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Abrasive wear of dental composite resins

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Abrasive wear is a major problem in the application of dental composite resins. In this study the friction and wear behaviours of two types of dental composites: one containing relatively coarse filler particles and some microfillers (Estilux) and another containing only microfiller particles (Durafill), have been investigated by using a scratch testing machine. Experimental results show that the coefficients of friction in both composites are essentially constant for applied loads up to 20 N. The wear resistance of Durafill is better than Estilux. Under the same testing conditions, the size, shape and distribution of the filler particles are more important variables than applied load and sliding speed in controlling the wear mechanism. It is shown that for Estilux, plastic ploughing by the diamond indenter is the predominant mechanism. For Durafill, however, the formation and propagation of tensile cracks on the worn surface is the main wear mechanism. The effects of two different indenters, diamond and enamel, on the basic wear mechanisms are also discussed.

1. Introduction

The wear behaviour of composite resins is a research area of continuing interest because dental restorative materials are often inferior in wear resistance compared to amalgams [1]. To improve their wear properties, it is necessary to understand the surface failure mechanisms of these materials during wear. Clinical investigations show that "posterior composites" (i.e. composite resins used to restore posterior teeth) are subjected to different wear in the occlusal area (OCA) and the contact free area (CFA) [2, 3]. Abrasive wear is believed to be the main wear mechanism in CFA where the material loss is caused by food friction. In the OCA the mechanism contributing to wear is mainly two-body abrasion, i.e. the sliding action between two surfaces of teeth or filling composite materials [4].

Long-term clinical study should be the most reliable method for abrasion wear studies. The disadvantages are difficult and complex measuring procedures and time consuming. Wear tests in laboratories, are, therefore, desirable for the evaluation of wear behaviour of dental materials under controlled and reproducible testing conditions. Many kinds of *in-vitro* wear experiments have been reported, including two-body and three-body abrasion tests [5–8]. Due to the different experimental designs and measuring systems, the results obtained are not directly comparable [9]. A possible way of comparison is to consider the ranking of the tested materials within each study. However, this ranking may vary from one study to another. Two-body abrasion is a promising method, since it is easier to control the main test parameters which are believed to control the wear process, e.g. applied load and sliding speed. Previous studies were conducted on *in vitro* "two-body" systems where the dental materials were abraded by sliding against the abrasive materials, e.g. silicon carbide [10], steel [5] and enamel [11]. It was claimed that the wear rate results were highly reproducible and could be used to compare and rank these dental materials. It was also claimed that there were good correlations between the experimental wear rates and clinical wear measurements.

The purpose of the present investigation is to characterize the wear resistance and the wear mechanism of two dental composite resins by single-pass scratch testing which simulates the material removal mechanisms that occur during abrasion.

2. Materials and experimental procedure

The dental composite resins used in this study are hybrid and microfilled composites which are specified in Table I [12]. The materials were packed in a steel mould ($20 \times 4 \times 3$ mm). To displace excess material from the mould two glass plates were placed across each end of the mould and compressed. The composite was then light-activated for 30 s from one end. A subsequent polishing process to a final finish with 0.25 μm diamond paste gave identical roughness of sample surfaces. Polished samples were then ultrasonically cleaned and stored in plastic bags before testing.

In-vitro wear tests were performed with a scratch wear testing machine (Fig. 1). During the test the indenter (d) moved horizontally over the test specimen (b) at a speed of about 6 mm/s while the tangential

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TABLE I Dental composite resins evaluated in abrasive wear tests

Material	Type	Manufacturers	Filler	Filler vol.[a] (%)	Mean particle[a] size (μm)	Vickers[b] hardness
Estilux VS	Hybrid	Kulzer Germany	Colloidal silica	68.4	8.8	110.5
Durafill	IMC + SPP[c]	Kulzer Germany	Colloidal silica	37.5	0.04 (17.0)[d]	38.6

[a] Data taken from Williams *et al.* [12]. [b] Tested at 500 g for 15 s, room temperature. [c] Inhomogeneous microfilled composite with splintered prepolymerized particle. [d] Mean particle size of prepolymerized particles.

