle measurements appear much more sensitive to the type of surface changes under discussion than puncture load reductions – when in some cases the expected reduction would be within experimental error. An example of this can be seen in the effect of incorporating carbon black. A clear progressive reduction in the contact angle effect was observed for a series of vulcanizates (A, C and H) with the same sulphur cure system but with increasing amounts (O, 40 pphr and 85 pphr) of carbon black (SRF type) respectively. The results are shown in Fig. 9. At the same time, increasing carbon black content also increases the scatter in puncture load measurements so that it is very difficult to determine the corresponding puncture load reductions unequivocally. This result suggests that the presence of carbon black may act to inhibit the formation of the rubber layer or to remove it once formed.

4. Metal wear rates

The final stage in the investigation was to study how the previously discussed surface conditions affect metal wear rates. For this purpose, metal indentors were subjected to several thousand successive indentations in rubber blocks of the same vulcanizate. An automated "puncture table" controlled by a microprocessor was designed and built to facilitate the accumulation of such large numbers of punctures. This automatically moved the rubber block to a fresh position while the test machine cross-head was raised ensuring that puncture always occurred on fresh rubber at a prescribed minimum distance from any previous puncture. The puncture rate was increased to 20 cm min^{-1} for these experiments.

Examination of the indentor tip on an electron microscope enabled the change in corner radius to be measured and thus the volume of metal removed by puncturing to be deduced. Fig. 10 shows a typical series of SEM photographs for tool steel indentors. Photographs were taken at several points around the indentor circumference and average values taken for the change in corner radius. This technique proved to be reasonably accurate in detecting changes in corner radius of about 5 μm and above.

All indentors used in these experiments had initially corner radii of between 1 and 5 μm. Some metals (such as tungsten carbide) proved more amenable to the production by polishing of sharp 90° corners than others. Fig. 11 illustrates the increase in corner radius observed for a titantium indentor over about 10 000 punctures of vulcanizate D. There appears to be an approach to an "equilibrium radius" of about 60 μm after about 10^4 punctures. These radius changes can be expressed in terms of volume of metal removed and hence the gradients of curves such as Fig. 11 can be used to determine wear rates expressed as μm^3 per puncture. Table II summarizes such results for various different metals and different rubber vulcanizates.

The type of metal used for the indentor is clearly the dominant factor in determining wear rates. There is a general trend for the hardest indentor materials to exhibit the lowest wear rates, and, as shown by Fig. 12, in most cases wear rate correlates with metal hardness. There is an interes-

