

Effects of graphite particle addition upon the abrasive wear of polymer surfaces

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The abrasive wear performance of vinyl ester resins modified with various volume fractions (5, 10, 15, 20 and 30%) of graphite powder has been measured. Using a conveyor belt driven testing machine developed locally, it has been possible to realistically simulate the effect of three-body abrasive wear upon these graphite modified polymer samples. A comparison of the calculated dimensionless wear rates obtained for these surfaces reveals that the effect of the graphite powder depends strongly upon the volume fraction of particles in the resin matrix. It appears that, for intermediate volume fractions, the presence of graphite powder in the resin matrix reduces the abrasive wear of the polymer surface. Scanning electron microscopy has been used to probe the mechanisms of abrasive wear of the pure resin and graphite modified surfaces. It appears that the embedded graphite particles can act as a lubricant during the abrasion process thus reducing the wear rate. The effect of increasing graphite powder volume fraction upon the abrasive wear mechanism is discussed. © 2001 Kluwer Academic Publishers

1. Introduction

Friction and wear can be considered to be the responses of a tribological system. In general, the coefficients of friction and wear are not material constants but rather they are parameters describing the state of contact of the interacting bodies in the tribological system. In certain special states of contact however, and for technical convenience, they may be treated as material properties in an engineering sense [1]. Intuitively, friction and wear are closely related, with an increase in friction leading to a corresponding increase in the observed wear of the material. In reality however, the relationship between friction and wear is not always this straightforward. Indeed, under certain conditions an inverse relationship between friction and wear can be observed [2]. In the case of a composite material the situation is further complicated by the fact that the composite surface often exhibits tribological behaviour widely different from that of its individual constituents [3]. Moreover, it is the exact size, shape and distribution of these constituents that is important in determining the

wear resistance and friction properties of the composite surface [4].

There have been a number of investigations of polymer matrix composites subjected to sliding and abrasive wear. These studies indicate that the wear resistance of the surface depends on the material properties, wear mechanisms and external conditions (such as applied pressure, contact velocity, relative contact motion and the properties of the wear counterparts) [5, 6].

The study of abrasive wear of bulk solids is further complicated by the fact that the wear process can occur as either two-body abrasion, three-body abrasion or a combination of the two [4]. In two-body abrasion, the two surfaces in contact are constrained to move in two dimensions and as such wear occurs through abrasive material on one surface sliding over the other surface. On the other hand in three-body abrasion, the abrasive material is trapped between the two surfaces in contact but is free to roll as well as to slide. Although in practice three-body wear is more common, laboratory scale wear tests (e.g. pin-on-disc tests) are

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typically designed only to simulate two-body abrasion. Typically, wear rates obtained under two-body conditions are much higher than those actually observed in three-body wear, sometimes by as much as an order of magnitude [7]. Furthermore, some of the processes that may occur during three-body abrasion (such as ploughing, wedge formation, cutting, micro-fatigue and micro cracking) may not be accurately modelled by a simple two-body test [8]. In reality, because of the complexity of the abrasion process, no single mechanism can completely account for all of the material loss.

A variety of solid lubricants have been employed in low-friction materials, due to their beneficial effects on the tribological characteristics when pre-dispersed into base polymer before moulding [9]. Graphite is a well-known solid lubricant and the purpose of this study was to establish its effect upon the material removal mechanisms observed during realistic three-body abrasive wear.

In this study, the effect of the addition of graphite particles upon the abrasive wear mechanism exhibited by polymer surfaces has been investigated. In particular, the influence of graphite particle volume fraction upon the observed wear rate has been measured. These measurements, together with scanning electron microscopy, have been used to demonstrate that the graphite particle volume fraction has a significant effect upon the abrasive wear mechanism observed and hence the wear rate. A possible mechanism to explain the observed results is discussed.

2. Experimental

The resin used for manufacturing all the polymer materials was Derakane 441-400 (Epoxy Vinyl Ester, Dow Plastics), which is a vinyl ester based resin containing about 7.5 wt % of a carboxyl-terminated butadiene acrylonitrile (CTBN) liquid polymer reacted into the resin base. The resin was cured with 2 weight % methyl ethyl ketone peroxide (MEKP). The graphite particle modified resin specimens were fabricated by mixing the required amount of resin with calculated amount of graphite powder to obtain the desired graphite particle volume fraction. This mixture was then laid on a single sided mould and cured at room temperature. Five samples were fabricated for the wear tests described here containing 5, 10, 15, 20 and 30 volume % graphite powder.

Ignimbrite particles were chosen as the abrasive wear medium due to their hardness and highly angular particle shape. Ignimbrite consists of vitric (glass) shards, variable proportions of pumice fragments and crystals, and a further variable proportion of lithic (stone) fragments. Primary devitrification results in pumice while secondary devitrification results in shards, which exhibit varying degrees of flattening and welding, and sometimes centrally directed tiny crystals. The typical hardness of the ignimbrite particles was measured to lie in the range, VHN ~255–295, as measured on the Vickers hardness scale. The particle sizes were measured to lie in the range 2.0–8 mm.

All of the abrasive wear testing was conducted using an open three-body linear sliding abrasive wear tester

that has been developed at the University of Newcastle. A bin and hopper arrangement delivers the abrasive particles onto a conveyor belt and under the test specimen and carrier. The carrier is loaded with steel weights allowing the normal load on the specimen to be varied. For the experiments described here the applied pressure was set to 5.4 kPa. Shear load cells attached to the carrier allow continuous monitoring of the friction force. The conveyor belt has a variable speed hydraulic drive to allow the incident flux of abrasive particles to be altered. Approximately 0.5 m³ of wear medium can be stored in bin and hopper and drawn out as the conveyor belt moves beneath the hopper exit creating a flow of wear media with a uniform bed depth of approximately 15 mm. After the media passes beneath the test specimen, it is circulated back to the bin and then hopper via a bucket elevator. For the experiments described here the belt speed was set to be 0.55 m s⁻¹. A detailed description of the testing machine is given elsewhere [10].

Dimensionless wear rates were calculated according to Equation 1 [9]. Cumulative and progressive wear rates have been calculated for a number of specimens and compared.

$$W = \frac{(M_1 - M_2)}{\rho AVt} \quad (1)$$

M_1 and M_2 are the specimen mass before and after test respectively, ρ is the density of composite, A is the apparent wear surface area, V is the belt speed and t is the running time. Measurements were taken every 15 minutes to monitor the kinetics of the wear process and to ensure that the system had reached a steady-state wear condition. Previous wear studies have shown that, although the mean size of the abrasive particles is relatively large (2–8 mm), there are sufficient numbers of smaller abrasive particles within the particle size distribution to ensure that it is possible to obtain a reliable measure of the abraded contact area [11].