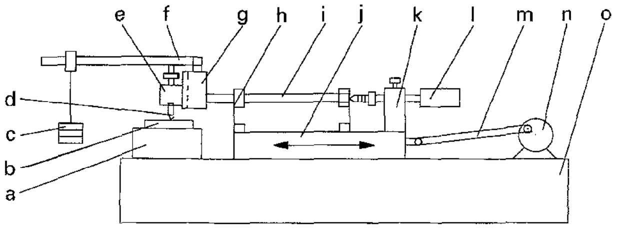

Figure 1 Schematic drawing of the scratching apparatus. a-sample holder; b-sample; c-weights; d-indenter; e-indenter holder; f-lever; g-indenter slideway; h-leaf spring; i-driving bar; j-reciprocating slider; k-transducer holder; l-transducer; m-linkage; n-motor; o-basement.

force was recorded by the strain-gauged transducer (l). Loads applied on the sliding indenter were 2.5, 6.10, 15 and 20 N, respectively. A conical diamond indenter with a tip radius 83 μm and two enamel indenters with tip radii 252 and 293 μm were used to slide across the composite samples. The enamel indenters were made of extracted teeth. The tooth was mounted on a steel bar and turned to a cone with an apex angle 120° (identical to that of the diamond indenter). The tip radius of the enamel indenter was controlled by the polishing process on a turning machine and measured with a computerized image analyser.

The mechanisms of surface failure were studied using scanning electron microscopy (SEM) and the cross-sections of the scratched groove were observed and measured with a laser confocal microscope. Five parallel scratches resulting from different applied loads were made on each sample and three tests were performed for each composite material and testing conditions.

3. Results and discussion

3.1. Coefficient of friction

Fig. 2 gives the results of the frictional force (F) and the applied load (P) during single-pass sliding with both the diamond and enamel indenters. The frictional force increases linearly with applied load. Compared to the enamel indenter, the frictional force caused by the diamond indenter is higher at the same applied load. From the slope of these straight lines the coefficients of friction (μ) for the diamond indenter sliding on both Estilux and Durafill remain approximately equal at 0.48. With the enamel indenter, however, the coefficients of friction are 0.38 and 0.28 for Estilux and Durafill, respectively. The friction coefficient does not appear to depend on the applied load up to 20 N.

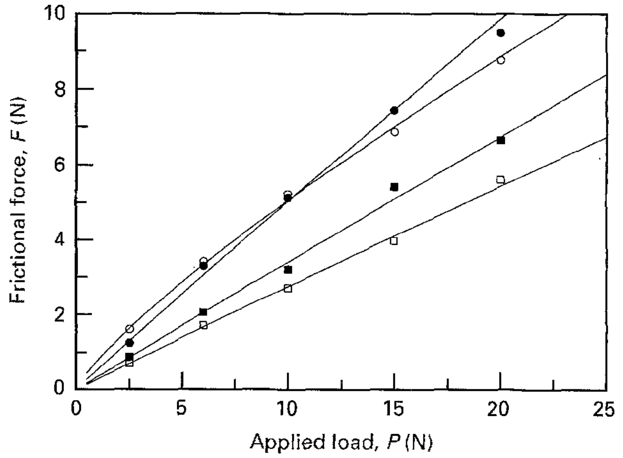

Figure 2 Relationship between the frictional force (F) and the applied load (P): diamond indentor on ○ Durafill, ● Estilux; enamel indenter on □ Durafill, ■ Estilux.

The large difference in μ when using diamond and enamel indenters (Fig. 2) is attributed to their very different hardness. Due to the relatively low hardness of the enamel indenter it also suffered damage due to wear, leading to a reduction in μ.

3.2. Wear rate

Wear is usually expressed as the amount of material removed from a surface such as weight loss or volume loss per unit sliding distance. Thus,

$$\Delta V = L \cdot \Delta S \tag{1}$$

where ΔV is the specific loss of volume during wear testing, calculated from an assumed triangle cross-section with the groove width and depth values as its side length and corresponding height; ΔS is the cross-section area of the groove and L is the sliding distance. The specific wear rate is therefore given by

$$\bar{W} = \frac{\Delta V}{L} \tag{2}$$

Alternatively, the specific wear rate can be evaluated from

$$W_s = \frac{\Delta V}{L \cdot P} \tag{3}$$

dividing $\bar{W}$ by the applied load, P.

