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A rolling-ball device for producing surface fatigue and its application to dental materials

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A new method of producing and evaluating surface fatigue using a rolling-ball device has been developed. The method involves constraining a rolling ruby ball between the ''v'' groove of a rotor and the test specimen. The ball applies a compressive stress to the surface of the test material whilst it rolls in a circular pattern across the specimen surface. The fatigue life is defined as the time taken for surface degradation to begin to occur. The method is simple and reproducible and allows fatigue data to be gathered using a relatively small number of specimens. A series of model dental composites having varying filler fractions (23.7–66.4 vol%) were used to assess the potential of the method. The pattern of material loss as well as scanning electron microscopy examination of the damaged surfaces of test specimens confirmed that a fatigue mechanism was responsible for material loss. The fatigue life varied markedly with filler volume fraction being optimized at values in the range 30–50 vol%. Lower and higher volume fractions reduced the fatigue life. Filler silanation significantly improves fatigue life. The results suggest that the rolling ball device will prove useful in comparing the properties of different materials and in the development of improved products.

1. Introduction

It is recognized that wear processes of dental restorative materials may be related in part to surface fatigue. One of the major limitations to the use of posterior composites has been localized material loss in contact areas, due to fatigue [1, 2]. Despite this clinical problem it has been difficult to devise a method which is suitable for the study of the surface fatigue of composites *in-vitro*. Methods used previously include compressive fatigue [3–5] and flexural fatigue [5–8] which involve testing cylindrical or beam specimens of materials to destruction through cyclic loading. These methods, though providing some useful information about the test material suffer some disadvantages. Firstly, the bulk failure observed when specimens undergo catastrophic failure may not be related to loss of surface material by 'fatigue wear". Secondly, experimental procedures designed to cause bulk fracture by fatigue normally produce a large scatter in the results. The consequence of this is that either differences between materials are difficult to demonstrate and/or that very large numbers of test specimens are required.

Other approaches have involved the application of multiple compressive forces onto the surfaces of test materials in order to produce surface (as opposed to bulk) degradation [9, 10]. In these experiments the fatigue characteristics were assessed indirectly by observing cracks and damage zones on the sectioned surfaces of the test specimens. A similar method has been used [11] to induce marginal defects in composites through a fatigue mechanism.

The purpose of the work reported here was to develop a method for the study of the surface fatigue of restorative materials with the following design criteria:

(1) The method should produce loss of material by surface fatigue, not bulk fracture.
(2) The method should be capable of allowing $> 10^6$ cycles of stress to be applied over a reasonable time period.
(3) The results should be reproducible and enable testing to be performed with fewer specimens than are normally required for fatigue tests.

The method which was devised to meet these requirements is based on the principle of a "rolling ball" applying cyclic loading onto the surface of a test specimen. Surface failure is detected by the development of a "fatigue track".

Preliminary work has established the most appropriate test conditions for use with the method [12–14]. The purpose of the current work was to evaluate the use of the rolling ball device as a means of studying surface fatigue by using a series of model dental composites. The materials had varying filler volume fraction. In some, the filler was silanated whilst in others it was not.

2. Experimental procedure

2.1. Rolling ball device

The rolling ball surface fatigue device is shown in Figs 1 and 2. It consists of a balanced beam which is constructed from a quartz rod pivoted at a frictionless

