The ball mill as a means of investigating the mechanical failure of dental materials

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ABSTRACT
Objective: The main purpose of this paper is to present a new method of predicting clinical performance using mechanical loading in a ball mill.

Methods: A series of four experiments (two involving a hybrid composite and one each on orthodontic brackets and bands) is described in which the ball mill was used to subject specimens to mechanical fatigue.

Results: A reproducibility study using composite beam specimens showed no significant difference between the Mean Survival Time (MST) in all the three experimental runs (P = 0.42). When subjected to thermal cycling, the MST of the cycled group was 155.0 min compared to 247.0 min for the control group (P < 0.01). The MST of untreated and sandblasted brackets was 7.9 h and 14 h respectively (P < 0.01). There is also a significant difference (P < 0.001) in the MST of sandblasted bands when compared to the untreated bands.

Conclusions: The ball mill proved to be a convenient and reproducible means of producing mechanical fatigue and may be useful in predicting the clinical performance of dental materials.

KEY WORDS: Ball mill, Fatigue, Composites, Orthodontics


INTRODUCTION
The properties of dental materials are often evaluated using test specimens and in environments which do not closely approach that of the clinical situation, but in many cases are carefully controlled. While initial strength is an obvious requirement for clinical function, the durability of the material subjected to oral stresses over a period of time is equally important. Materials are subjected to mechanical, thermal and chemical processes in the mouth during routine drinking and eating, which by their cyclic nature invariably induce fatigue. Fatigue may lead to material or bond failures and may be classified as follows:

1. Mechanical fatigue—results from fluctuations in externally applied stresses or strains.
2. Thermomechanical fatigue—results when the temperature of the cyclically stressed materials also fluctuates.
3. Corrosion fatigue—results when the cyclic loads are imposed in the presence of a chemically aggressive or embrittling environment.
4. Fretting fatigue—occurs as a result of pulsating stresses along with oscillatory relative motion and frictional sliding between surfaces.

In a fatigue test, either the fatigue limit or the fatigue life is normally assessed. The fatigue limit is the stress below which failure is unlikely to occur within a predetermined time period, while the fatigue life is measured when a fixed fatigue stress is applied to a specimen until it fails.

The majority of research reported in the dental literature on fatigue pertains to metallic materials, but in recent years much interest has been shown in non-metallic materials and composites. The progression of fatigue damage can be generally classified into the following stages:

1. Substructural and microscopic changes which cause deformation of a permanent nature.
2. Creation of microscopic cracks.
3. Growth and coalescence of microscopic flaws to form ‘dominant’ cracks.
4. Structural instability or complete fracture. At this stage fatigue damage can occur at either a microscopic or macroscopic level. When damage occurs at a macroscopic level, it can lead to bulk fracture.

There are essentially two means of producing fa-
Mechanism of the ball mill

In a rotary mill, the energy is consumed in keeping the mill shell, the grinding media and the material charge in motion, and fractures occur as a byproduct of the passage of the material through the mill. When rotary mills were first developed, rods were often used instead of balls, and Haultain and Dyer characterized the motion of the tumbling rods in a rotating shell. There are three types of motion:

1. Rotation of the rods around their own axis.
2. Rolling of the rods down the surface of the mass (cascading).
3. Parabolic free fall above the mass (cataracting).

This has been further clarified in recent studies and it has been suggested that there are three zones of comminution in the mill (Fig. 1). Each zone is characterized by the type of action produced by the balls, namely the crushing, grinding and tumbling zones. The crushing zone consists of the falling balls crashing on the material, so that the material caught between the balls is pulverized. Grinding occurs between ball layers in the charge, where the ball layers slip past one another, grinding any material caught between them.

Grinding can take place by several mechanisms, including:

1. Impact or compression, due to forces applied almost normally to the particle surface.
2. Chipping due to oblique forces.
3. Attrition due to forces acting parallel to the surfaces.

Finally, tumbling is characterized by cascading balls that roll over one another; breakage here may be caused by impact, but an impact of a much lower energy level.

When ball milling, the factors that can be varied are:

1. Total charge.
2. Type, size and weight of the ball charge.
3. Speed of rotation.
4. Duration of grinding.

Total charge

The charge of a ball mill is the percentage of the mill interior filled with the grinding media, including the voids between the media and the batch charge. It has been suggested that optimum efficiency can be achieved when the ball mill is 60–70% full but others have suggested that the optimum charge is 30–50%.