Fig. 3 shows the effect of the applied load (P) on the loss of volume (ΔV) during scratch testing. Clearly, ΔV increases with P for both indenters. However,

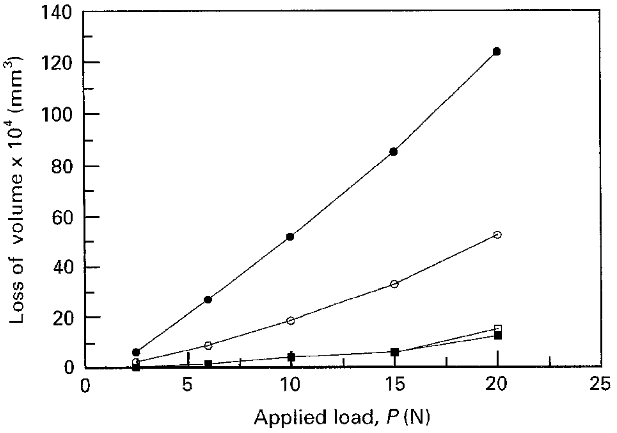

Figure 3 Effect of applied load (P) on the loss of volume (ΔV); diamond indentor on ○ Durafill, ● Estilux; enamel indenter on □ Durafill, ■ Estilux.

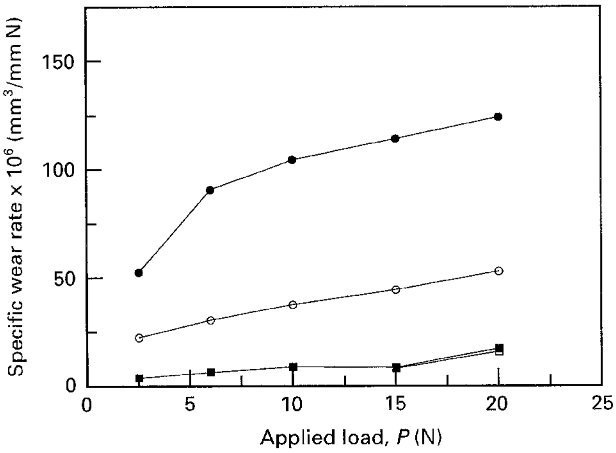

Figure 4 Specific wear rate W_s versus applied load (P) in both dental composite resins: diamond indentor on ○ Durafill, ● Estilux; enamel indenter on □ Durafill, ■ Estilux.

compared to the diamond indenter, ΔV due to the enamel indenter is relatively small and there is almost no difference for both dental composites at higher loads (≥ 15 N). However, volumetric wear of Durafill when scratched by the enamel indenter at loads below 15 N could not be measured (Fig. 3), as scratches on the surface were too shallow (< 0.4 μm) to be detected by the laser confocal microscope. It is also shown in Fig. 3 that the wear resistance of Durafill is better than Estilux when scratched by a diamond indenter. This difference becomes larger for higher applied loads. A similar result was reported in the two-year clinical study by McComb and Brown [13] who showed that the wear rate of Durafill was less than those of Miradapt and Profile (hybrid composite resins not dissimilar to Estilux). Fig. 4 shows the relationships between the specific wear rate and the applied load for both dental materials with diamond and enamel indenters.

