A Comparison of the Mechanical Properties of Three Glass-Ionomer Cements

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A comparison was made on the mechanical properties of three glass-ionomer cements, one of which was of experimental fiber-reinforcing and auto-curing type. Two others were proprietary auto-curing and light-activating cements. Biaxial flexure test was conducted on disc samples. Three-point bending test was also carried out on bar samples to determine modulus of elasticity and strength. All samples were kept at 37°C and 100 % RH for 24 h before testing under ambient conditions. A prolonged fracture process was observed in the experimental cement, demonstrating the effect of fiber incorporation in stabilizing the fracture process. The proprietary cements failed in a brittle manner. Comparison of the mechanical properties identified three characteristics. These were a high Weibull modulus resulting from the stabilization in fracture process, a modulus of elasticity value comparable to that of dentin, and a high biaxial flexure strength close to that of a dental resin composite.

Key words: Glass-ionomer cement, Fiber reinforcement, Biaxial flexure strength

INTRODUCTION

Glass-ionomer cement has been increasingly used in recent years because of its favorable properties such as adhesion to tooth structure and metal, anti-cariogenic property, mild pulpal irritation and bioactive characteristic1-6). There have been many attempts to improve the properties of this cement7-11). Fiber incorporation is an interesting means of achieving better mechanical performance. Inclusion of alumina fibers was reported to improve flexure properties12) and incorporation of carbon fibers showed increases in fracture toughness and modulus of elasticity13,14). Brittle behavior of glass-ionomer cements can be changed to more ductile or predictable one by fiber incorporation, and this will prevent catastrophic fracture caused by surface and internal flaws which is common to brittle material. However, such resistance to catastrophic fracture or fracture stability of fiber-reinforced cements has not yet been demonstrated. Assessment of such improvements requires a test method which does not suffer from surface or edge defects. In an attempt to characterize the mechanical properties of current glass-ionomer cements we chose three glass-ionomer cements, one of which contained glass fibers, and conducted a biaxial flexure test in the present study. Bending test was also carried out to obtain modulus of elasticity and strength of the three materials.
MATERIALS AND METHODS

Materials and sample preparation

The fiber-reinforcing cement* was an experimental auto-curing material (hereafter, material E). Two others were proprietary materials, auto-curing** (C) and light-activating cements† (F). The powder/liquid rations (by mass) employed were 3.0 (E), 2.8 (C) and 3.0 (F). Mixing was carried out using a plastic spatula and following the manufacturers' instructions. Mixed cements were placed in Teflon moulds and pressed using a glass slab. Sizes of the moulds were 14 φ mm × 1.25 mm for biaxial flexure test and 20 mm × 5 mm × 1.6 mm for bending test. The auto-curing cement was allowed to set under a Teflon sheet and a pressure of 0.2 MPa for 10 min from the start of mixing. The light-activating material was pressed under the same pressure for 1 min and then cured for 20 s for the disc and 40 s for the beam samples through the glass slab. Two irradiations (20 s each) were made to cover the whole length of the beam. A visible-light curing unit‡ equipped with a 13 φ mm tip was used. The prepared samples were then coated with paraffin and stored in an incubator maintained at 37°C and 100 % RH for 24 h. After removing each sample from the incubator, the dimensions and mass were recorded and the test was made within 10 min. Ambient conditions during the preparation and test of samples were 23±1 °C and 50±10 % RH.

Biaxial flexure test

The disc sample was placed on three-ball (1.5 mm in diameter) supports which were equally spaced around a circle of 11.5 mm in diameter. A loading ball (1.5 mm in diameter) was positioned in the middle of the sample and the test was carried out at a cross-head speed of 0.1 mm/min by use of a universal testing machine*. The biaxial flexure strength was calculated by the following equations15,16:

\[ \sigma = \frac{AP}{t^2}, \]

\[ A = \frac{3}{4\pi} [2(1+\nu)\ln(a/b) + (1-\nu)(2a^2-b^2)/2R^2 + (1+\nu)] \]

where \( P \) is the maximum load, \( t \) thickness of the sample, \( \nu \) Poisson's ratio, \( a \) radius of the support circle, \( R \) radius of the sample. A value of 0.35 was used for Poisson's ratio17. For point loading the stress can be calculated by using an equivalent radius \( b' \). A formula, \( b' = (1.6b^2 + t^2)^{1/2} - 0.675t \), has been used where \( b \) is the actual radius of contact for \( b < 0.5t \)16. In the present study a value of 0.2 mm was used as an estimation for \( b \) from measurements made on samples after testing. At least 7 samples were tested for each material.

