In Vitro Evaluation of Shear Bond Strength of Three Primer/Resin Cement Systems to Monolithic Zirconia

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Purpose: To investigate the shear bond strength (SBS) of various primer/resin cement systems to monolithic zirconia under different levels of storage. Materials and Methods: Disk-shaped specimens of monolithic zirconia (10 × 3 mm, n = 72) were polished with silicon carbide paper, and the bonding surfaces were sandblasted with aluminum oxide (Al₂O₃). The samples were divided into three groups (n = 24) according to primer/cement system: Z-PRIME Plus/DUO-LINK (Bisco); Clearfil Ceramic Primer Plus/PANAVIA SA (Kuraray); and Single Bond Universal Adhesive/RelyX Ultimate (3M ESPE). After bonding was completed, each group was divided into two subgroups (n = 12) under different levels of 24-hour storage and thermocycling. The specimens were embedded in acrylic molds, and SBS tests were conducted. Modes of failure were also evaluated. The data were analyzed using one- and two-way analysis of variance (ANOVA) and Tukey Honest Significant Difference test. Significance was set at \( P < .05 \).

Results: The highest and lowest SBS values were observed in the Kuraray (12.52 ± 1.34 MPa) and Bisco (5.32 ± 0.54 MPa) systems, respectively, in the thermocycled groups (\( P < .05 \)). Similarly, in short-term storage groups, Kuraray had the highest (16.47 ± 1.5 MPa) and Bisco the lowest (7.43 ± 1.06 MPa) SBS values (\( P < .05 \)). Regardless of adhesive system used, thermocycling significantly decreased the SBS of all cement groups (\( P < .05 \)). Of the failures, 49% were adhesive, 45% were mixed, and 6% were cohesive. Conclusion: A methacryloyloxydecyl dihydrogen phosphate–containing resin cement is recommended to provide a durable bond for monolithic zirconia. Int J Prosthodont 2019;32:519–525. doi: 10.11607/ijp.6258

Recently, the demand for metal-free, esthetic, and biocompatible all-ceramic materials with high mechanical properties has increased.\(^1,2\) Yttria-stabilized tetragonal zirconia was previously introduced for dental use as a core material for all-ceramic restorations.\(^3,4\) When compared to other all-ceramic systems, zirconia has the highest resistance to fracture.\(^5\) Clinical failure of zirconia restorations is mostly related to chipping or fracture of the ceramic veneer.\(^6–8\) The causes of failure are the following: differences in the coefficient of thermal expansion (CTE) between the zirconia core and ceramic veneer; rapid cooling rates; improper framework design; low flexural strength; and low fracture toughness of the ceramic veneer compared to the zirconia infrastructure.\(^9\) Other factors may have an effect, such as the thickness of the porcelain, the amount of occlusal force, and the location and size of occlusal contacts.\(^10\) Various techniques have been introduced to overcome the chipping problem, including computer-aided design/computer-assisted manufacturing (CAD/CAM)–produced veneering materials, modification of firing temperatures, and framework designs.\(^11–13\) Recently, with advances in CAD/CAM technology, a full-contour zirconia restoration called monolithic zirconia has been introduced to eliminate veneer cracking.\(^14,15\)
The amount of retention of a cemented restoration depends on many factors, including the height, width, and taper of the abutment and type of luting agent.\textsuperscript{16} Monolithic zirconia has been found to be useful for patients with limited interocclusal space because of its ability to resist occlusal loads.\textsuperscript{17} Loss of retention because of shorter abutments for patients with limited interocclusal space may be a common cause of fixed restoration failure. Therefore, using adhesive cements with a durable and acceptable bonding can overcome this problem. In addition, using adhesive cements with a ceramic restoration also enables the optical properties of the restoration to be improved.\textsuperscript{16}

Many manufacturers produce different types of adhesive systems that have different compositions and monomer types. For this reason, selecting the best type of adhesive system according to clinical situation is difficult for clinicians.\textsuperscript{18,19} Monolithic zirconia has a polycrystalline structure and lacks silica in its composition. When compared to glass-ceramics, etching with hydrofluoric acid (HF) is useless and does not enable chemical bonding between the silane and silica materials. Therefore, choosing the appropriate adhesive system is critical to producing strong and durable adhesion between monolithic zirconia and resin cements.\textsuperscript{20}

To the best of the present authors’ knowledge, although multiple studies have evaluated bond strength values for zirconia substructures, few have investigated the effect of different adhesive systems on the bond strengths of monolithic zirconia restorations.\textsuperscript{21} Therefore, the purpose of this study was to evaluate the shear bond strength (SBS) of three types of primer/resin cement systems to monolithic zirconia with short-term storage and thermocycling conditions. The null hypothesis was that no significant difference would exist between SBS values of the various primer/cement systems under either of the storage conditions.