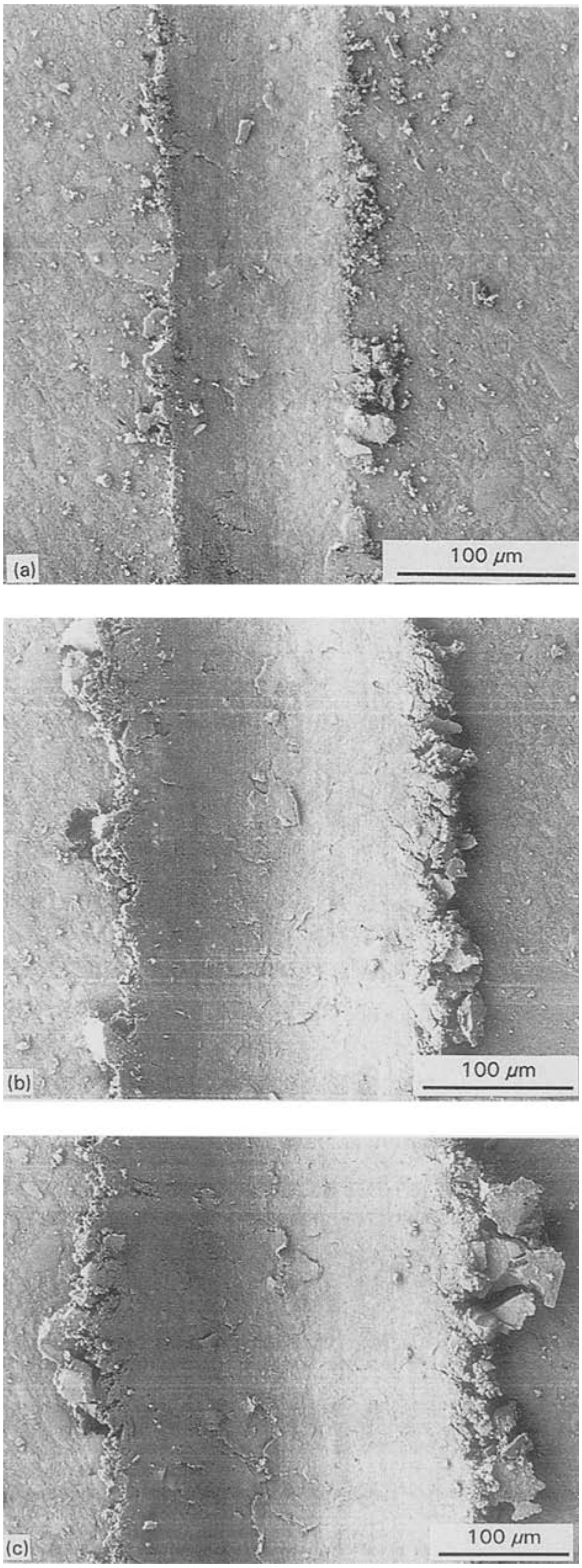

Figure 5 Scratches generated by diamond indenter on Estilux at loads: (a) 2.5 N; (b) 10 N; and (c) 20 N (scratching direction: top to bottom).

3.3. Wear mechanisms

3.3.1. Hybrid type composite (Estilux)

The wear grooves generated by the diamond indenter on Estilux showed a ductile mode as illustrated in Fig. 5. There was not much change in this type of surface failure over the whole load range studied. Plastic deformation was obvious in the wear groove and this meant that plastic ploughing was the predominant wear mechanism. Deformed material could be seen piling up on both sides of the groove (Fig. 5). Also, there were some dislodged wear debris near the

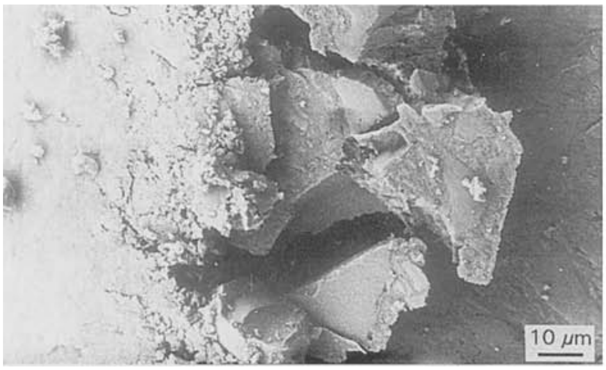

Figure 6 Displaced Estilux wear debris containing both fillers and matrix resin at the edge of wear scar by a diamond indenter under an applied load of 20 N.

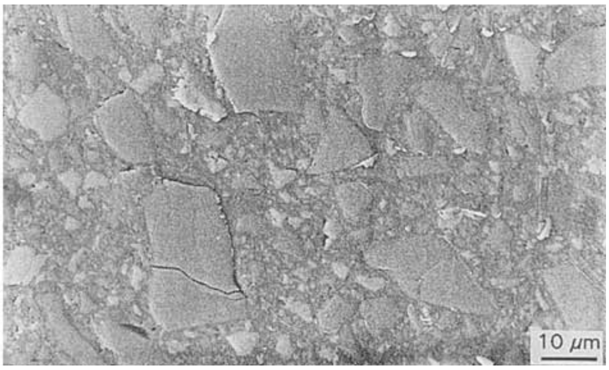

Figure 9 Fractured and debonded filler particles on Estilux surface scratched by an enamel indenter at an applied load of 20 N (scratching direction: top to bottom).

Figure 7 Thin layers of composite resins deposited on the wear track, which were generated by the diamond indenter with a 10 N applied load (scratching direction: top to bottom).