3. Results and discussion

The variation of dimensionless wear rate for the pure resin and the five graphite powder modified surfaces is shown in Fig. 1. The results indicate that the wear

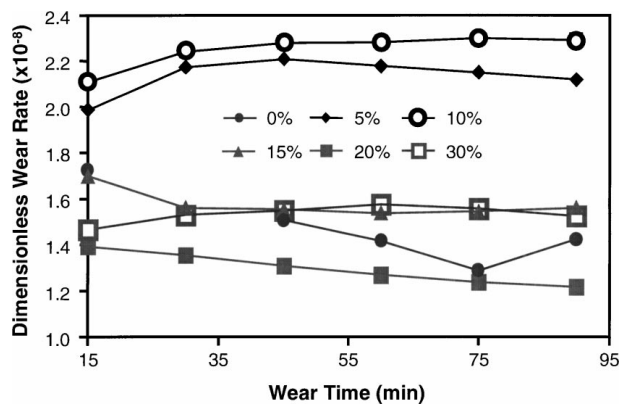


Figure 1 Variation of dimensionless wear rate as a function of wear time for graphite particle modified resin samples with graphite volume concentrations of 0, 5, 10, 15, 20 and 30%. All of the samples demonstrate some degree of time-dependent wear behaviour before the wear rate reaches an asymptotic value and the equilibrium wear is established.

rate for all of the samples is initially time-dependent and that there is a “bedding-in” of the wear specimen during which time the wear rate either increases or decreases gradually until finally equilibrium, or steady state, wear is established. It is commonly understood that this so-called transient wear is due to asperities and inhomogeneities in the wearing surface [12]. The gradual increase in wear rate can be attributed to the removal of a more wear resistant resin skin before the homogenous surface structure is exposed. The gradual decrease in wear rate is most likely due to the presence of prominent surface asperities. The load experienced by these surface features, and hence the wear rate, is initially very high. As the wear progresses, the surfaces are worn more evenly resulting in a wear rate that decreases to an asymptotic level. Therefore, in order to compare the wear rates of the six different surfaces, the samples are worn initially to remove these resin rich surfaces and to establish the steady state wear surface.

Fig. 2 shows the variation of the measured equilibrium wear rate, that is the wear rate at the asymptotic limit of each of the curves in Fig. 1, as a function of graphite powder concentration. The data in Fig. 2 exhibit a systematic variation of the equilibrium wear rate as a function of increasing graphite powder volume fraction. The reproducibility of the data was checked by repeating the entire wear measurement at least twice for each surface studied. It was found that although there were batch-to-batch variations of as much as $\pm 15\%$ in the measured wear rate, the observed systematic variation in the data was reproducible to within $\pm 5\%$.

It seems that the effect of the graphite powder modification can be considered to occur in three distinct concentration regimes. In the first regime, increasing the volume fraction of graphite powder from 0–10% results in an immediate increase in the measured wear rate compared to that of the pure resin. It would seem that the presence of graphite powder at these concentrations has a detrimental effect upon the abrasive wear resistance of the resin matrix. In the second regime, from 10–20 volume % of graphite powder, the wear rate of the graphite modified surfaces systematically decreases, reaching a minimum wear rate (at 20 volume %) that is lower than the wear rate of the

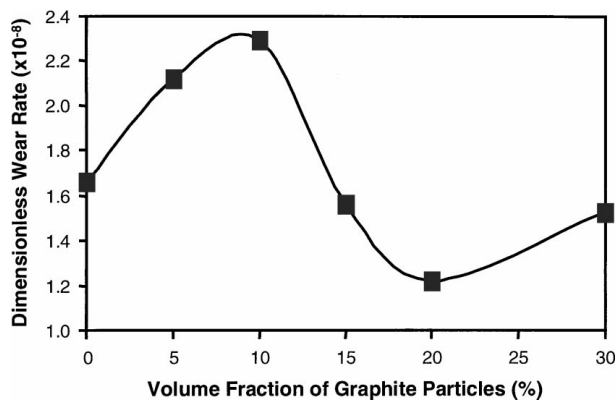


Figure 2 Variation of the dimensionless wear rate at equilibrium as a function of the volume fraction of graphite particle reinforcement for surfaces abraded by ignimbrite particles. The observed systematic variation, with a maximum wear rate at 10 volume % and a minimum wear rate at 20 volume % of graphite particle reinforcement, was reproducible to within $\pm 5\%$.

pure resin. In the third and final regime, increasing the concentration of graphite powder embedded in the resin matrix beyond 20 volume %, leads to a corresponding increase in the measured wear rate. Therefore, it would appear that the presence of graphite can enhance the abrasive wear resistance of a polymer surface, but that the concentration of graphite powder plays a critical role in determining its efficacy.

Graphite is one of a variety of solid lubricants employed in low-friction polymer materials. For example, polytetrafluoroethylene (PTFE), silicones, molybdenum disulphide and graphite are commonly used solid lubricants, due to the beneficial effect they have upon the tribological characteristics of composite materials when pre-dispersed into the base polymer before moulding [9]. These lubricant packages decrease the coefficient of friction and thus the wear rate. The lubricating effect of graphite is a consequence of its molecular structure, which consists of hexagonal carbon sheets (bound by strong sp^2 hybridised carbon – carbon bonds) held together by weaker van der Waals interactions. It is the motion of these carbon sheets, which are relatively free to slip over each other, which provides graphite with its known lubricity. However, the presence of graphite powder in the polymer resin, as with any impurity species, will also disrupt the homogeneity of the polymer matrix. It seems plausible that the presence of structurally weak graphitic regions is likely to lead to a necessarily weaker resin structure, which in turn is likely to be associated with a corresponding decrease in abrasive wear resistance.

Thus, it would seem that there are two competing factors that influence the abrasive wear performance of the graphite modified polymer surface. Firstly, there is the inherent weakening of the polymer matrix due to the disruption of the matrix structure caused by the graphite powder, which would tend to increase the observed abrasive wear rate. Secondly, there is the self-lubricating effect of the graphite particles, which tends to decrease the observed wear rate. The observed changes in wear rate as a function of increasing graphite powder concentration can be explained in terms of these two competing factors. In the first concentration regime, (0–10 volume % graphite powder), the weakening of the polymer matrix by the graphite is the dominant process and the wear increases. In the second regime (10–20 volume % graphite powder) however, the lubricity of the graphite particles plays an increasingly important role in determining the abrasive wear performance of the polymer surface and the wear rate of the surface drops to a minimum. Interestingly, the minimum wear rate, observed at a concentration of 20 volume % of graphite powder, is lower than the wear rate of the pure resin. This observation demonstrates that, in terms of abrasion, the weakening effect of the graphite powder upon the matrix structure can be overcome by the self-lubricating nature of the graphite particles to produce a surface with an enhanced wear resistance.

In order to study the details of the wear mechanisms occurring in the three wear regimes, scanning electron microscopy (SEM) was used to determine the role that graphite plays in determining the behaviour of these modified polymer surfaces during abrasive wear. In

particular, the effect of the graphite particle concentration upon the dominant wear mechanisms that occur during the abrasion process was investigated.