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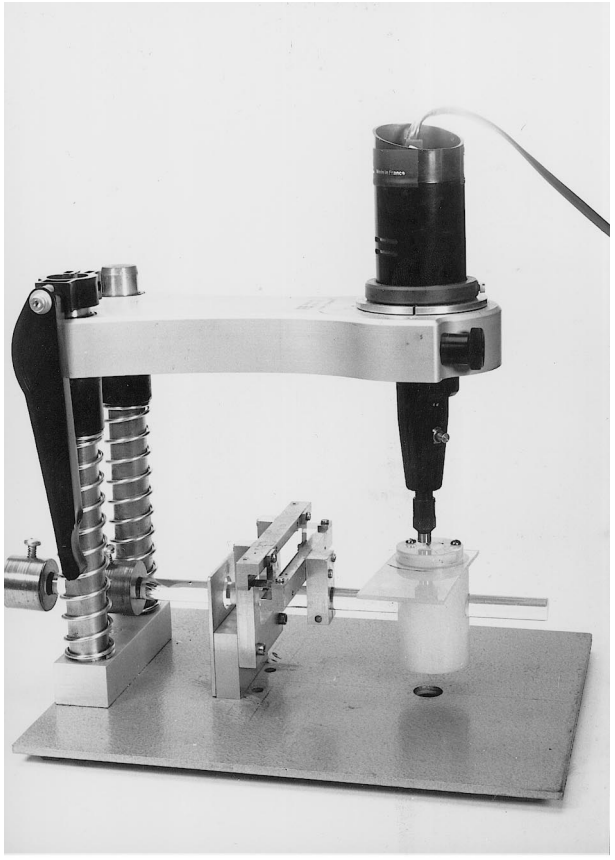

Figure 1 Photograph of the rolling ball surface fatigue apparatus.

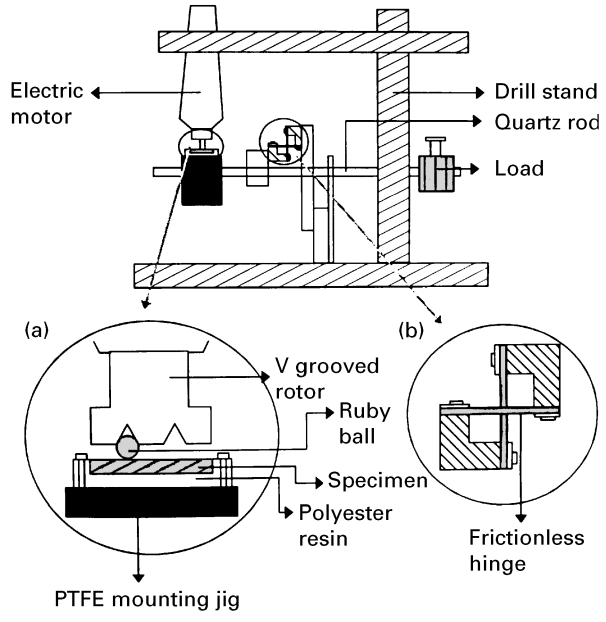

Figure 2 Line diagram of the rolling ball surface fatigue apparatus.

stainless steel hinge. The specimen holder is located at one end of the balanced quartz beam and this is counterbalanced by weights at the other end of the beam. The other main component of the equipment is the electric motor which is used to drive a "V" grooved stainless steel rotor. The rolling ball is constrained between the "V" groove of the rotor and the test specimen surface resulting in three point contact (2 contacts with the "V" groove of the rotor and one with the specimen surface). This ensures that the ball rolls and does not slide during testing. The load on the ball during testing was determined by the position of the counterweights on the balanced beam. After setting up the equipment the counter weights were moved to a position such that a predetermined load was transferred to the rotor-ball-specimen assembly. The test load was confirmed using a calibrated load-cell. The speed of rotation of the rotor and the ball were determined using two methods. Firstly, a stroboscope was used to determine the rotational speed of the rotor (a dab of white paint was used to facilitate this procedure) and the speed of the rolling ball as it completed a circuit of the "V"-groove rotor and specimen surface. Secondly, a digital tachometer was used to confirm the speed of rotation of the rotor and to eliminate any harmonic effects of the strobe light.

2.2. Test materials

Ten model resin-matrix dental composites were used in the evaluation (Table I). They were manufactured by Shofu Inc, Kyoto, Japan. The resin matrix comprised a blend of Urethane Dimethacrylate (70%) and Ethyleneglycol Dimethacrylate (30%) along with small quantities of suitable activators/initiators. The filler consisted of a blend of a glass having a mean particle size of 3.1 μm and silica having a mean particle size of 0.04 μm. In some materials the glass had been silanated whilst in others it had not. The filler fraction varied from 23.7–66.4 vol%.