**MATERIALS AND METHODS**

**Sample Preparation**

A power analysis was performed to determine the sample size for this study, and it was determined that the minimum number for each group should be 12 (power: 0.80, $\alpha$: .05, effect size [d]: 0.542, standard deviation [SD]: 3.7). The total number of samples was 72. Monolithic zirconia samples (Starceram Z, H.C. Starck) were prepared from presintered blocks using a CAD/CAM system (CORITEC 350i Loader, imes-icore) and then sintered to the required final dimension (10 mm in diameter and 3 mm in height) in a special high-temperature furnace (Table 1). The specimen dimensions were determined according to International Organization for Standardization (ISO) standards for dental ceramics (ISO 6872, 2008).

**Surface Treatments**

The bonding surfaces of the monolithic zirconia specimens were polished consecutively with 320-, 600-, 800-, and 1,200-grit silicon carbide papers (English abrasive) under water cooling on a polishing machine (Phoenix Beta Grinder/Polisher, Buehler) to achieve a standardized surface roughness (Ra). The Ra of each sample was measured using a surface profilometer (Perthometer M1, Mahr). Three measurements for each specimen were recorded at different locations and in different directions, and the average Ra value of each specimen was

### Table 1  Primers and Resin Cements Used

<table>
<thead>
<tr>
<th>Materials</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Lot no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Starceram Z, Monolithic zirconia</td>
<td>H.C. Starck</td>
<td>$\text{ZrO}_2$ (87% to 95%); $\text{Y}_2\text{O}_3$ (4.9% to 5.3%); $\text{HFO}_2$ (&lt; 5%); Diiron trioxide (0.1% to 2%)</td>
<td>50585968</td>
</tr>
<tr>
<td>RelyX Ultimate adhesive resin cement</td>
<td>3M ESPE</td>
<td>Base: Methacrylate monomers/radiopaque, silanated fillers/initiators/ stabilizers/rheologic additives; Catalyst: Methacrylate monomers/radiopaque, alkaline fillers/initiators/ stabilizers/ pigments/rheologic additives fluorescence dye/dark cure activator</td>
<td>644702</td>
</tr>
<tr>
<td>Single Bond Universal Adhesive</td>
<td>3M ESPE</td>
<td>MDP/Dimethacrylate resins/HEMA/Vitrebond Copolymer/filler/ethanol/water/ initiators/silane</td>
<td>609973</td>
</tr>
<tr>
<td>CLEARFIL CERAMIC PRIMER PLUS</td>
<td>Kuraray</td>
<td>3-Methacryloxypropyl trimethoxysilane/MDP/ethanol</td>
<td>1V0001</td>
</tr>
<tr>
<td>DUO-LINK composite luting cement</td>
<td>Bisco</td>
<td>Base: Bis-GMA/TEGDMAA/JDGA/glass filler; Catalyst: Bis-GMA/TEGDMAA/glass fillers</td>
<td>1700000706</td>
</tr>
<tr>
<td>Z-PRIME Plus</td>
<td>Bisco</td>
<td>MDP/carboxylic acid monomer/biphenyl dimethacrylate/ethanol</td>
<td>16000075</td>
</tr>
</tbody>
</table>
calculated as the mean of the three measurements. The mean Ra was 0.106 ± 0.040 µm, and any sample that deviated from this value was polished again using silicon carbide papers. Then, airborne-particle abrasion was applied with a machine (Basic Master, RENFERT) on the bonding surface with 120-µm aluminum oxide (Al₂O₃) for 15 seconds at 3.5-bar pressure and 10 mm from the surface. Finally, the disk specimens were ultrasonically cleaned in distilled water for 3 minutes.