It is recognised that the abrasive wear occurs mainly by three mechanisms known as micro ploughing, micro cutting and micro cracking [12]. Micro ploughing is also referred to as plastic grooving, whereby material is displaced sideways by plastically deforming the abraded material. During this mechanism no material is removed from the surface and it is typically present when hard particles abrade a soft and ductile material. Micro cutting refers to chipping of the abraded surface, whereupon surface material in front of abrading particle is actually removed as small fragments. This mechanism depends mainly on the ability of the particle to penetrate the surface and hence upon the relative hardness of the two materials in contact as well as the particle geometry. The micro cracking mechanism is generally accepted to involve the formation of cracks on the surface due to localised stress without actually removing material from the surface. The repeated application of these localised surface loads results in the propagation of these micro cracks until eventually material is removed from the surface as relatively large particles. Micro cracking is observed when the critical surface pressure is exceeded which is given by

$$P_{\text{crit}} \propto \left(\frac{K_{\text{IC}}^2}{H} \right) \quad (2)$$

where K_{IC} is the mode I toughness and H is the hardness of the material being abraded [12]. Table I summarises the measured hardness data for the experimental samples.

A low magnification scanning electron micrograph of the resin after abrasion by ignimbrite particles is shown in Fig. 3. The SEM pictures of the abraded resin surface clearly show wear marks along the direction of the flow of the wear media, which are consistent with a 2-body wear process. In addition, short and deep scratch-like grooves are also present together with short micro cracks on the surface, which appear to be randomly distributed. At higher magnifications (Fig. 4), a fine wear debris can be observed on the surface. These particles appear to be the product of surface fatigue, caused by sections of the surface cracking away, rather than through micro cutting of the surface. This observation suggests that the fatigue damage is caused by highly angular ignimbrite particles that are free to roll under the specimen, which create repeated indentations in the resin surface. These repeated impacts lead to the formation of micro cracks in the relatively brittle resin surface, which lead to a brittle fracture of small sections of the surface.

Fig. 5 shows an electron micrograph of the resin surface with 5% graphite particle reinforcement after wear by ignimbrite particles. Comparing this image with that of the pure resin surface shown in Fig. 3 it is clear that the 5% graphite-modified surface exhibits an increased density of fatigue failure areas. SEM images obtained at higher magnifications (Fig. 6) also appear to indicate that the fatigue affected areas are larger than for the pure resin surface (Fig. 4). This observation is consistent with the increase in wear rate observed for these samples and can be attributed to a weakening of the resin matrix due to the addition of the graphite particles. Similar micrographs were also observed for wear of

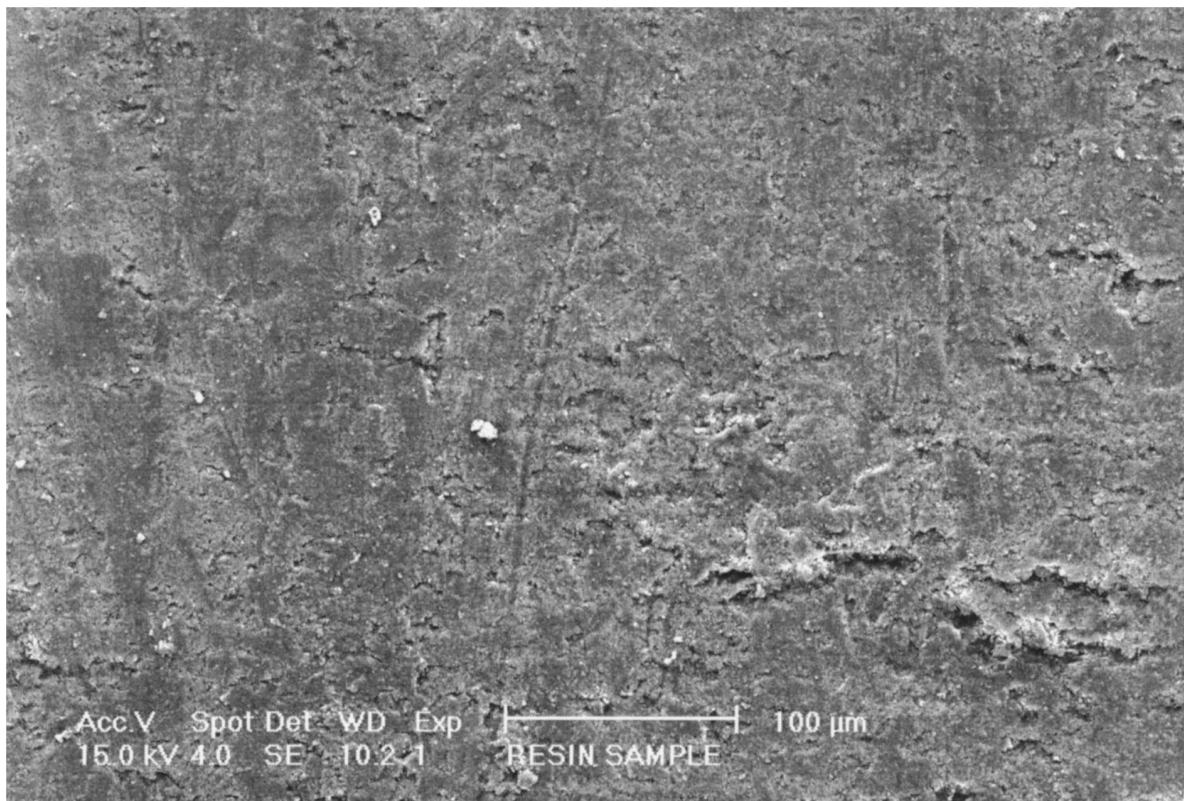


Figure 3 Low magnification SEM image of the resin surface following abrasion by ignimbrite particles. Wear marks are observed on the surface together with short deep scratches. Material loss appears to occur predominantly through fatigue damage of the surface with many pits and craters clearly visible. The location of the majority of the craters does not appear to be correlated with the visible scratches on the surface.