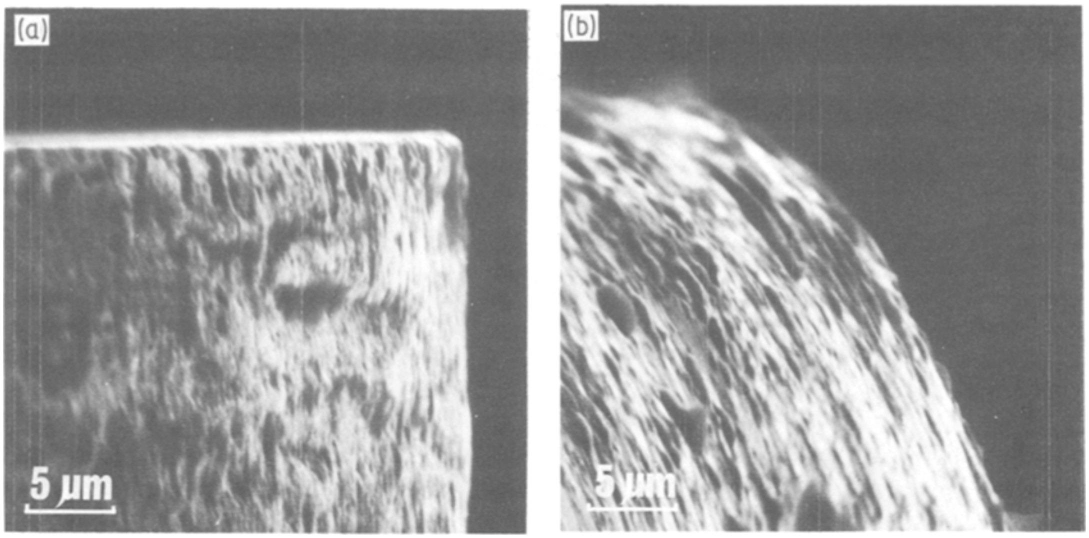

Figure 10 SEM photographs of the corners of tool steel indentors (a) before and (b) after 10^4 puncture events.

ing exception to this in comparing the results for titanium with the results for silver steel. Titanium consistently showed very much lower wear rates than harder silver steel indentors. Titanium showed wear rates below the general trend while silver steel showed rates sharply above it. This suggests that metal hardness is not the only factor.

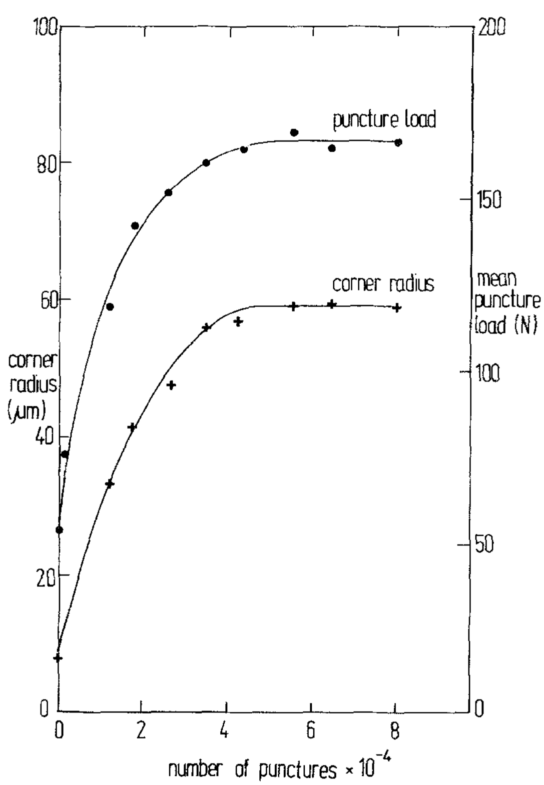

Figure 11 Increase in indentor corner radius with successive rubber punctures for the Ti-64 indentor.

Rubber vulcanizates containing carbon black filler gave significantly higher wear rates than the same rubber formulation unfilled. Indeed the fine "scoring" marks which could be observed on all indentor surfaces were more pronounced for cases where carbon black filler had been present. The size of these score marks was about 500 nm, which is somewhat greater than the known carbon black particle size of 70 nm.

Experiments with the three unfilled sulphur-cured vulcanizates A, B and D showed that the wear rate of the silver steel indentor in D was about three times that in A, which in turn was four times that in B. This is precisely the order of increasing self-lubrication. The result was confirmed for a titanium indentor which showed a 50% greater wear rate in vulcanizate D than in A. This suggests that the rubbery layer detected by the contact angle measurements lubricates the indentor and inhibits wear at its corner.

Some additional wear rate experiments were carried out in the presence of an applied liquid lubricant (TRIFLON – an amyl acetate/alcohol mixture containing 200 nm PTFE particle in suspension) with three different metal indentors (titanium, tool steel and tungsten carbide) in vulcanizate E. The wear rates were indistinguishable for the titanium indentor but reduced by the presence of the lubricant for tool steel and tungsten carbide by about a factor of two. These results are shown in Table III. By way of com-