**Luting Procedures**

Monolithic zirconia disks were divided into three main groups (n = 24) based on the type of primer/resin cement system (Table 1): Z-PRIME Plus/DUO-LINK (Bisco) (group 1); Clearfil Ceramic Primer Plus/PANAVIA SA (Kuraray) (group 2); and Single Bond Universal Adhesive/Relux Ultimate (3M ESPE) (group 3). Then, the monolithic zirconia disks were placed in a specially designed, separable translucent plexiglass mold, with a clearance of a 5-mm diameter and 3-mm height above the zirconia material to enable application of primer agents and resin cements. The mold was two pieces and connected with screws. First, the primer agent of each system was applied as a single thin continuous layer according to the manufacturer’s instructions. Then, resin cements were applied three times as 1-mm layers on the bonding surfaces until the mold was completely filled with the material. The resin cements were light cured with halogen photopolymerization (Optilux 501, Kerr) from the top. The bonded samples were removed from the mold after curing by loosening the screws, and the thin excess materials were removed.

**Storage and Aging**

Each group was divided further into two subgroups (n = 12) according to the different storage conditions. In short-term storage, samples were stored in distilled water for 24 hours at 37°C. In long-term storage, the samples were subjected to 5,000 cycles of thermocycling between 5°C and 55°C, with a dwell time of 15 seconds at each temperature and a transfer time from one bath to the other of 10 seconds.

**SBS Test**

After short- and long-term storage procedures were completed, all samples were embedded in a chemically cured acrylic resin with metal blocks. An SBS test was performed using a universal testing machine (Instron 3345). All samples were achieved at a crosshead speed of 1 mm/minute. Knife-edge blade equipment was employed parallel to the shearing force and as near as possible to the interface between the monolithic zirconia and resin cement (Fig 1). The forces during failure were registered in Newtons (N) and then changed into MPa using the following formula:

\[
\text{Shear stress (MPa)} = \frac{\text{load (N)}}{\text{area (mm}^2\text{)}}
\]

\[
\text{Area} = \pi \times d^2, \quad \text{and} \quad d = \text{exact diameter of the bonding surface.}
\]

The mean fracture load and SD for each group were calculated from these data.

**Failure Analysis**

A stereomicroscope (Carl Zeiss Surgical) at ×12 magnification was used to assess the mode of failure, which was classified as an adhesive failure between the monolithic zirconia and resin cement, a cohesive failure within the resin cement, or a mixed failure that was a combination of both.

**Statistical Analyses**

During data assessment, an IBM SPSS 22 program was used for statistical analyses. The conformity of the parameters to the normal distribution was assessed using the Kolmogorov-Smirnov test, and it was determined that the parameters were conformed to the normal distribution. Two-way analysis of variance (ANOVA) was used to analyze the effects of cement type, storage type, and their interaction on SBS. One-way ANOVA was used to test the differences in SBS between monolithic zirconia and resin cements. Tukey Honest Significant Difference (HDS) test was used for multiple comparisons. Student t test was used for the intergroup comparisons of parameters with normal distribution. The significance was evaluated at \(P < .05\).

**RESULTS**

Two-way ANOVA indicated that a statistically significant difference existed between the mean SBS values of the three groups of resin cements (\(P = .000; P < .05\)). A statistically significant difference was also found between the mean SBS values of the two storage groups.
Table 2  Evaluation of Effect of Cement Type and Storage Level on Shear Bond Strength

<table>
<thead>
<tr>
<th>Source</th>
<th>Type III sum of squares</th>
<th>df</th>
<th>Mean square</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corrected model</td>
<td>965.8199</td>
<td>5</td>
<td>193.164</td>
<td>159.411</td>
<td>.000</td>
</tr>
<tr>
<td>Intercept</td>
<td>7,824.906</td>
<td>1</td>
<td>7,824.906</td>
<td>6,457.587</td>
<td>.000</td>
</tr>
<tr>
<td>Cement</td>
<td>790.9206</td>
<td>2</td>
<td>395.4603</td>
<td>326.358</td>
<td>.000</td>
</tr>
<tr>
<td>Storage</td>
<td>164,7994</td>
<td>1</td>
<td>164,7994</td>
<td>136.003</td>
<td>.000</td>
</tr>
<tr>
<td>Cement*storage</td>
<td>10.09981</td>
<td>2</td>
<td>5.049905</td>
<td>4.167</td>
<td>.020</td>
</tr>
</tbody>
</table>

Two way analysis of variance. df = degrees of freedom.