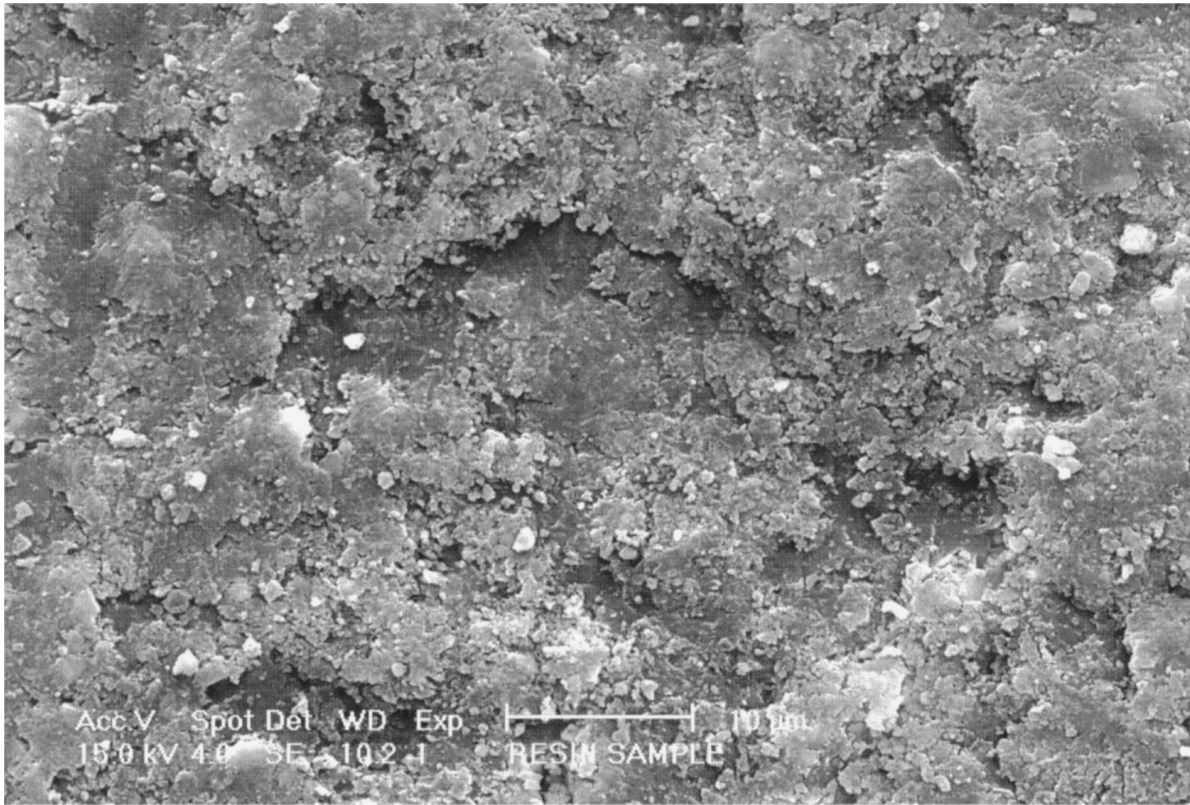


Figure 4 High magnification SEM image of the resin surface following abrasion by ignimbrite particles. Evidence of wear debris is clearly present on the surface and is consistent with impact fatigue of the surface leading to sections of the surface cracking away.

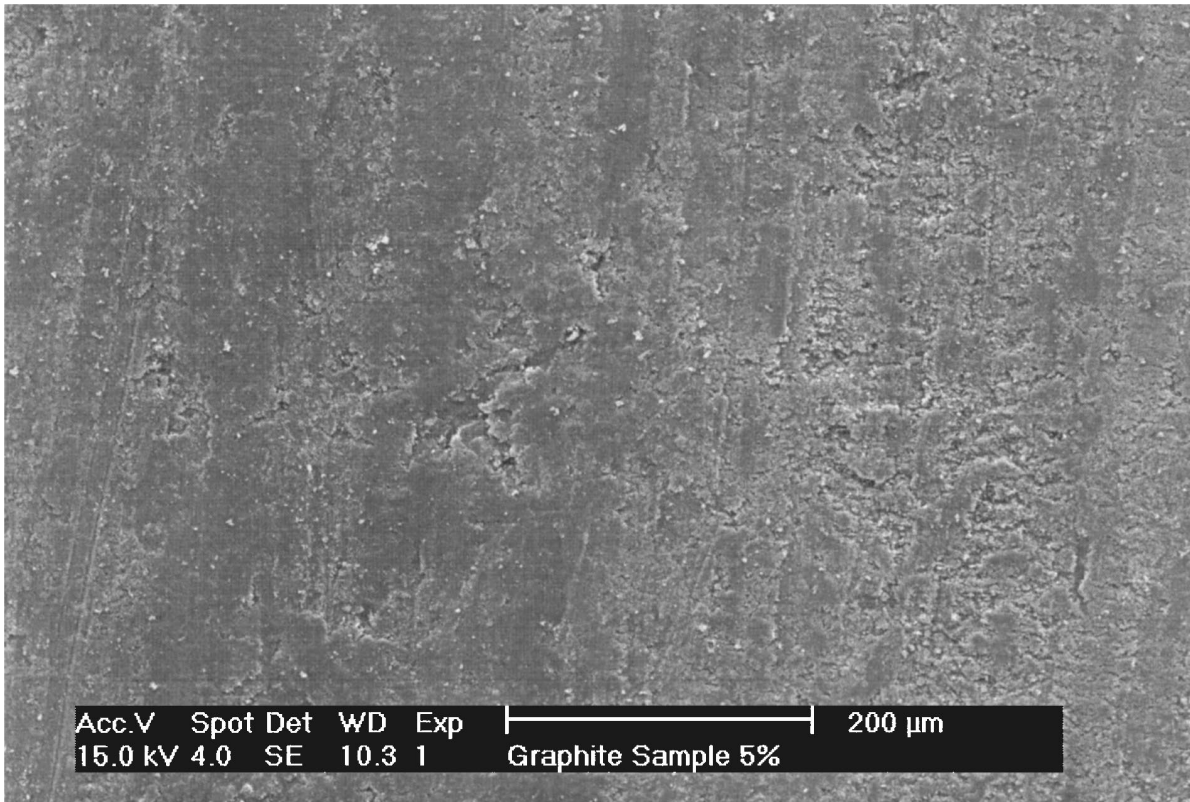


Figure 5 Low magnification SEM image of the resin surface modified with 5 volume % of graphite particles following abrasion by ignimbrite particles. Score marks are visible on the surface together with a high density of fatigue damaged areas.

the 10% graphite modified surfaces. Thus, it appears that the addition of graphite particles at low concentrations (5–10%) actually enhances the effect of the three-body abrasion process and hence increases the wear rate.

However, when the volume fraction of graphite particle reinforcement is increased to 15%, the wear mechanism appears to alter significantly. The surface morphology of a 15% graphite particle modified sample after abrasion by ignimbrite is shown in Fig. 7, and