Type, size and weight of the ball charge

A range of sizes of balls or particles from the same material can be used as the charge media in the ball mill. In order to achieve the maximum grinding power, the size of the balls should be the minimum size capable of performing the grinding action. The surface area of a given weight (or volume) of the balls varies inversely as the square of the diameter. Thus, by replacing balls with others half their size, the effective surface area for grinding is increased. The weight of balls to be used can be obtained by the sum of the volume of balls to be used and their specific gravity. An equal proportion of balls of different sizes should be represented.

Speed of rotation

The grinding efficiency is greatly affected by the speed of rotation. It has been shown that as the speed of the mill is increased, work input initially increases in proportion to the speed. Then, as slippage increases, the work input increases more slowly than the speed; it continues to increase until a critical speed is reached beyond which the power input decreases rapidly due to the solids being centrifuged onto the mill shell. It has been suggested that the movement of a single ball in a smooth-lined ball mill reaches its critical position when
the weight of the ball balances the centrifugal force on the ball, i.e. at the highest point of its path. This is a theoretical speed and is referred to as the 'critical' speed of the ball mill. The equation quoted for the critical speed is:

\[ N_c = \frac{42.3}{\sqrt{D_M \cdot D_b}} \]

where \( N_c \) is the 'critical speed' in r.p.m., \( D_M \) is the inner diameter of the ball mill and \( D_b \) is the diameter of the largest ball in metres.

In practice, ball mills are driven at 50–90% of their critical speed, the choice being influenced by efficiency and economic considerations.

The duration of grinding

A long period of grinding will not necessarily bring about greater size reduction. The aim should be to grind the material so that the desired particle size is quickly achieved throughout the batch. The time selected for grinding may be influenced by the temperature of the charge.

The development of the ball mill for dental applications

The first use of a ball mill in this laboratory was to induce fatigue on orthodontically banded and bonded teeth prior to bond strength testing. The same principle of ball milling as used in mineral processing can be applied to produce mechanical fatigue, thereby simulating the oral forces. However, the charge, force and the temperature at which the ball mill operates need to be altered for this purpose.

It was the aim of this work to determine the optimum conditions for using the ball mill as a means of subjecting dental materials to fatigue and to evaluate the method using dental composite restoratives and orthodontic luting cements.

MATERIALS AND METHODS

Apparatus

A method of inducing and testing mechanical fatigue using the ball mill was developed. A 0.5 l capacity cylindrical ceramic ball mill (Fig. 2) (Pascall Engineering Company, Crawley, Sussex, UK) was used. To operate the mill, a motor which is external to the oven causes a pair of rubber rollers to rotate at a predetermined speed. These in turn cause the rotation of the mill. Steatite spheres of diameter ranging from 12.8 to 26.5 mm were chosen. Before any experiment was carried out the maximum range of potential energy that could be generated in the ball mill was investigated. The internal diameter of the ball mill was 90 mm. The potential energy generated by the smallest and the largest steatite spheres weighing 22.14 g and 2.81 g when dropped from a 90 mm height was calculated. The potential energy that can be generated by a single impact in the ball mill was determined to be in the range of 0.003 J to 0.02 J.

In each experiment the ball mill was charged with 470 ± 1 g of steatite spheres, 250 ml of distilled water and the test specimens all maintained at 37°C. The mill rotated at 100 r.p.m. (i.e. 60% of the critical speed). At specified intervals the mill was opened and the number of failed/fractured specimens noted. Fresh distilled water at 37°C was then added and all the intact specimens were returned to the mill and testing resumed.

Four series of laboratory experiments (two involving a hybrid composite and one each on orthodontic brackets and bands) will be described to demonstrate the dental applications of the ball mill.