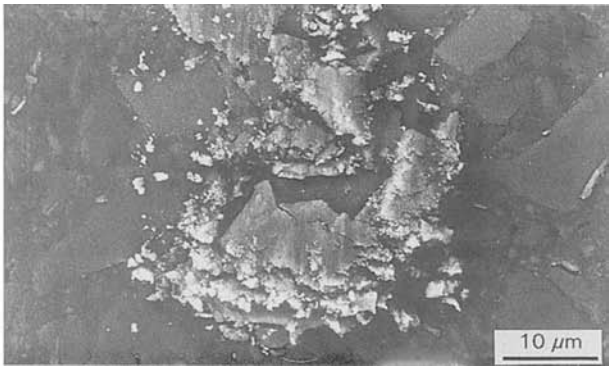

Figure 10 Wear debris of Estilux by an enamel indenter with an applied load 15 N.

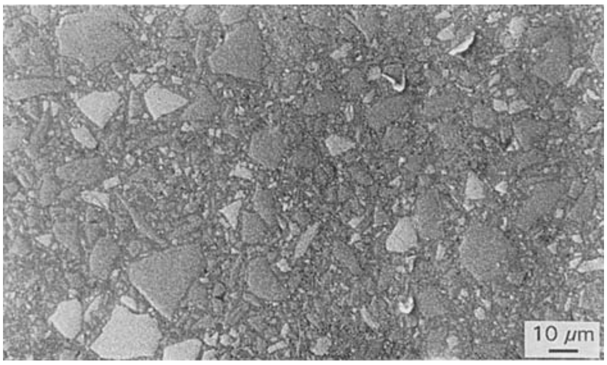

Figure 8 Worn surface of Estilux by an enamel indenter at an applied load of 6 N (scratching direction: top to bottom).

edge of the groove, which contained both polymer matrix and filler particles (Fig. 6). It was noted that thin layers of the composite material scraped off by the indenter were deposited in the wear track as shown in Fig. 7.

Compared to the diamond indenter, the Estilux surfaces were much less damaged by the enamel indenter. Scratches on Estilux at loads below 6 N produced little damage (Fig. 8) with only some polymer flakes visible on the surface. This was probably because filler particles protruding slightly from the polished surface were in complete contact with the enamel indenter. Due to the relatively low hardness of the enamel (408 ± 33 H_V), the indenter was also subjected to a certain degree of wear by the hard filler particles (SiO_2) of the composite resin. Hence, the filler particles could protect the matrix and reduce the wear rate. At higher loads, the contact zones were under higher stresses which might fracture the particles or cause their debonding from the matrix (Fig. 9). These particles were subsequently plucked out from the specimen surface. Fig. 10 shows the wear debris formed, which appears to contain both fractured and small size detached fillers.

3.3.2. Microfilled type composite (Durafill)

In wear testing of Durafill, the results reveal significant brittle fractures. Tensile cracks were found distributed along both sides of the wear track (Fig. 11). In addition, there were some flakes in the wear groove. This phenomenon was also observed in the clinical study by Roulet [14]. The formation and propagation of the tensile cracks were related to the fracture toughness (K_{Ic}) and the ratio of Young's modulus (E) to yield stress (σ_y) at a local zone. Because Durafill, a microfilled composite material, has a much lower Young's modulus than Estilux [12], the E/σ_y value is very low, limiting its capacity to flow plastically. In addition, K_{Ic} of this composite material is only about 0.65–0.71 $\mathrm{MPa\,m^{-3/2}}$ [15, 16]. Therefore, under the action of an applied load, the stress at the local zone can easily reach the critical fracture stress and cracking occurs.

The tensile cracks are closely linked to the microstructure of Durafill. As the filler particles are only