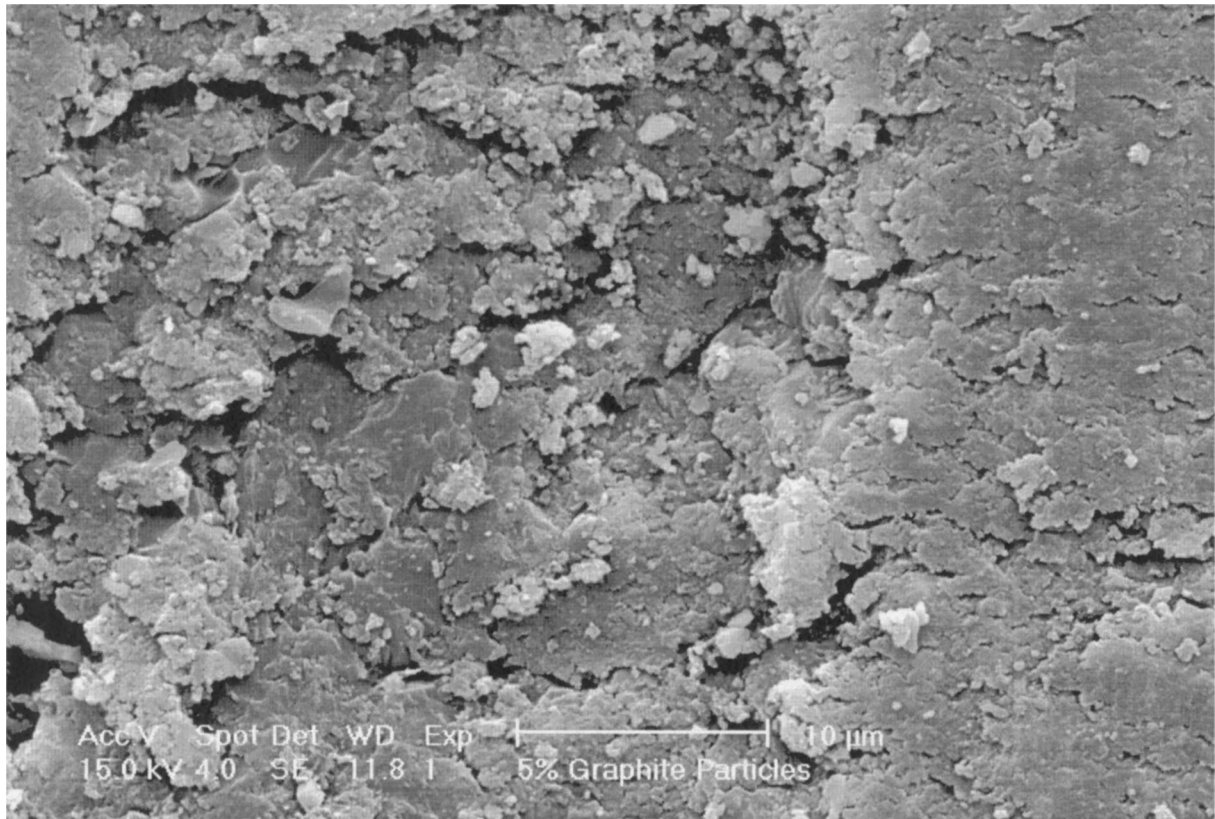


Figure 6 High magnification SEM image of the resin surface modified with 5 volume % of graphite particles following abrasion by ignimbrite particles. As with the pure resin sample, the main material loss appears to occur through fatigue damage of the surface but the 5% graphite modified sample appears to exhibit an even higher density of damaged areas than the pure resin sample.

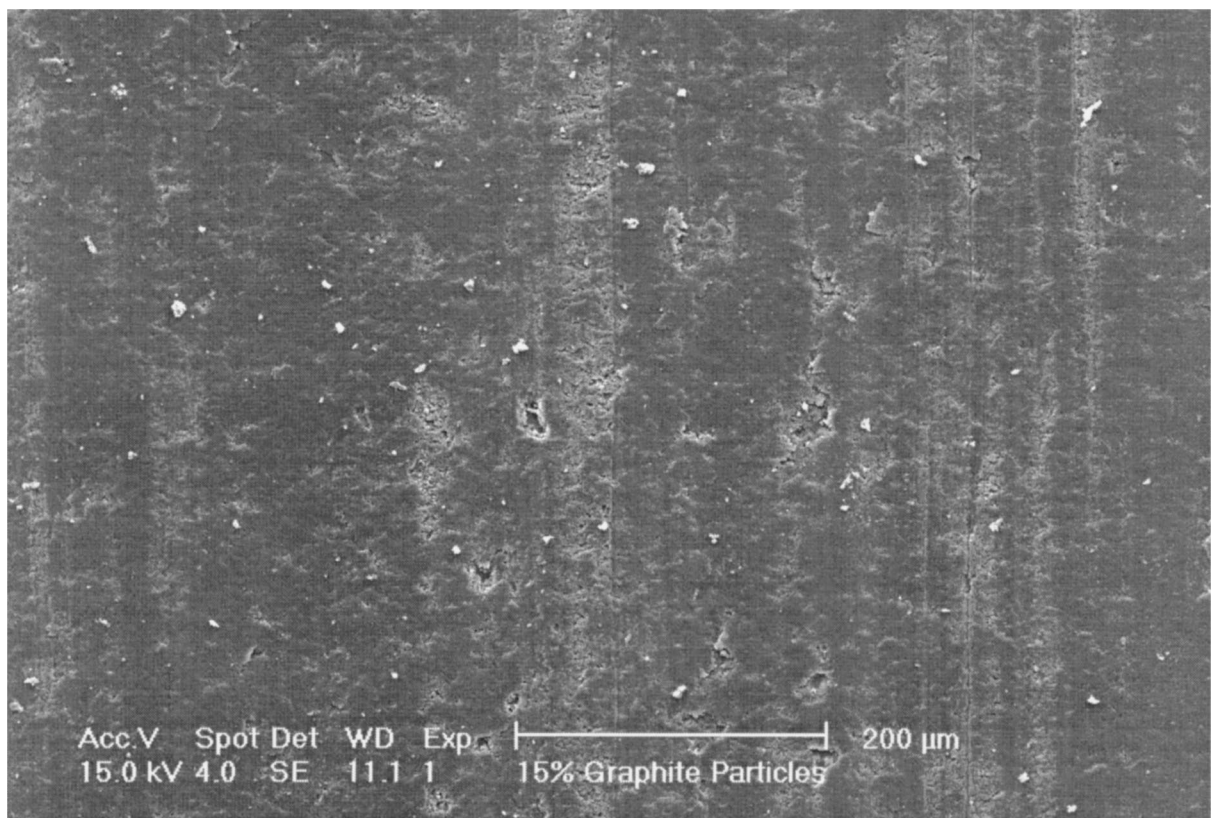


Figure 7 Low magnification SEM image of the resin surface modified with 15 volume % of graphite particles following abrasion by ignimbrite particles. This surface exhibits a relatively lower density of surface pitting and cracking than the pure resin and 5% graphite modified sample following abrasion. Furthermore, there now seems to be a much stronger correlation between the location of the surface craters and the linear wear marks and scratches that are observed to run from the top to the bottom of the electron micrograph.

shows a number of significant differences from the pure resin and 5% graphite modified samples. First, the surface has a much smoother appearance indicating that the 15% graphite surface has undergone much less wear than the other surfaces. Second, the surface density of fatigue affected areas appears to be much lower than for the other two samples. Comparing the high magnification micrograph of the 15% graphite surface shown in Fig. 8 with that of the pure resin surface shown in Fig. 4, there again appears to be a general decrease in the density of fatigue affected areas. Third, and most important, the fatigue affected areas observed in Fig. 7 now appear to be, in general, more closely associated with the wear marks that are observed on the surface. Indeed, there appears to be a network of micro cracks associated with the grooves that are observed to run from the top to the bottom of the micrograph. The presence of lateral micro-cracks in the surface together with the observed surface debris is consistent with previous studies of the wear of brittle materials [7]. Micro cutting by the hard abrasive ignimbrite particles leads to the presence of thin scratch-like grooves on the surface along the wear direction. However, as a sharp particle slides over the surface forming a plastic groove, lateral cracks grow upwards to the free surface from the base of the subsurface deformed region driven by a residual stress associated with the deformed material [13]. As illustrated in Fig. 9, the subsequent repeated application of an applied load causes these micro cracks to coalesce and eventually material is removed by brittle fracture [16].