Table 3a  Analysis of Shear Bond Strength (MPa) According to Different Primer/Cement Systems and Storage Levels (Mean ± SD)

<table>
<thead>
<tr>
<th></th>
<th>Thermocycling</th>
<th>Short-term storage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bisco</td>
<td>5.32 ± 0.54</td>
<td>7.43 ± 1.06</td>
</tr>
<tr>
<td>3M</td>
<td>8.89 ± 1.05</td>
<td>11.91 ± 0.84</td>
</tr>
<tr>
<td>Kuraray</td>
<td>12.52 ± 1.34</td>
<td>16.47 ± 1.5</td>
</tr>
<tr>
<td>P</td>
<td>.000</td>
<td>.000</td>
</tr>
</tbody>
</table>

One-way analysis of variance.

Table 3b  Analysis of Shear Bond Strength (MPa) According to Different Primer/Cement Systems and Storage Levels (P value)

<table>
<thead>
<tr>
<th>Comparison</th>
<th>Thermocycling</th>
<th>Short-term storage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bisco–3M</td>
<td>.000</td>
<td>.000</td>
</tr>
<tr>
<td>Bisco–Kuraray</td>
<td>.000</td>
<td>.000</td>
</tr>
<tr>
<td>3M–Kuraray</td>
<td>.000</td>
<td>.000</td>
</tr>
</tbody>
</table>

Tukey post hoc test.

Table 4  Number (Percent) of Modes of Failure in Each Group

<table>
<thead>
<tr>
<th>Group (storage level)</th>
<th>Adhesive failure</th>
<th>Cohesive failure</th>
<th>Mixed failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bisco (24 h)</td>
<td>10 (83)</td>
<td>0</td>
<td>2 (17)</td>
</tr>
<tr>
<td>3M (24 h)</td>
<td>7 (58)</td>
<td>0</td>
<td>5 (42)</td>
</tr>
<tr>
<td>Kuraray (24 h)</td>
<td>7 (58)</td>
<td>0</td>
<td>5 (42)</td>
</tr>
<tr>
<td>Bisco (5,000 TC)</td>
<td>8 (67)</td>
<td>0</td>
<td>4 (33)</td>
</tr>
<tr>
<td>3M (5,000 TC)</td>
<td>2 (17)</td>
<td>1 (8)</td>
<td>9 (75)</td>
</tr>
<tr>
<td>Kuraray (5,000 TC)</td>
<td>1 (8)</td>
<td>3 (25)</td>
<td>8 (67)</td>
</tr>
<tr>
<td>Total</td>
<td>35 (49)</td>
<td>4 (6)</td>
<td>33 (45)</td>
</tr>
</tbody>
</table>

TC = thermocycling.

Table 4 lists the percentages of the mode of failure for each specimen. None of the groups showed cohesive failure at the short-term storage level. Adhesive failure was the predominant failure type in the short-term storage groups, whereas mixed failure was the predominant failure type in the thermocycled groups.

**DISCUSSION**

Zirconia cementation has recently been a subject of interest because achieving an acceptable retention between cements and zirconia is a considerable challenge for researchers. A lack of knowledge about the cementation of monolithic zirconia is apparent in the literature. Various luting agents such as zinc phosphate, glass ionomer, and Bis-GMA–based resins for cementing zirconia have been analyzed. However, an ideal luting system has yet to be defined, especially for monolithic zirconia.