2.3. Test specimens

Disc specimens (12 mm diameter and 1.5 mm thick) of each material were prepared using metal moulds backed with Mylar matrix and a sheet of polymethylmethacrylate (Perspex, ICI, UK). The mould was slightly over-filled and a second Mylar strip placed over the surface. Hand pressure was used to press down the strip using a Perspex sheet, expressing the excess material. After removing the top Perspex sheet, the materials were cured by overlapping 20 s exposures to a curing light source (Visilux II, 3M Co) for a total of 2 min to ensure an optimum cure. The whole curing procedure was then repeated on the second surface of the disc. The "first-cured" surface was used in all test procedures. This surface was ground and

TABLE I Composition of model composites

Material	Filler fraction (vol %)	Filler silanation
1	23.7	yes
2	33.0	yes
3	43.0	yes
4	52.2	yes
5	57.0	yes
6	61.7	yes
7	66.4	yes
8	51.0	no
9	41.3	no
10	29.0	no

polished using 800 grit carborundum paper followed by 7 μm alumina on a rotary pre-grinder.

The specimen holders were polyester resin blocks (approx. 30 mm diameter × 15 mm thick) having a recess of 1.5 mm deep which housed the test specimen. Specimens were cemented into the recess of the block with the "prepared" surface uppermost.

2.4. Test procedure

The specimen holders, containing the test specimen was located at one end of the balanced beam. Its position was fixed through two pins which pass through the holes in the specimen block and into the PTFE mounting jig (Fig. 2). The specimen was levelled using a spirit level and the test load (200g) established through altering the position of the counter-weight. A 2 mm diameter ruby ball was located between the test specimen surface and the "V" groove of the rotor. A distilled water drip was used to wet the specimen surface during testing. The rotor was switched on and the rotor and ball set to rotate at a pre-determined speed (the ball completes 17 revolutions per second). The speed of rotation was regularly monitored throughout each test and minor adjustments to the motor made when required.

2.5. Fatigue track depth determination

At regular intervals during testing the motor was switched off and the specimen removed from the test rig for evaluation. Profilometry was performed in order to determine the depth of any fatigue track which had developed. Having previously determined that the profile results were reproducible at different sectors of the fatigue track [12, 13], the standard evaluation procedure was to profile each specimen twice at each stage. The two profiles were at 90° and resulted in four equidistant determinations of fatigue track depth. The profiling instrument (Surformeter SF101, Planar Products, UK) had a maximum z displacement of 200 μm and was accurate to -0.1 μm. After profiling, the specimens were replaced on to the equipment for further testing. The fatigue life was defined as the time (number of cycles) up to the point where surface degradation occurred. It was not easy to precisely determine this point and so the time to produce a track depth of 5 μm was used in order to compare materials. This point was determined by interpolation.

3. Results

Throughout the period of testing the speed of rotation of the rolling ball was 17 rps whilst the speed of rotation of the rotor was 34 rps.

Figs 3 and 4 show representative plots of fatigue track depth against time (plotted as number of fatigue cycles) for two materials, one with a silanated filler and one with an unsilanated filler. In each case the results from 3 separate tests are plotted with different symbols. The reproducibility of the test is apparent. In each case the materials withstand many thousands

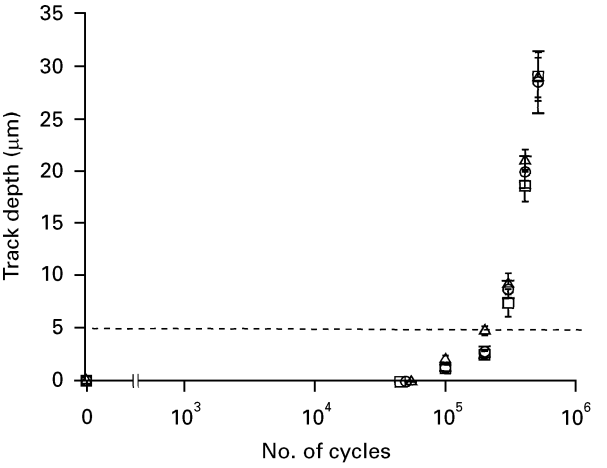

Figure 3 Fatigue track depth plotted against number of test cycles for a material containing 57.0 vol% fraction of filler for: (○) sample A, (□) sample B and (△) sample C. The glass filler was silanated. The fatigue life was defined as the number of cycles taken to generate a track of 5 μm. Error bars represent 1 s.d.