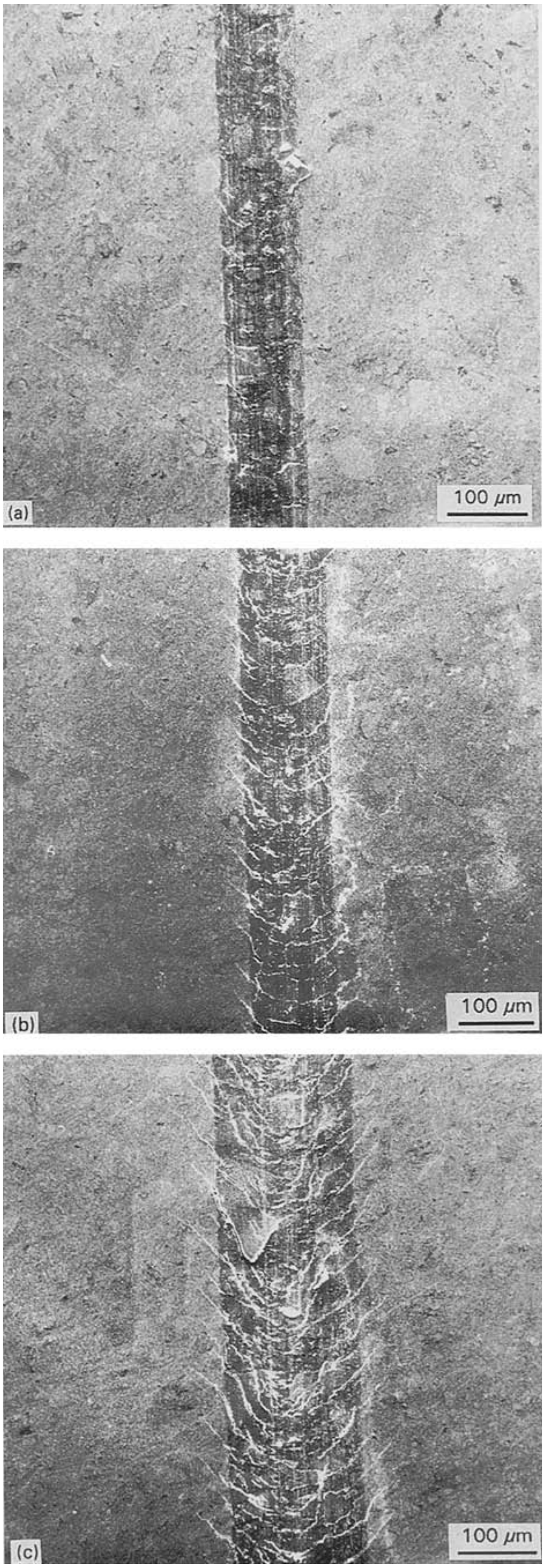

Figure 11 The worn surfaces generated by a diamond indenter on Durafill at loads of (a) 2.5 N, (b) 6 N and (c) 15 N (scratching direction: top to bottom).

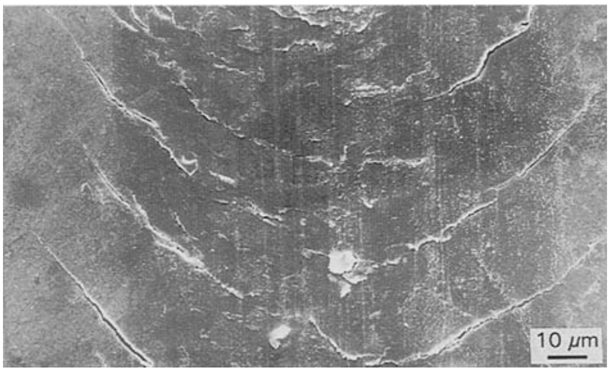

Figure 12 Crack formation at the interface between the prepolymerized particles and the matrix of Durafill at an applied load of 2.5 N by a diamond indenter (scratching direction: top to bottom).

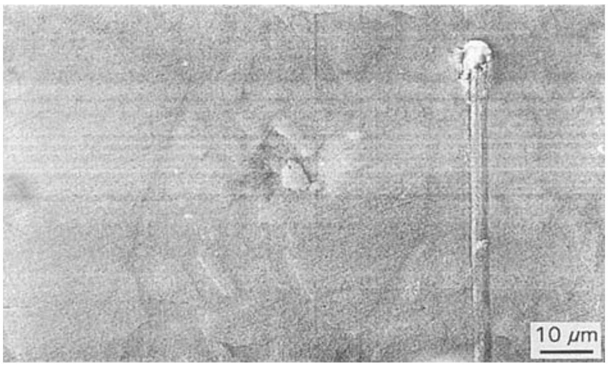

Figure 13 Microgroove formed by a wear particle. Enamel indenter sliding on Estilux at an applied load of 6 N (scratching direction: top to bottom).

40 nm, the interface between the particles and the matrix is not the most important factor. Instead, the bond strength of the prepolymerized splintered particles may be very significant. As the bond is relatively weak [17–19], cracks may tend to follow the prepolymerized particle–matrix interface, Fig. 12. Consequently, these particles are easily debonded and detached, which contributes to wear. However, no direct evidence has been obtained in this study.