Three-point bending test

The beam sample was placed on supports 15.5 mm apart and the test was conducted at the same speed as for the biaxial flexure test. The modulus of elasticity was calculated for loads less than half of the maximum load and for a linear part of the force-displacement plot. Bending strength was also calculated. At least 11 samples were tested for each material.

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** CHELON-FIL, ESPE, Germany, batch no. 0024/0037
‡ Fuji II LC, GC Corporation, Japan, batch no. 100835/070731
‡‡ Optilux 400, Model 401, Demetron Research Corporation, Connecticut, USA
* AUTOGRAPH AG-E, Shimadzu Corporation, Kyoto, Japan
Statistical analyses
Weibull plots were performed on results of the biaxial flexure test and the modulus was calculated from the slope of ln $\sigma$ and ln ln [1/(1 - $P_f$)] plot, where $\sigma$ is the biaxial flexure strength, $P_f$ the fracture probability. The latter is defined by the relation $P_f = i/(N+1)$, where $i$ is the rank in strength and $N$ denotes the total number of samples\(^{18}\). Test results were also subjected to Student $t$-test.

RESULTS
Table 1 shows biaxial flexure strengths and Weibull moduli of the three cements, together with thickness and mass of the samples. Typical force-displacement curves obtained from the biaxial flexure test and Weibull plots of the strengths are shown in Fig. 1 and Fig. 2, respectively. The light-activated material F gave the highest average biaxial flexure strength. This was followed by the material C and then the fiber-reinforced material E. The three strengths were significantly different at the level of $p<0.05$. Material E gave the highest Weibull modulus followed by C and then F. Both C and F exhibited catastrophic brittle behavior, while E showed yield like behavior before the peak load and then a prolonged fracture process.

<table>
<thead>
<tr>
<th>Material</th>
<th>No.</th>
<th>Strength (MPa)</th>
<th>Weibull modulus</th>
<th>Thickness (mm)</th>
<th>Mass (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>E</td>
<td>7</td>
<td>32.2 (1.7)</td>
<td>17.1</td>
<td>1.67 (0.08)</td>
<td>520.7 (30.6)</td>
</tr>
<tr>
<td>C</td>
<td>7</td>
<td>42.1 (6.1)</td>
<td>6.3</td>
<td>1.47 (0.07)</td>
<td>455.1 (26.2)</td>
</tr>
<tr>
<td>F</td>
<td>7</td>
<td>75.2 (12.5)</td>
<td>5.2</td>
<td>1.30 (0.03)</td>
<td>423.9 (11.8)</td>
</tr>
</tbody>
</table>

The figures shown are average (standard deviation).

Fig. 1 Typical force-displacement curves from biaxial flexure test of three glass-ionomer cements, C (auto-cured), F (light-activated) and E (experimental fiber-reinforced).

Fig. 2 Weibull plots of biaxial flexure strength data of three glass-ionomer cements, E (experimental fiber-reinforced), C (auto-cured) and F (light-activated).
Table 2  Results of three-point bending test

<table>
<thead>
<tr>
<th>Material</th>
<th>No.</th>
<th>Modulus of elasticity (GPa)</th>
<th>Strength (MPa)</th>
<th>Thickness (mm)</th>
<th>Mass (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>E</td>
<td>12</td>
<td>6.4 (0.8)</td>
<td>18.4 (2.7)</td>
<td>1.75 (0.05)</td>
<td>343.0 (14.3)</td>
</tr>
<tr>
<td>C</td>
<td>11</td>
<td>13.3 (2.1)</td>
<td>29.2 (5.4)</td>
<td>1.69 (0.04)</td>
<td>349.3 (11.0)</td>
</tr>
<tr>
<td>F</td>
<td>11</td>
<td>9.3 (1.0)</td>
<td>80.5 (8.8)</td>
<td>1.69 (0.04)</td>
<td>352.9 (12.6)</td>
</tr>
</tbody>
</table>

The figures shown are average (standard deviation).