Reproducibility study using composite beam specimens (Experiment 1)

Thirty specimens of composite (P50, 3M, Minnesota, USA) of approximately 2 × 2 × 25 mm were prepared using a polyethylene mould. Composite was packed into the mould sandwiched between two matrix strips and two polymethylmethacrylate plates (Perspex, ICI, Welwyn, Herts, UK). Hand pressure was applied to the plates to ensure that the material was properly packed into the mould extruding all excess material. After removing the plates, the specimens were placed on a moving bed and cured on both sides using a Curing Light (Luxor, ICI, Macclesfield, Cheshire, UK) for 1 min per mm of material. After curing, the mould was sectioned using a band saw to aid removal of the specimens, thus preventing unwanted stress being applied to the specimens prior to testing. Excess flash on the specimens was removed using 800 grit carborundum paper. All specimens were stored in distilled water for 24 h at 37°C before testing. Ten specimens were tested in each run of the ball mill. The ball mill charge was as shown in Table I. At 30 min intervals, the ball mill was stopped and opened and the number of frac-
Table I. Ball charge used in the experiments

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Total weights (g)</th>
<th>Ball diameters (mm)</th>
<th>Total charge (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 and 2</td>
<td>470 ± 1</td>
<td>12.8, 19.8, 26.5</td>
<td>84</td>
</tr>
<tr>
<td>3 and 4</td>
<td>470 ± 1</td>
<td>17.4, 24.5, 26.5</td>
<td>84</td>
</tr>
</tbody>
</table>

The intact specimens and a fresh sample of distilled water at 37°C were placed in the mill and testing resumed. Testing continued up to a maximum of 5 h.

Effect of thermal cycling on survival of composite beam specimens subjected to ball milling (Experiment 2)

Sixty specimens of P50 were prepared as described above. The specimens were divided into two groups, each of 30 specimens. One group was sorted in distilled water for 24 h at 37°C (control group) and the other was subjected to 2000 thermal cycles. Each thermal cycle consisted of 60 s immersion in a water bath at 60°C, 60 s immersion in a water bath at 24°C and 60 s immersion in a water bath at 4°C with a change-over time of 55 s. All specimens were then tested as in experiment 1.

Survival of bonded orthodontic brackets subjected to ball milling (Experiment 3)

Thirty stainless steel brackets (Johnson and Johnson, Slough, Berks., UK) were bonded to extracted human premolar teeth—one bracket was bonded to each tooth. Ten brackets were bonded with Right-On (T.P., La Porte, Indiana, USA), a no-mix adhesive, following acid etching of the enamel with 37% phosphoric acid for 1 min. The remaining 20 brackets were bonded with Ketac-cem (Espe, Seefeld, Oberbay, Germany), mixed according to the manufacturer's instructions; ten had their bases sandblasted with 60 µm alumina prior to cementation—no enamel etching was carried out prior to bonding. The three sets of specimens were colour coded with an indelible pen and were then placed in the ball mill that contained a charge as indicated in Table I. Testing continued for a maximum of 20 h and after each hour the mill was opened and all bonded specimens that had failed were removed. A fresh sample of distilled water at 37°C was then added to the mill and testing recommenced.

Survival of cemented orthodontic bands subjected to ball milling (Experiment 4)

Twenty first molar bands (Johnson and Johnson) were cemented to extracted human third molars with Ketac-cem mixed according to the manufacturer's instructions. Half of the samples had their fitting surfaces sandblasted with 60 µm alumina prior to cementation. The sandblasted and untreated specimens were colour coded and placed in the ball mill containing the same weight and volume of water as used for the bonded specimens. Testing was carried out as in experiment 3 but ended at 7 h, as all bonded specimens had failed (defined as loosening of the band) by that time.

Statistical analysis

Mean survival times (MST) were determined using survival analysis (BMDP1L, University of California, USA), while a Generalized Wilcoxon (Breslow) test was used to compare the mean survival times.

RESULTS

Reproducibility study using composite beam specimens

Figure 3 shows that the test method is reproducible and the scatter between the three runs is small. Table II shows the mean survival times for all three runs which were not significantly different from each other.

Effect of thermal cycling on survival of beam composite specimens subjected to ball milling

It is evident from Fig. 4 that the control specimens survived the earlier stages of the ball milling process, whereas the specimens that have undergone 2000 thermal cycles failed rather quickly, even in the early
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Fig. 3. Reproducibility study using P50.

stages. At least 40% of the control specimens survived the test period of 5 h whereas only 20–30% of the thermocycled specimens survived. The MST for the control group was 247.0 min compared to 155.0 min for the thermocycled group. Statistical analysis with Wilcoxon revealed that the different MST for control and cycled groups was highly significant (P < 0.01).