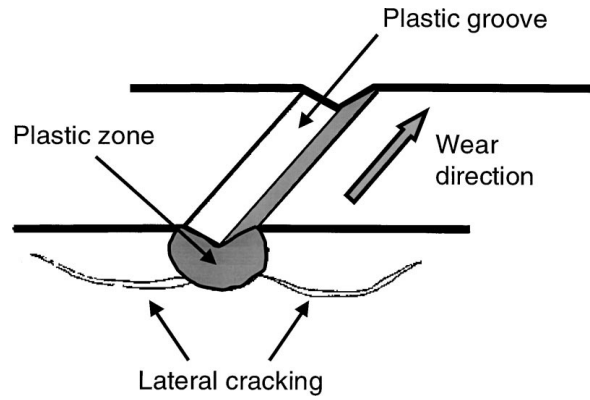


Figure 9 Schematic illustration of the material removal process that occurs for brittle material worn by a hard abrasive particle (after Evans [14]). The abrasion process results in the formation of a plastic groove, which is observed as a micro scratch on the surface, and an associated subsurface region undergoes plastic deformation. In order to relieve the stress generated by this plastic deformation, a network of lateral cracks is created under the surface and when these coalesce and reach the surface then sections of the surface can crack, or spall, away.

Therefore, it would appear that for graphite particle concentrations above about 15% there is a transition in the dominant wear mechanism present on the surface. For graphite particle concentrations below 15%, material removal from the surface occurs predominantly by the impact of ignimbrite particles that are free to roll under the specimen surface (3-body abrasive wear). However, for graphite particle concentrations above 15%, features associated with ignimbrite particles sliding over the surface (2-body abrasive wear) dominate

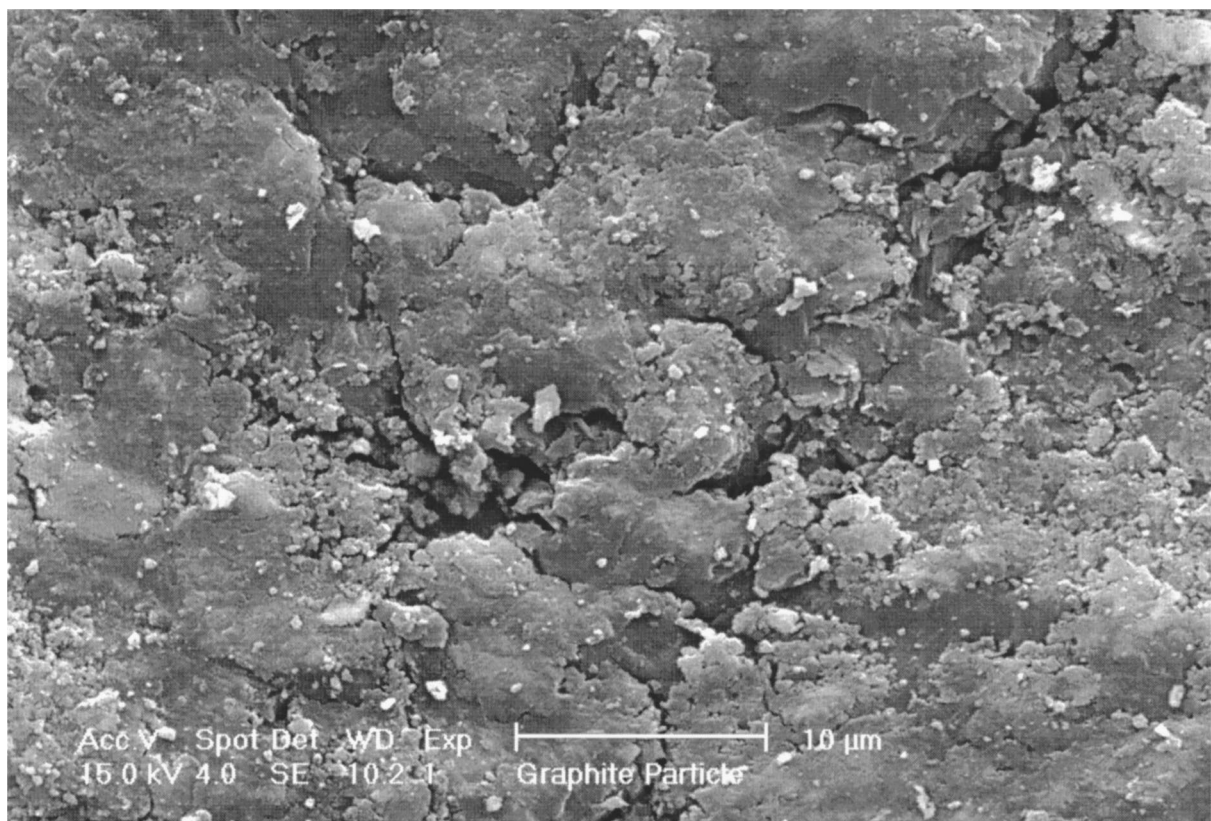


Figure 8 High magnification SEM image of the resin surface modified with 15 volume % of graphite particles following abrasion by ignimbrite particles. Although surface craters and their associated wear debris are observed on the surface, the density of these is lower than that observed following wear of the pure resin surface.