TABLE II Indentor wear rates

Vulcanizate	Vulcanizate hardness (IRHD)	Indentor	Indentor hardness ($kg\ mm^{-2}$)	Wear rate (μm^3 per puncture)
E	56	SS	300	1000 ± 100
E	56	Ti-2	152	300 ± 20
E	56	Ti-64	350	204 ± 14
E	56	HSTS	1200	104 ± 10
E	56	TC	1300	7 ± 1
A	45	Ti-64	350	96 ± 8
A	45	SS	300	34 ± 4
A	45	HSTS	1200	10 ± 2
D	32	Ti-2	152	80 ± 9
D	32	SS	300	91 ± 10
D	32	HSTS	1200	13 ± 2
D	32	Ti-64	350	120 ± 10
B	47	SS	300	8 ± 2

SS silver steel; Ti-2 commercially pure titanium; Ti-64 titanium alloy; HSTS tool steel; TC tungsten carbide.

parison, the tool steel wear rate was a factor of ten lower in vulcanizate A without addition of a liquid lubricant. The addition of liquid lubricants would therefore appear to have less effect on metal wear rates than the type of rubber vulcanizate used. The factors controlling the metal wear rates thus appear in most cases to be, in order of importance, first the metal hardness, second the amount of carbon black incorporated in the rubber and third lubrication effects at the metal/rubber contact zone – the self lubricating rubbery boundary layer being somewhat more effective than added liquid lubricants.

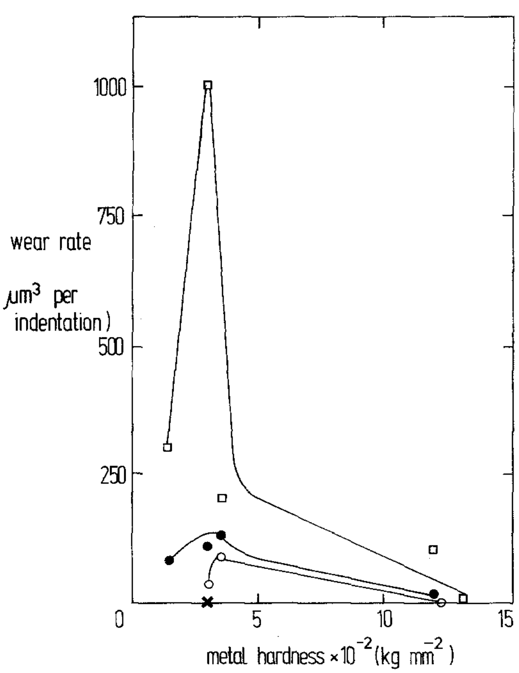

Figure 12 Metal wear rate plotted against metal hardness. ○ vulcanizate A; × vulcanizate B; △ vulcanizate C; ● vulcanizate D.

5. Discussion

The present investigation has explored some of the surface phenomena associated with the lubrication and wear of cylindrical metal indentors used to puncture the surface of solid rubber blocks. The fact that soft unfilled rubber blocks can cause progressive metal wear itself suggests that more than one obvious mechanism may be involved. Previous work has compared wear rates of different polymers and found the results to depend strongly on the type of polymer degradation caused by rupture and on the likely reaction of degraded polymer radicals with the metal oxide layers [6]. In the present case attention was restricted to one polymer type and other aspects of the rubber vulcanizate explored, namely the effect of the cross-linking system and the presence of carbon black filler.

The results suggest the following picture of what happens at the indentor tip.