Table 2 shows moduli of elasticity and strengths obtained from the three-point bending test, together with thickness and mass of the samples. The average modulus of elasticity was highest with material C and lowest with E. The three moduli were significantly different at the level of p<0.05. The average bending strength was highest with material F and lowest with E. These strengths were also significantly different at the level of p<0.05.

DISCUSSION

Various types of biaxial test have been introduced as reliable tests for brittle material\textsuperscript{15,16,19}. The present ball-on-three-ball test provides a point-load condition and thus minimizes frictional stresses\textsuperscript{19}. The three supports make the testing possible even when a sample is slightly warped and the test results are unaffected by edge conditions\textsuperscript{15,19}. In their experiment on dental cements, Ban et al. showed deep penetration of the loading ball (1 mm in diameter) into the upper surface of thick samples\textsuperscript{20}. In general this does not occur with thin brittle samples where little permanent deformation takes place before fracture, but a realistic radius of contact must be estimated\textsuperscript{16}. The actual radius of contact, instead of using the radius of loading ball\textsuperscript{20}, was measured on samples after testing and this value was used for the calculation of biaxial flexure strength in the present study.

The strength was highest with material F, followed by C and then E in both biaxial flexure and bending tests. However, the bending strength of F was unexpectedly higher in comparison with its biaxial flexure strength. This may be attributed to the extended light exposure needed to cover the entire length of the beam samples in spite of their smaller volume (Tables 1 and 2). The highest biaxial flexure (75 MPa) and bending (81 MPa) strengths of material F are approaching the values reported for a dental resin composite (104 MPa for biaxial and 98 MPa for four-point bending test)\textsuperscript{21}. With the polymer incorporation in material F, it may be more appropriate to use a Poisson's ratio of 0.24\textsuperscript{17} for the calculation of the biaxial flexure strength and this will give a value of 70 MPa. The bending strength (29 MPa) of materials C was higher than those reported, 21 MPa (24 h) and 20 MPa (7 d)\textsuperscript{22,23}. This material had the highest value for modulus of elasticity (13 GPa) which was approaching that reported for dentin (15–19 GPa)\textsuperscript{24,25}. In terms of stiffness this material is the best replacement for dentin among the three cements studied.

One of the most important findings obtained in the present study is a prolonged failure process that the fiber-reinforced cement demonstrated. This significant resistance to fracture was shown by supplementary SEM study, where glass fibers were seen bridging and holding two fracture parts together (Fig. 3-1), in contrast to complete separation of the broken pieces in the catastrophic brittle fracture of the two other cements (Figs. 3-2 and 3...
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-3). The bond between glass fibers and matrix play a major role in stabilizing the fracture process of fiber-reinforced materials. An excessive bond will break the fibers with little, if any, pull-out of fibers and the broken ends can be seen in the plane of fracture surface. On the other hand an insufficient bond will result in complete fiber pull-out and it does not contribute in prolonging the fracture process. For these reasons an optimum bond is essential in maximizing the fracture resistance or imparting fracture stability. This fracture behavior in material E appears to be the result of some achievement in optimizing the bonding between fibers and matrix. The force-displacement curve of this material (Fig. 1) also indicates that frictional pull out of the fibers from the matrix occurs progressively during their continued displacement beyond the peak load. The onset of yield is probably due to microcracking in the matrix which will continue up to the peak load whereupon the microcracks will localize into a major crack, extending through the matrix but bridged by the fibers. The predictable nature of the strength of this cement was also shown by the Weibull modulus (Table 1). The high modulus indicates a close grouping of fracture stresses or a more predictable fracture behavior. The catastrophic fracture behavior of the other cements is typical of brittle ceramic material where strength is controlled by the size of the maximum flaw and the fracture toughness. For the fiber-reinforced material the crack stability indicates that failure is not dominated by flaw size but rather microcracking and fiber-matrix interfacial strengths. Thus the fiber-reinforced material is expected to be less prone to defects and hence exhibits a relatively higher Weibull modulus. It can be advantageous to use a material of a high Weibull modulus than a material with a slightly higher fracture strength.