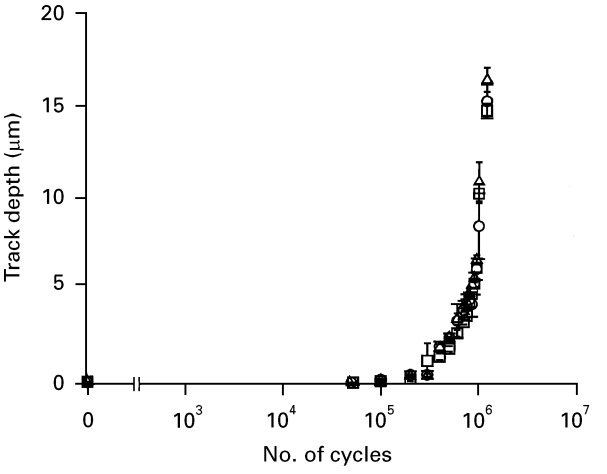

Figure 4 Fatigue track depth plotted against number of test cycles for a material containing 41.3 vol% fraction of filler. Key: (○) Sample A, (□) Sample B, (△) Sample C. The glass filler was unsilanated. Error bars represent 1 s.d.

of cycles of fatigue before surface degradation occurs. The onset of surface degradation is followed by a more rapid rate of material loss. The time to produce a fatigue track depth of 5 μm, as calculated by interpolation, was used as an estimate of the fatigue life.

Fig. 5 shows a plot of fatigue life (number of cycles) against filler volume fraction for all test materials. Materials with silanated and un-silanated fillers are plotted with different symbols. The error bars shown on this graph are standard deviations. The effect of filler volume fraction on fatigue life is highly significant ($p < .001$, Anova and Tukey test). The fatigue life of the composite system is optimized in the range 30–50 vol% filler content. The filler contents of the silanated and un-silanated samples were not identical, hence making a formal statistical comparison difficult. However, it seems clear from Fig. 5 that within the optimum range filler silanation is required to produce fatigue resistance.

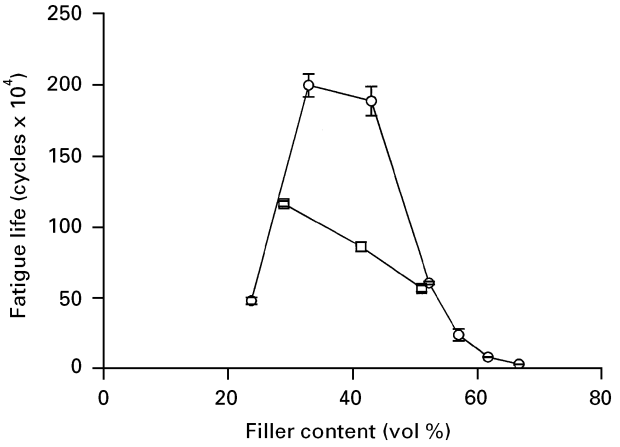

Figure 5 Fatigue life (number of cycles survived before surface degradation) plotted against filler volume fraction for; (○) silanated and (□) un-silanated glass filler. Error bars represent 1 s.d.

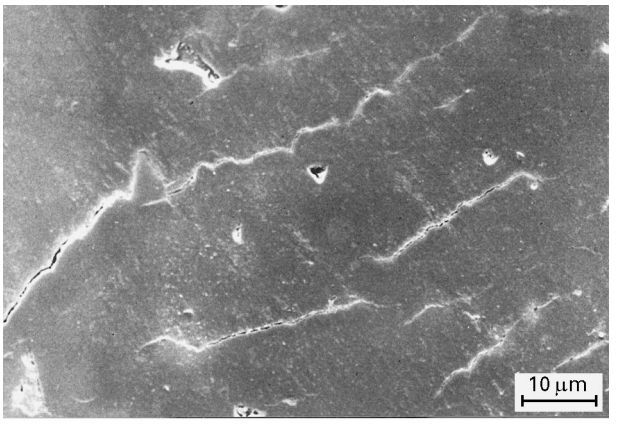

Figure 6 SEM photomicrograph showing surface of material which has been subjected to rolling ball fatigue. This view was recorded before the detection of a fatigue track. Note the surface cracks and the lack of scratches due to sliding.