This study found significant differences among the SBS values of the three primer/cement systems and between two storage conditions ($P = .000; P < .05$). The interaction between the two factors was significant ($P = .020; P < .05$) (Table 2). According to one-way ANOVA, a statistically significant difference existed between the SBS values of the cements at both storage levels ($P = .000; P < .05$) (Table 3). Post hoc evaluation was performed to determine the cement type that caused the difference (Table 3), and the SBS of the Kuraray cement was found to be significantly higher than the Bisco and 3M cements ($P < .05$). The mean SBS of the 3M cement was found to be significantly higher than the Bisco cement. The comparison between the SBS values of the different types of cements in short-term storage and thermocycling groups indicated that the mean SBS value of the three adhesive systems in short-term storage was statistically higher than that of the thermocycled groups ($P = .000; P < .05$).
dihydrogen phosphate (MDP) monomer in both the cement and primer of the Kuraray system. It was estimated that MDP eliminated hydrophilic components, such as water, and subsequently improved the bond strength of composite resin cements to zirconia. Although all tested groups had surface pretreatment with MDP-containing primer, additional MDP in resin cements may enhance the bond strength. It was reported that the phosphate ester group of the MDP bonds directly to metal oxides.

The results of the current study are in agreement with those of previous studies. It was demonstrated that surface treatment with MDP-containing primer reinforced the bond strength initially and even after aging by thermocycling. de Oyague et al. demonstrated that an MDP-containing adhesive system was best for zirconia bonding and that pretreatment of the surface was not necessary. Kim et al. reported that bonding to zirconia was improved when MDP-containing primer was applied. Wegner and Kern showed that resin cements containing MDP with sandblasted zirconia surfaces yielded better results. Similarly, Piwowarczyk et al. claimed that MDP-based cements showed higher and more stable adhesion to zirconia. A study by Luthy et al. reported that all cements lost their SBS after aging except those containing MDP. Wolfart et al. and Re et al. found greater adhesion with the cements containing MDP.

MDP is a comparatively hydrophobic monomer because it has a 10-carbon chain and contains two terminal ends: a hydrophilic phosphate, which chemically adheres to the surface of zirconia, and a polymerizable methacrylate, which adheres to the resin cement. Many commercial ceramic primer systems exist and are prepared for use with zirconia ceramic restorations. They differ in terms of the type and concentration of phosphate ester monomers, time of application, clinical mechanism for use, and proprietary formulas. The self-adhesive resin cements that contain phosphate monomers are assured to be more effective in adhering to non–glass-based polycrystalline ceramics. Functional phosphate monomers act as mediators between organic and inorganic substrates and are similar to organosilanes. Phosphate monomers contain an organofunctional group. This group reacts first with the resin cement’s organic matrix on one end and with the phosphate ester group on the other end. When the hydroxyl and functional phosphate ester groups react on the ceramic oxide’s surface, a bond is achieved.

MDP has the ability to develop a strong ionic and hydrogen bond with metallic oxide in the zirconia ceramic surface. An MDP-containing primer has crosslinking branches (polymerization groups) that react with the Bis-GMA and 2-hydroxyethyl methacrylate of a composite resin matrix and achieves strong bonds when the composite resin polymerizes. Some studies have explained that resin composite cements that contain Bis-GMA primarily work through micromechanical interlocking. Furthermore, the self-adhesive phosphate monomer PANAVIA SA cement of the Kuraray system has a terminal-end hydroxyl group, which provides hydrolytic stability of the resin under acidic conditions and water. This may explain the Kuraray system having the highest SBS in this study. In addition, the 3M system had better results than the Bisco system, which is a conventional Bis-GMA resin cement. The 3M system has methacrylate monomers that establish the primary bond with the methacrylate resin in the primer and may improve the bond strength.

Resin cements are recommended for use with zirconia more than other types. The chemical adhesion created by resin cements is the most critical factor for bonding to zirconia, which necessitates that the appropriate cement type be selected. This is the reason why various types of resin cements were tested in this study. In addition, the surface pretreatment of zirconia derived from modifying the surface structure is also a crucial factor. Sandblasting with silica-coated Al2O3 particles using, for example, Rocatec or CoJet systems (3M) was found to be an effective surface treatment, as it generated a silica-rich surface on the zirconia. Another means of increasing micromechanical interlocking between zirconia and adhesive systems involves sandblasting with Al2O3 to improve adhesion, which increases irregularities as well as the roughness, surface energy, and wettability of the zirconia. Therefore, in this study, the bonding surfaces of monolithic zirconia samples were sandblasted with Al2O3 particles.