**Survival of bonded orthodontic brackets subjected to ball milling (Fig. 5)**

Only one of the brackets bonded with Right-on failed (after 2 h of ball milling)—all others survived the test period of 20 h. Of the brackets bonded with Ketac-cem, five sandblasted brackets failed, whereas all untreated brackets failed over the 20 h test period. The MST for the untreated brackets bonded with Ketac-cem was (7.9) h, while the MST was 14 h for the sandblasted brackets bonded with Ketac-cem (P < 0.01).

**Survival of cemented orthodontic bands subjected to ball milling (Fig. 6)**

All the banded specimens had failed by 7 h of ball milling. The MST for bands with untreated and sandblasted fitting surfaces was calculated. This was 2.3 h and 5.9 h respectively; this difference was highly significant (P < 0.001).

**DISCUSSION**

This paper has described the ball mill and has indicated that it can be used as a means of simulating mechanical loading in dental materials testing. Before
was later used in experiments 1 and 2. However, the within a reasonable period of time (e.g. 24 h), and this be appropriate for different materials. The most ap-
balls) to ascertain its suitability for our study. Thirty diameter balls. The new ball charge produced results the smallest diameter balls were replaced by 26.5 mm be unreasonable as a basis for a future standard test, despite 52 h of ball milling. As this was considered to specimens were tested in the ball mill, 10 specimens at 1 capacity (i.e. 9.5 mm, 12.8 m and 19.8 mm diamter men as in experiment 1. All the specimens survived this method along with an analysis of the mechanisms of material failure.

The ball mill has been used previously in mineral processing but its adaptation for use in dental materials testing required alteration to both the charge and the testing temperature. The conditions were different for orthodontic brackets and bands for two reasons. First, there is a need to adopt specific test conditions for different types of material under test and, secondly, changes were made as our understanding of the ball mill improved.

A pilot study was carried out using the ball charge recommended by the manufacturer for a ball mill of 0.5 l capacity (i.e. 9.5 mm, 12.8 m and 19.8 mm diameter balls) to ascertain its suitability for our study. Thirty specimens were tested in the ball mill, 10 specimens at a time as in experiment 1. All the specimens survived despite 52 h of ball milling. As this was considered to be unreasonable as a basis for a future standard test, the smallest diameter balls were replaced by 26.5 mm diameter balls. The new ball charge produced results within a reasonable period of time (e.g. 24 h), and this was later used in experiments 1 and 2. However, the same range of ball charge diameter was not used in experiments 3 and 4 as the specimens involved in these experiments were larger, and as results could not be obtained in a reasonable period of time the 12.8 mm diameter steatite spheres were replaced by balls of a larger diameter. It seems that different ball charges will be appropriate for different materials. The most ap-
propriate total charge determined for each of the experiments performed is given in Table I.

Experiments 3 and 4 were performed by using distilled water initially at 37°C, and testing was carried out at room temperature in contrast to experiments 1 and 2 where testing was carried out at a maintained temperature of 37 ± 1°C in an oven. This was not possible for the earlier experiments (presented here as experiments 3 and 4), as a means of placing the ball mill in the oven had not been developed at that time. However, the temperature of the water was noted after each test interval in experiments 3 and 4 and the temperature had dropped by only 2°C following each hour of testing, probably due to the energy generated in the mill.

Central to the efficacy of the ball mill for materials testing is the mechanism of failure. The precise mechanism of failure has not been adequately described in the engineering literature; however, three processes by which a mineral may be gradually broken down in the ball mill during processing have been proposed[17]. First, the balls fall onto the material fracturing it by impact, compressive and tensile forces. Secondly, the larger particles of the materials being milled act on the smaller particles in much the same way as the balls bringing about autogenous grinding. Lastly, all the contents of the ball mill may slide over themselves and over the ball-mill wall causing compressive or shear forces leading to further breakdown by attrition or shear failure[16].

The mechanism described above implies that fracture of a material is by a single load application or impact. However, if loading is insufficient to cause failure through a single application it is likely that fracture may occur through repeated loading, i.e. a fatigue mechanism. It can therefore be inferred that these same processes may have occurred in the ball mill when used for testing dental materials.