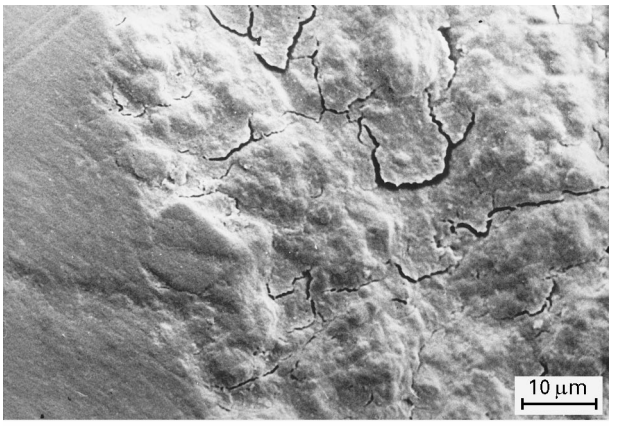

Figure 7 SEM photomicrograph showing surface of material which has been subjected to rolling ball fatigue. This view was recorded after the detection of a fatigue track. Note the loss of material, the exposure of subsurface cracks and the lack of scratches due to sliding.

4. Discussion

The term fatigue is normally considered to apply to the changes in the properties of a material which can occur due to repeated application of a stress. It is normally implied that such changes can lead ultimately to cracking and failure. When applied stresses primarily affect the surface or near-surface regions of a material crack initiation and propagation may occur in such a way as to cause loss of surface material which contributes to the process of wear. There is now a body of evidence which suggests that this is an important factor in determining localized loss of material from posterior restorations [1, 15]

In the method which we have developed, fatigue stresses are imparted to the surface layers of the test material through a rolling ruby ball. Ceramic balls have been used previously in wear studies [16, 17] but in those studies a sliding motion between the ball and the test surface was used to produce material loss through a 2-body abrasive wear mechanism. In the current work there is no abrasive effect through sliding and, typically, there is no material loss until thousands of test cycles have been completed (Figs 3 and 4).

There are three independent pieces of evidence to support a fatigue mechanism rather than abrasion in the rolling ball test. Firstly, the point contacts on the ruby ball (i.e., two point contact on the "V" groove rotor and one on the specimen) ensure that rolling occurs. Measurements of the rotational speed of the rotor and ball also offer proof that the ball is rolling and not sliding. Simple arithmetic can be used to show that under conditions of pure rolling, the ball takes exactly twice as long to complete one revolution as the rotor. Since the ratio of the rotational speed of the rotor to that of the ball was always 2 we have proof that the contact between the ball and the surface of the test specimen was a rolling contact. Secondly, the nature of the curves of depth loss against number of cycles, offers support for a fatigue mechanism [18].

There is a period of between 4×10^4–2×10^6 cycles for all the materials over which no surface damage occurs. This is followed by rapid surface breakdown indicative of slow initial crack growth followed by catastrophic failure. Damage by an abrasive wear mechanism would occur right from the start of the test and would likely cause material loss to occur as a near linear function of time [17]. Thirdly, scanning electron microscopy (SEM) studies [13] of the surfaces of the test specimens shows the absence of scratches in the fatigue wear track (Figs 6 and 7). Before any track can be detected using profilometry, surface cracking may be evident in the area subjected to stress (Fig. 6). Further testing causes crack propagations and loss of surface material (Fig. 7). During the later stages of testing it is conceivable that fatigue debris could become entrapped between the ball and the test surface leading to some scratching. This is not a problem with the current test, however, as the fatigue life is determined as the time when surface breakdown begins.