A characteristic mechanical property found in each cement suggests three possible avenues for further development of glass-ionomer cement, but with some reservation for the role of resin in bioactive glass-ionomer cements. Further evaluation is necessary at an elevated temperature for a wide range of glass-ionomer cements and for the success of the optimum bonding between fiber and matrix under clinical conditions.

CONCLUSION

The present study has identified characteristic mechanical properties of three glass-ionomer cements. Material E gave the highest Weibull modulus resulting from its stabilized fracture process. Material C was closest to dentin in terms of stiffness (modulus of elasticity). The highest strength of F indicated that this cement was entering into the family of dental resin composites.

ACKNOWLEDGMENT

The authors acknowledge the support given by the staff of the University of Sydney Electron Microscope Unit.
Fig. 3  Representative scanning electron micrographs of three glass-ionomer cements after biaxial flexure test (original magnification, ×170). Experimental fiber-reinforced (top), auto-cured (middle) and light-activated cement (bottom).
REFERENCES

臼歯部用コンポジットレジンの水中浸漬下のクリープ

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市販の臼歯部用コンポジットレジンと前歯部用コンポジットレジンが、各々2種類について、その圧縮クリープと回復について水中浸漬下で研究した。
試験した臼歯部用コンポジットレジンの500時間後のクリープひずみは8.3kgf/mm²の応力下では1%未満であった。この結果、これら臼歯部用コンポジットは咬合応力に十分耐え得ることを示していた。前歯部用コンポジットレジンではフィラー含有量が臼歯部用に比べて少ないため、臼歯部用にくらべてクリープひずみが大きかった。コンポジットの回復は試験直後に著しく大きかった。500時間後のクリープ試験後の試料の吸水率は、臼歯部用コンポジットレジンでは、ある低応力下ではほぼ一定であった。

グラスアイオノマーセメントの機械的性質の比較

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三種のグラスアイオノマーセメントを選んで機械的性質を測定し、各セメントの特性を検討することにより、今後の材料開発の参考とした。三種のセメントのうち一種は、ガラス繊維を含有した自硬性の試作材料で、他の二種は市販の自硬性および光照射型セメントであった。パイアキシャルまたは通法の三点曲げ試験をおこない、後者からは弹性係数も求めた。37℃、相対湿度100%で24時間保存した試料を、室温で試験した。市販のセメントが典型的な脆性破壊挙動を示したのに比し、繊維を含有した試作材料では、破壊過程を著しく遅延する効果が認められ、これはワイヤー係数が大きくなること、すなわちより安定した破壊挙動につながった。市販のセメントで得られた特性は、象牙質に近似した弾性率と歯科用複合レジンに匹敵する強度であった。

アークイオンプレーティング法により Ni-50Ti 合金上に創成した TiN 膜の表面性状と耐食性

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アークイオンプレーティング法によりインプラント用 Ni-50Ti 合金上に創成した窒化チタン薄膜の構造と耐食性を調べた。X 線光電子分光装置を用いた角度分析により、創成された窒化チタン薄膜は三層構造となっており、最表面から TiOx, TiNx (x>1), TiN と化学状態が変化していることが明らかとなった。また、コーティング層からニッケルは検出されなかった。0.9% NaCl 浸液中におけるアノード分極曲線を測定したところ、窒化チタンでコーティングした Ni-50Ti 合金の不動態保持電流は自然浸没電位から+500mV (vs. Ag/AgCl) までは研磨状態の合金と比較しても約 1/100 となり、耐食性が向上することが明らかとなった。しかし、脱不動態電位が研磨状態の+1200mV から+500mV に低下し、孔食感受性が高くなった。分極抵抗の測定から、自