Thermal cycling has been used as a storage method to simulate clinical conditions. Both water storage and thermocycling are employed as common techniques to test materials in in vitro studies to show their suitability for in vivo situations. Testing specimens with thermocycling accelerates the diffusion of water by changing the temperature that produces stress at the interface of two materials. This stress is related to the difference in thermal expansion of the two materials. In this study, 5,000 iterations of thermal cycling were conducted, which, according to Yap et al., is equal to 6 months of clinical function. Harper et al. showed that a dwell time of 15 seconds or more was unsuitable for clinical practice because the vital teeth of patients cannot sustain direct contact with hot or cold substances for a long period. For this reason, the duration of the water bath was set to 15 seconds in this study.

All primers used in this study had MDP monomers. The exact percentages of the active monomers were not available from manufacturer data sheets. Clearfill Ceramic Primer Plus (Kuraray) contains 3-methacryloxypropyl trimethoxysilane, MDP, and ethanol. Blatz et al.
reported that the use of a silane coupling agent/MDP-containing bonding agent plays a critical role in the durability and reliability of the composite bond to sandblasted Procera AllZirkon and is not affected by the composite resin cement, regardless of the type. This may explain why the Kuraray system achieved the best results in the current study. Although Z-PRIME Plus (Bisco) has two monomers (organophosphate and carboxylic), which makes it compatible with numerous resin cements and ensures a good effect on bonding with resin cements after sandblasting,48 Z-PRIME Plus (Bisco) had the lowest bond strength values. In addition, Single Bond Universal Adhesive has another monomer (Vitrebond copolymer) that provides a more consistent bond with different moisture levels.49 This may explain why it achieved better results than Z-PRIME Plus (Bisco) in the present study.

Regardless of the types of primer/cement systems used in this study, the SBS values were significantly decreased after being subjected to 5,000 thermocycles. Thermocycling exhibited a moisture absorption effect, which led to an increased CTE and a decreased glass transition temperature. These results were mostly derived from the use of slow thermal gradients that shear in the hydrolytic effect of water within the resin-zirconia interface. Degradation of the composite resin cement itself was also a factor. In thermocycling, a gradual change in temperature may promote a more effective aging process.49 Analysis of the mode of failure is crucial to explaining bond strength results. In the present experiment, low bond strength values were mostly associated with adhesive failures. This finding is similar to that of other research.37,50 This may explain the fact that adhesive failure was most predominant in the Bisco system. Cohesive failure was observed in the self-adhesive resin cements of Kuraray and 3M after the thermocycling test. This type of failure is consistent with the high bond strength values that were obtained and is related to the chemical affinity between zirconia and acidic monomers of self-adhesive cements. Three adhesive systems resulted in mixed failure patterns (adhesive/cohesive) and were predominant in the Kuraray system. Mixed failure was explained by intermediate bond strength values.37

Although significant differences in SBS among the tested cements were found, a value limit of 10 to 13 MPa is suggested as the minimum acceptable SBS in clinical situations.51 Therefore, the bond strength of the Bisco system (5.32 to 7.43 MPa) and thermocycled 3M (8.89 MPa) may be considered low to ensure good clinical service. On the other hand, Kuraray (12.52 to 16.47 MPa) had acceptable SBS values for clinical application.

This study has some limitations. First, the experiment was performed in a laboratory environment without contaminated specimens or oral moisture conditions that could affect the clinical application. The bond strength of the resin cement was sensitive to mechanical and chemical influences in the intraoral cavity. In addition, only a single brand of monolithic zirconia was used, and an airborne-particle abrasion surface treatment was the only treatment employed. Moreover, only the thermocycling effect was examined. In this study, the SBS test, which has the disadvantages of inhomogeneous stress distribution, stress concentration at the substrate area, and predominantly tensile stresses rather than shear stresses, was conducted. The medium used to perform the storage was distilled water, and thus no saliva was used. In future studies, other types of monolithic zirconia brands and/or other pretreatment methods should be compared. The results of this study require clinical verification.

CONCLUSIONS

The following conclusions could be drawn from this study:

- In both the short-term storage and thermocycling conditions, the Kuraray system had the highest SBS, and Bisco had the lowest SBS.
- The highest bond strength was obtained when both the primer and cement included MDP in their compositions. Thermocycling decreased the SBS of monolithic zirconia for all adhesive systems tested.

ACKNOWLEDGMENTS

The authors deny any conflicts of interest.

REFERENCES