With the current experimental set-up it is difficult to precisely determine the moment at which surface breakdown begins so an arithmetic method of determining the time to reach a fixed, small fatigue track (5 μm) was used. This proved a convenient way of calculating the result and enable meaningful statistical comparisons to be performed. It is hoped that future refinements of the equipment will enable the "end-point" to be determined more precisely and that

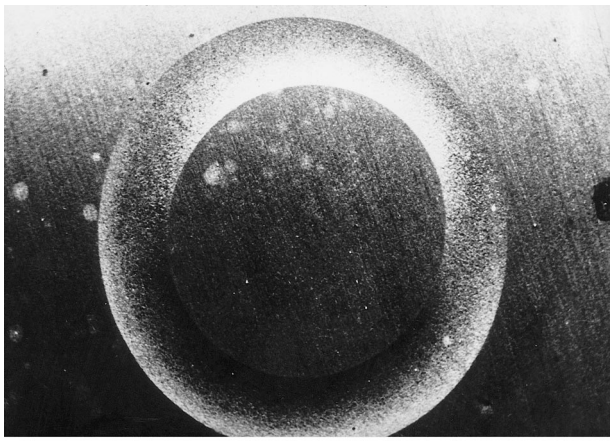

Figure 8 Low power SEM of surface of test specimen illustrating the formation of a single track.

continuous monitoring will be possible. The current test regime is quite labour intensive, as regular profiles must be made. Also when the specimen is replaced on the instrument after profiling there is a possibility that it may not be replaced in exactly the same position. This would result in multiple tracks being produced in the specimen surface. SEM was used to confirm the presence of only one track on the specimen surface (Fig. 8).

The rolling ball method gives reproducible results enabling fatigue testing to be performed with a relatively small number of specimens. Previously, the large number of specimens combined with the extended test time required for each has been identified as a major drawback for other types of methodology used to study fatigue [5, 7]. Such tests can also occupy the use of a major item of mechanical test equipment and have serious implications on the other research requirements of a busy laboratory or test-house.

In the rolling ball method the ruby ball is constrained by a 3 point contact between the "V" groove in the rotor and the test material surface. SEM imaging of the stainless steel rotors after testing showed evidence of fatigue breakdown after the rotor had been used for $> 10^8$ cycles. For this reason, rotors were replaced at frequent intervals and when individual tests required $> 10^7$ cycles for completion a fresh rotor was used for each test. None of the results described here required testing for more than 2×10^6 cycles and so rotors could be safely used for ten tests before replacement.

Our previous work has shown that the test conditions can significantly affect fatigue life [12–14]. The test load is a most important parameter. It must be of an appropriate value and must be maintained constant throughout the test. The beam system used in the rolling ball apparatus is ideal in terms of meeting the second of these two requirements. Regarding the first requirement, the fact that surface breakdown begins at $> 10^4$ cycles in all cases and sometimes occurs after 10^6 or more cycles suggests that the chosen load is sensible and results in clinically meaningful data.

The relationship between filler volume fraction and fatigue life is interesting. There appears to be a plateau region over which fatigue life is optimized. For the model system used here this was in the range of 30–50 vol%. This casts doubt on the traditional view that it is best to maximize the filler volume fraction in order to increase hardness and reduce abrasive wear. This work suggests that at both very low and very high volume fractions the fatigue life is markedly reduced. At low volume fractions the explanation could be the relatively high deformations which could occur beneath the rolling ball. These cyclic deformations may eventually lead to microcracking. At extremely high volume fractions there may be an element of increased brittleness leading to more rapid crack propagation. Other workers have shown that the lengths of subsurface cracks developed in materials subjected to repetitive impact loads is greatest at very high and very low filler volume fractions [10]. Manufactures need to find a balance of properties which will satisfy the requirements of clinical use. Good resistance to abrasive wear but poor fatigue resistance would be unacceptable for some clinical applications of the composites.

The rolling ball method has proved a convenient method for studying the surface fatigue behaviour of model resin-matrix dental composites. The method differs from those methods which involve testing of the "bulk fatigue" characteristics. In this respect the current method may give results which are more clinically meaningful. Its use can now be widened to enable comparative testing between different types of materials and studies of the way in which material composition can effect fatigue.

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