Fracture of dental materials during ball milling is thought to occur by one of several possible mechanisms. First, by the force of impact on a perfect specimen as in the situation of a three-point bend test. This is unlikely to be a significant factor in the current test, as the potential energy generated by a single impact by the largest steatite sphere was approximately 0.02 J, whilst the energy required to fracture equivalent specimens of P50 is 0.73 J (+0.20) as determined in our laboratory using a three-point bend test. It has been previously suggested that breakage in the ball mill may be caused by impact in the tumbling zone of the mill, which is characterized by cascading balls that roll over one another, and this involves a much lower energy level than the maximum used in our calculation[5]. The energy required for fracture to occur is reduced by the presence of water. Failure caused by impact forces could also be ruled out, as nearly all control specimens in experiments 1 and 2 survived at least the first 30 min of ball milling. This is further supported by the specimen failure rate observed with thermocycling. If frac-
ture had been caused by a single impact, the failure trend would have been random or erratic and would not have followed any set pattern.

A second possible mechanism of failure may involve impact forces acting on chipped specimens. This is akin to that of a single edge-notched fracture-toughness test. The energy required to fracture a single edge-notched specimen of PSO is 0.02 J (±0.004), where the depth of the notch is half the depth of the specimen. Although this potential energy is quite similar to the maximum possible potential energy generated by the largest steatite sphere in the ball mill, fracture is unlikely to occur by this mechanism as the chip on the specimen is unlikely to be deep enough.

A third mechanism involves slow crack propagation through a prechipped specimen as in a normal fatigue process. Finally, mechanical action by the balls may cause gradual weakening of specimens leading to ultimate failure.

Specimen failure in the ball mill does not appear to be a random fracture process. The reproducible nature of the test results indicates a dependence on material properties which change under the influence of relatively small loads during contacts with the mill ball charge (Fig. 3). This is further supported by the effect of thermocycling (Fig. 4), where the specimens seem to have a shorter survival time when compared to the control group even though there was no significant difference in the flexural strength of the material (165 MPa and 162 MPa). The low stresses imparted by the steatite spheres are most likely to induce fatigue in the specimens, and failure probably occurs when the fatigue limit is reached.

It is normal to characterize fracture processes by microscopic imaging of the fracture surfaces. It has not been possible to characterize the fracture mechanisms of specimens broken in the ball mill, probably because fragments continue to be 'impacted' by the balls after fracture has occurred. This masked the structural features of the fracture surfaces which would otherwise produce useful evidence as to the mode of failure.

In relation to bonded and banded orthodontic attachments, bond strength tests give an incomplete indication of the clinical performance, and a more accurate method of testing bond reliability may be achieved by using simulated mechanical loading in a ball mill. The mean survival time was almost twice as long (14 h) for sandblasted brackets relative to untreated brackets (7.9 h), and 2.5 times longer for sandblasted bands (5.9 h) relative to untreated bands (2.3 h). One would expect, therefore, better clinical performance from brackets and bands with sandblasted fitting surfaces. This has been shown to be the case when the effect of sandblasting was investigated as part of a clinical trial. The shapes of the survival curves from the ball mill and the clinical studies were very similar, except, of course, for the fact that the ball-mill study was performed over a much shorter time-scale. It would not have been possible to propose this from the results of bond strength testing alone.

The test environment and mechanical loading in the mill are similar to that occurring in the mouth. It would appear, therefore, that the ball mill may be useful as a means of predicting the durability of restorative and orthodontics materials.

All techniques have their advantages and disadvantages, and to conclude this discussion it seems appropriate to mention those related to the ball mill. The advantages of this method compared to methods used by other workers in fatigue studies of dental materials are:

1. It is inexpensive in terms of equipment and time, and uses readily available equipment.
2. Tests are reproducible and results are produced with a relatively small number of specimens within a short period of time.
3. It produces results which can be related to clinical survival data.

The disadvantages of the ball mill are:

1. The exact forces in the mill cannot be precisely determined.
2. It is possible that some of the fractures may occur by mechanisms other than fatigue.

The experimental method described in this paper forms a basis from which a standard testing regimen could be established. From our results the following conclusions may be drawn:

1. The ball mill offers a convenient and reproducible means of producing mechanical fatigue.
2. It may be useful as a potential method of predicting the clinical performance of new restorative materials and luting agents for orthodontic brackets and bands.

References