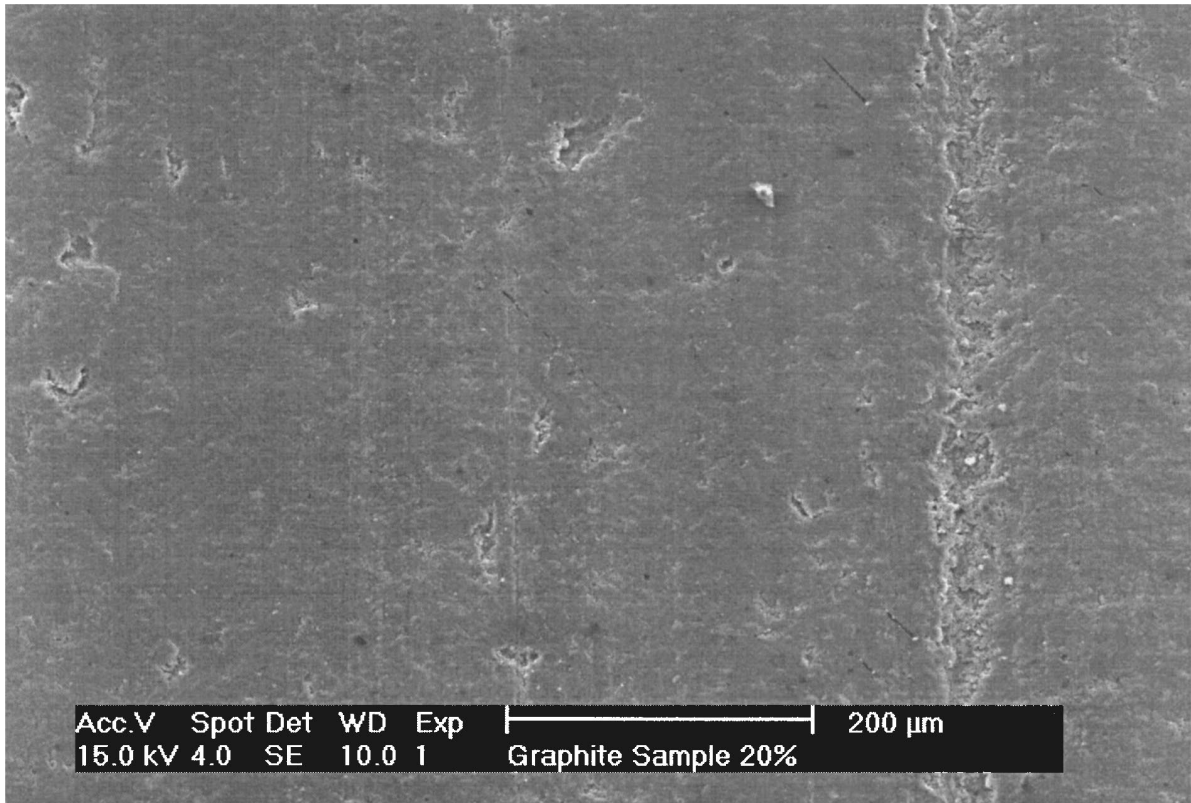


Figure 10 Low magnification SEM image of the resin surface modified with 20 volume % of graphite particles following abrasion by ignimbrite particles. Compared with the 15 volume % graphite modified sample, the surface morphology is again smoother, with fewer pits and craters observed. The morphologies observed on the 20 volume % graphite modified samples exhibited the smoothest morphologies of all of the samples tested, which is entirely consistent with the observation that these materials gave the lowest wear rates at equilibrium. The groove running from top to bottom on the right hand side is a good example of a plastic groove formed by a hard particle sliding across the surface and indicates that 2-body (rather than 3-body) wear dominates the material loss on this surface.

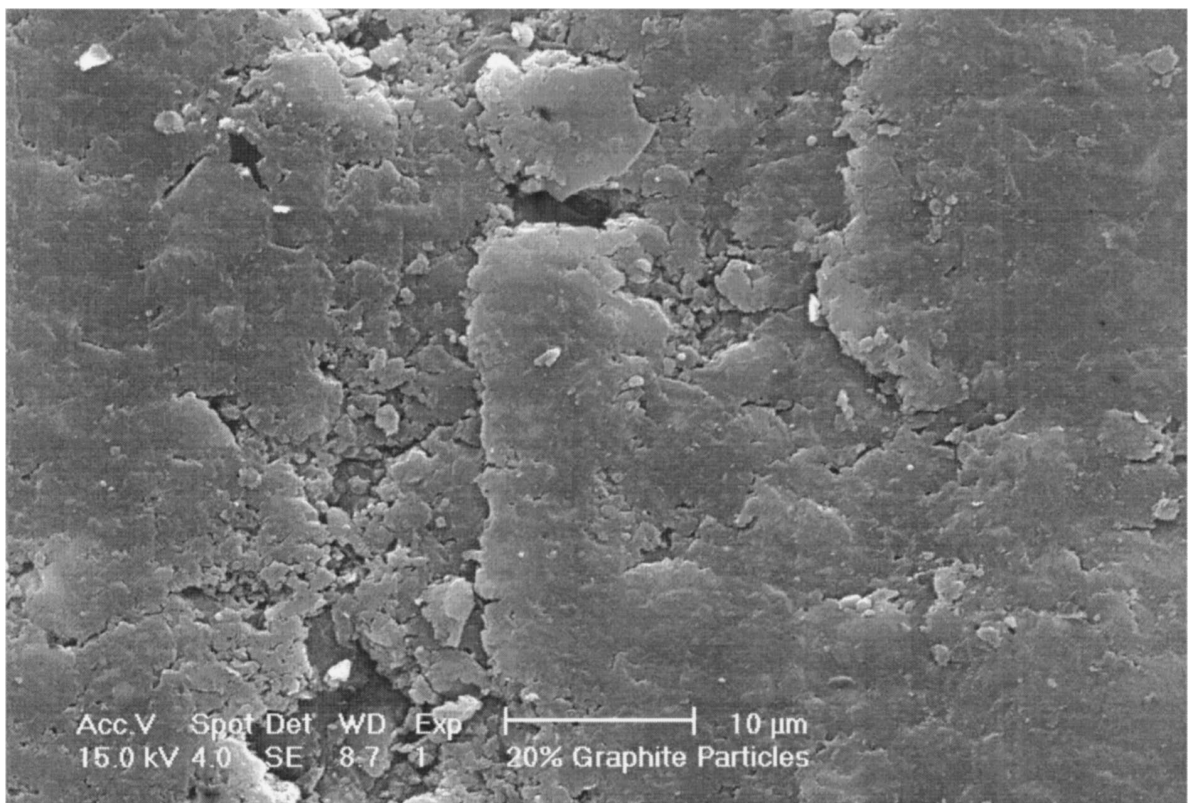


Figure 11 High magnification SEM image of the resin surface modified with 20 volume % of graphite particles following abrasion by ignimbrite particles. The density wear debris on the surface is now quite low and is, in the most part, associated with the surface groove that have been gouged out of the surface by the motion of the hard abrasive ignimbrite particles.