As shown in Figs. 3 and 4, in the case of the diamond indenter, Durafill has better wear resistance than Estilux. This result is related to the formation of tensile cracks in Durafill. As the formation and propagation of the cracks absorb a lot of energy compared to the other processes of wear, there is less available energy for the formation of transfer films and their detachment is reduced, leading to a decrease in the specific wear rate.

Similar to Estilux, the enamel indenter sliding on Durafill surface at low loads (≤ 6 N) caused almost no material removal on a macroscopic level except for microgrooves formed by debris particles left behind (Fig. 13) and minor debonding of the prepolymerized particles from the matrix resin. At higher loads, however, a small amount of material was removed and tensile cracks could be seen at the interfaces. Fig. 14 clearly illustrates this wear phenomenon.

3.4. Comparison of diamond and enamel indenters

In this study, two types of indenters were used. The purpose is to investigate the effect of indenter material

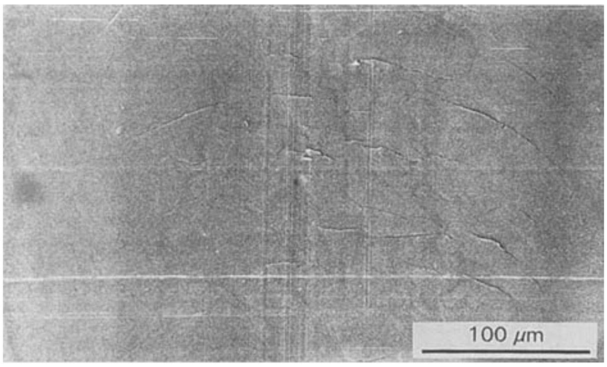

Figure 14 Scratched surface on Durafill by an enamel indenter showing tensile cracks at an applied load of 20 N (scratching direction: top to bottom).

on the wear results and to choose an indenter suitable for evaluating wear of a dental material. Comparison of the results obtained with diamond and enamel indenters shows that a better simulation of actual abrasion is obtained with the enamel indenter. This is because the diamond indenter can cut through the filler particles with ease and frequently produces large wear particles that contain both matrix material and filler particles, whereas the enamel indenter abrades the fillers much less efficiently.

Compared to diamond, the enamel indenter is easily subjected to mutual wear damage because of its low hardness. For example, the tip radius of the enamel indenter sliding on Estilux changed from the original 252 μm to 382 μm over the range of loads tested, whereas for sliding on Durafill it changed from 293 μm to 313 μm [17]. Therefore, the diamond indenter may be more suitable to study the fundamental wear behaviour of dental composite resins, but the enamel indenter may provide a more successful simulation of the material removal processes that occur in the clinical situation.

The experimental results, however, clearly indicate that the basic wear mechanisms are different depending on the type of indenter (diamond or enamel). It is noted that in using a diamond indenter, plastic ploughing is the main wear mechanism in Estilux and tensile microcracking is predominant in Durafill. However, when the enamel indenter is used, the wear mechanism is changed. For Estilux, if the applied load is equal to or below 6 N, polymer flakes are formed in wear but no plastic ploughing is found. At higher load (> 6 N), particle fracture and debonding are the main wear mechanisms. In Durafill, when the applied load is below 6 N, no tensile cracks can be found (Fig. 13). But if the load is higher than 6 N, tensile cracks are detected on the worn surface.

4. Conclusions

In-vitro studies of the material removal mechanisms during abrasion of dental composite resins have been conducted by using single-pass scratch tests with diamond and enamel indenters. For the range of applied load used the coefficient of friction (μ) remains constant for both Estilux and Durafill. With the enamel indenter, μ is higher in Estilux than Durafill. For the diamond indenter, μ is the same for both composite resins.

The material loss in Durafill was found to be much less than that in Estilux when subjected to abrasive wear using a diamond indenter. Observations using SEM showed a ductile failure mode of Estilux where the material was plastically deformed and then either dislodged to form wear debris or forced to pile up on both sides of the wear groove. With the enamel indenter, much less surface damage was found on the worn surface of Estilux, except that some filler particles were broken and plucked out at higher loads. In contrast, brittle tensile cracking was the dominant surface failure mechanism of Durafill. The formation and propagation of tensile cracks were closely linked to the degradation of the polymer matrix and the interface of the prepolymerized particles because Durafill is a matrix-rich composite resin.

Acknowledgements

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