TABLE III Indentor wear rates on vulcanizate E lubricated and unlubricated

Test condition	Indentor	Hardness ($kg\ mm^{-2}$)	Wear rate (μm^3 per puncture)
Unlubricated	Ti-64	350	196 ± 15
Lubricated	Ti-64	350	208 ± 12
Unlubricated	HSTS	1200	104 ± 8
Lubricated	HSTS	1200	40 ± 5
Unlubricated	TC	1300	7 ± 1
Lubricated	TC	1300	3 ± 1

The rubber surface first becomes elastically deformed as the indentor is forced into it. There may be some local slip at the edge of the indentor surface in contact with the rubber. The extent of such slip will be influenced by any surface lubrication and will have an effect on the force required for the indentor to rupture the rubber surface (the puncture load). The more lubrication there is the more the local slip and the lower the puncture load. When the rubber ruptures, segments of the molecular chain separate and some may be released as free radicals. This occurs while the metal of the indentor is in intimate contact with the rubber. Under these conditions the stability of the free radicals formed (possibly in the near absence of oxygen) may determine subsequent reactions to the metal oxide layer.

It is well known that clear freshly moulded rubber can exhibit substantial adhesion to a clean glass surface (see [9]) and that the energy of adhesion increases with the time of contact. This has been referred to as the dwell time effect. The strength of adhesion of molecular segments of the freshly ruptured rubber may adhere similarly under the action of van der Waals' secondary intermolecular forces. The strength of this adhesion may increase in time due to increased packing of molecular segments on the surface or due to some intrinsic increase in the adhesion of individual segments. When the indentor is withdrawn, some of the molecular segments remain on the metal leaving a thin rubbery covering. The experimental results suggest that there is a progressive increase in the extent of this covering with time according to a dwell time effect. A monomolecular layer is sufficient to alter contact angles radically – it could therefore be that the packing of ruptured rubbery molecular segments increases with time up to a complete covering. The experiments showed a strong increase in contact angle with dwell time.

Different types of rubber network may release types of molecular segment – or even segments of different length. The present results showed a large difference between monosulphidic and polysulphidic networks. The latter showed the larger increase in contact angle and the lower wear rates – indicating the formation of a more highly lubricating rubbery layer. It is possible that the thin rubbery layer experiences some oxidation immediately the indentor is withdrawn – and that the extent of oxidation is reduced for a polysulphidic network. Oxidized rubber is expected to be more polar and would therefore exhibit a lower contact angle to water.

Once the rubbery layer is formed, there may be a second stage of chemical reaction with the metal oxide surface. This may involve primary chemical bonds and the production of a metal oxide–polymer complex which is weaker than the metal oxide itself and detaches more easily from the surface. Subsequent indentations may then partly remove this old layer and reform with new rubbery material repeating the process. This provides a mechanism for the wear of the harder metal by the softer rubber. Wear may also be caused by the fatigue of metal oxide asperities under high local stresses during repeated punctures. If there is a substantial amount of carbon black present the carbon black particles could remove an old degraded layer more effectively than rubber alone. Their presence in the rubber at rupture may also inhibit the formation of a complete layer. This would explain both the lower "equilibrium" contact angle and the higher wear rate with carbon black-filled rubbers.

The results showed a general trend towards increasing wear rate with decreasing metal hardness. A harder metal means in fact a higher local plastic yield stress. This fact dominates the differences in wear rate. There was however one anomaly in that the softest metal tested (commercially pure titanium) showed much lower wear rates than expected for its hardness. This may be due to an exceptionally stable oxide layer which reacts much less readily with the polymer radicals. Titanium is noted for the stability of its oxide layer, the pure metal itself being extremely reactive in oxygen. It may be that the hardest metals tested are also noted for stable oxide layers. The underlying hardness will influence local deformations that could rupture the oxide layer locally and aid wear. The existence of a relatively thick and continuous rubbery layer, although providing ample material to complete the polymer–metal oxide reaction, may act as a lubricant and retard wear – there being less force to remove metal oxide products.

Looked at overall the results show that although the type of vulcanization system (polysulphidic or non-sulphidic) can have some effect on wear rate, this is much less than the effect reported for changes in polymer type. The absence of carbon black reduces the metal wear rate but does not eliminate metal wear. The stability and strength of the metal oxide layers may be the dominant factor in determining metal wear rates – since the harder metals tested were also associated with strong stable oxide layers.

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