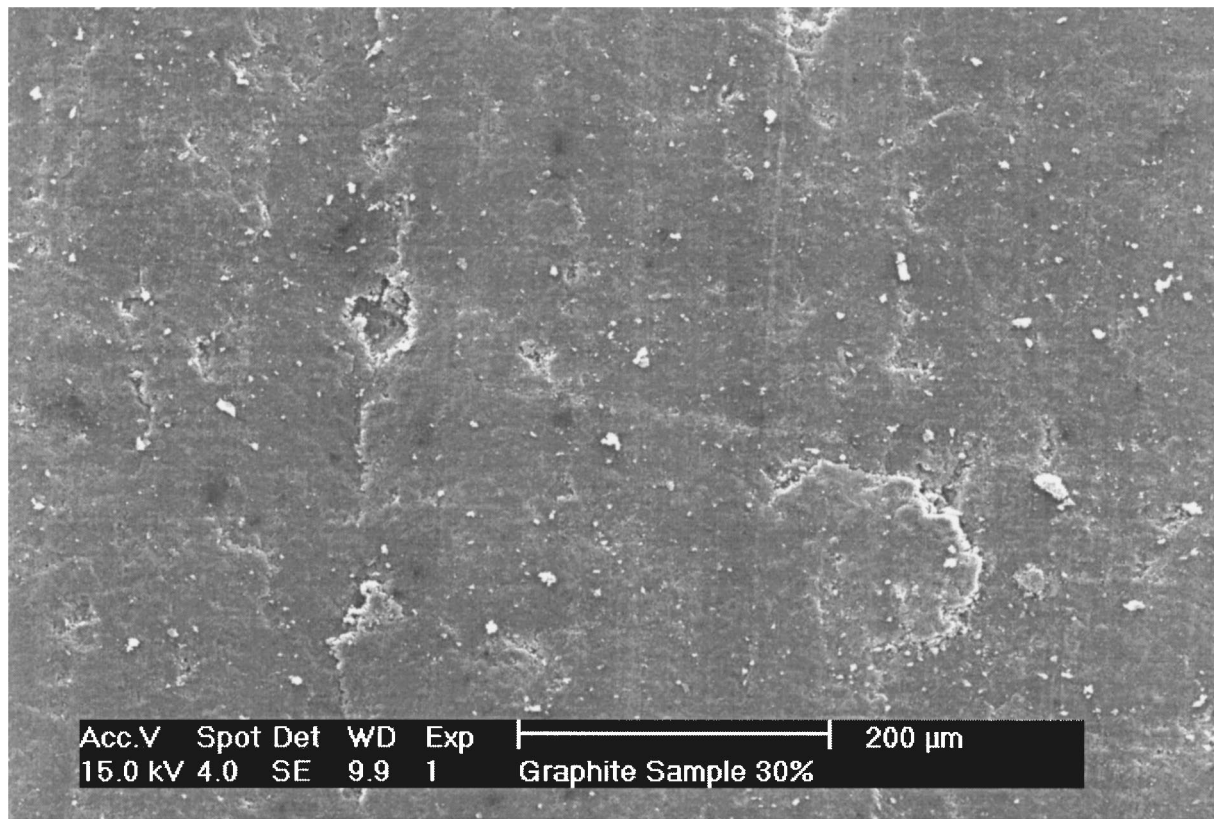


Figure 12 Low magnification SEM image of the resin surface modified with 30 volume % of graphite particles following abrasion by ignimbrite particles. Compared with the surface of the 20 volume % graphite modified material, this surface shows a much higher density of wear debris. Although the wear appears to still be dominated by the formation of craters and pits along the observed wear marks, the extent of these features is much greater than for the 20 volume % graphite surface. This observation appears to be consistent with a surface that is mechanically weakened by the further addition of graphite particles, despite the fact that the wear mechanism is still predominantly 2-body abrasion.

the observed morphology. Intuitively, this transition from 3-body to 2-body abrasive wear arises from a decrease in the surface friction (caused by the increase in graphite concentration) leading to a transition from ignimbrite particles rolling over the surface to sliding across the surface.

It is known that transitions from three body to two body wear can occur in response to changes in experimental conditions. For example, Trezona *et al.* showed that the abrasive wear mechanism can be changed from two body to three body wear due to variation of the test conditions [15]. They investigated the wear mechanisms over a range of loads (0.1 to 5.0 N), slurry concentrations (0.000031 to 0.24 volume fraction abrasive) and abrasive materials (SiC, Al₂O₃ and diamond). Wear mechanism maps, in terms of the applied load and slurry concentration for SiC, Al₂O₃ and diamond show the transition between two-body grooving and three-body rolling to occur at an approximately constant ratio of load to volume fraction concentration.

This trend towards two-body, rather than three-body abrasion continues as the concentration of graphite particles is increased to 20%. The morphology of the 20% graphite particle modified surface (Fig. 10) is smoother than that observed for the 15% graphite sample. There are now only very faint wear marks present on the surface and the density of fatigue affected areas is again reduced. Furthermore, there is a reduction in the degree of grooving and lateral cracking on the surface, which indicates that the increased concentration of graphite

TABLE I Measured Vickers hardness data for the graphite particle modified resin samples with graphite volume concentrations of 0, 5, 10, 15, 20 and 30%

Graphite particle volume concentration	Measured Vickers hardness number	Standard deviation of measurement
0	26.25	0.210
5	24.70	0.45
10	25.85	0.54
15	25.50	1.24
20	24.90	0.37
30	23.58	0.88

in the surface has also reduced the extent of two-body wear occurring at the surface. These observations are confirmed by the high magnification image of the 20% graphite surface shown in Fig. 11, which indicates that these surfaces have a much smoother morphology and less wear debris than those fabricated with lower volume fractions of graphite particles.

Fig. 12 shows an electron micrograph of a 30% graphite particle modified sample following abrasion by ignimbrite. Comparing this micrograph with that of the 20% graphite sample shown in Fig. 10, indicates that the density of wear debris has once again started to increase. Furthermore, the fatigue related areas are now significantly larger than those observed for the 20% graphite samples. Thus, it would appear that the wear mechanism has altered once more, and that the polymer

matrix has been further weakened by the presence of additional graphite in the structure.

4. Conclusions

The addition of graphite particle to resin polymers has a significant influence upon the tribological behaviour of the surfaces of these materials. As the volume fraction of graphite particles is increased from 0 to 30%, the observed wear rate varies systematically and appears to pass through three distinct behaviour regimes.

1. For graphite particle volume fractions less than or equal to 10%, the wear rate increases relative to that of the pure resin. SEM images indicate that the material loss in this regime occurs primarily through 3-body abrasive wear whereby the ignimbrite particles are free to roll across the specimen surface.

2. For graphite particle volume fractions greater than 10% but less than 20%, the wear rate decreases reaching a minimum value at volume fraction of 20%, which is lower than the wear rate of the pure resin. SEM images reveal that the predominant wear process is now 2-body abrasive wear, whereby the ignimbrite particles are primarily sliding over the specimen surface.

3. For graphite particle volume fractions greater than 20%, the wear rate once again increases. SEM images show that although 2-body wear is still the dominant material loss mechanism, the damage effect of the abrasive particles is enhanced in this regime.

This behaviour can be explained in terms of two competing effects that occur upon the addition of graphite particles to a resin matrix. First, the addition of graphite particles weakens the resin structure, probably by interfering with the cross-linking that occurs during curing. Second, at sufficiently high concentrations the graphite particles act as a solid lubricant and reduce the surface friction experienced by the abrading ignimbrite particles. It is the balance between these two competing effects that determines the wear performance in each of the three regimes. In regime 1, the weakening effect of the graphite particles upon the resin matrix dominates and hence the wear rate increases. In regime 2, the reduction in surface friction dominates any further weakening of the resin matrix. As a consequence, the predominant material removal mechanism

changes from 3-body to 2-body abrasive wear and the wear rate decreases. In regime 3, the weakening effect of the further addition of graphite particles again dominates the tribological nature of the surface and although material removal still occurs primarily through 2-body wear more material is removed in each wear event and thus the wear rate increases.

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