Effect of an internal coating technique on tensile bond strengths of resin cements to zirconia ceramics

Shuzo KITAYAMA1,2, Toru NIKAIDO1, Rena MARUOKA1, Lei ZHU1, Masaomi IKEDA3, Akihiko WATANABE4, Richard M. FOXTON5, Hiroyuki MIURA6 and Junji TAGAMI1,2

1Department of Restorative Science, Graduate School, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
2Global Center of Excellence (GCOE) Program; International Research Center for Molecular Science in Tooth and Bone Diseases at Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
3Faculty of Dentistry, School for Dental Technology, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
4Institute of Biomaterials and Bionengineering, Tokyo Medical and Dental University, 2-3-10 Kanda-surugadai, Chiyoda-ku, Tokyo 101-0062, Japan
5Department of Conservative Dentistry, Floor 25, King’s College London Dental Institute at Guy’s, King’s and St. Thomas’ Hospitals, King’s College London, London Bridge, London SE1-9RT, UK.
6Department of Restorative Science, Graduate School, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

Corresponding author, Shuzo KITAYAMA; E-mail: nuevo_centro@yahoo.co.jp

This study was conducted to enhance the tensile bond strengths of resin cements to zirconia ceramics. Fifty-six zirconia ceramic specimens (Cercon Base) and twenty-eight silica-based ceramic specimens (GN-1, GN-1 Ceramic Block) were air-abraded using alumina. Thereafter, the zirconia ceramic specimens were divided into two subgroups of 28 each according to the surface pretreatment; no pretreatment (Zr); and the internal coating technique (INT). For INT, the surface of zirconia was coated by fusing silica-based ceramics (Cercon Ceram Kiss). Ceramic surfaces were conditioned with/without a silane coupling agent followed by bonding with one of two resin cements; Panavia F 2.0 (PF) and Superbond C&B (SB). After 24 hours storage in water, the tensile bond strengths were tested (n=7). For both PF and SB, silanization significantly improved the bond strength to GN-1 and INT (p<0.05). The INT coating followed by silanization demonstrated enhancement of bonding to zirconia ceramics.

Keywords: Zirconia ceramics, Resin cement, Internal coating technique

INTRODUCTION

The popularity of all-ceramic restorations has increased in recent years due to their superior esthetic appearance and metal-free structure. Computer-aided design and manufacturing (CAD/CAM) has become an increasingly interesting alternative to manual, casting, or pressing techniques.

Zirconia is a high flexural strength ceramic which has been used as an orthopedic material. Based on these improved physical properties compared with alumina-based ceramics, zirconia ceramic was introduced to restorative dentistry for the restoration of posterior teeth. Polycrystalline zirconia is typically used in the tetragonal crystalline phase, stabilized with yttrium oxide (Y-TZP). Clinical applications of Y-TZP include all-ceramic cores and post systems and as copings for complete coverage all-ceramic crowns and fixed partial dentures.

Along with the strength of the material, the cementation technique is also important for the clinical success of a restoration. Due to their high fracture resistance, zirconia crowns and fixed partial dentures (FPDs) can be cemented using conventional luting methods recommended by the manufacturers. However, resin bonding between a tooth and a restoration is advocated for improving the retention, marginal adaptation, fracture resistance of restorations and inhibition of secondary caries.

Obtaining adhesion between resin cement and a ceramic surface requires surface pretreatment. The use of a silane coupling agent is recommended for glasses and porcelains in order to form a siloxane network with the silica in the ceramic surface, to improve the bond strength between the resin cement and the ceramic surface. However, these techniques do not improve the bond strength of zirconia and alumina ceramics because this chemical reaction is not possible with these ceramics. Also, their high crystalline content makes them resistant to hydrofluoric acid etching. For these high strength-ceramics, airborne-particle abrasion is an alternative method for roughening the ceramic surface.

Some studies have shown that a tribochemical silica coating increased the bond strength to airborne-particle abraded zirconia ceramic restorations. However, the marginal area of the zirconia frame sometimes chips when air-abraded or adjusted by burs. Some studies have shown that a tribochemical silica coating increased the bond strength to high-strength ceramics. However, it has also been reported that a tribochemical silica coating might be less effective for densely sintered ceramics than for glass-infiltrated ceramics. In addition, a tribochemical silica coating cannot cover all the abraded surface with silica.

In order to obtain better bonding to the internal surface of the zirconia-fabricated restoration, we...
propose a new laboratory technique, the so-called “Internal (INT) Coating Technique”. With the INT coating technique, the internal surface of the zirconia restoration is partially or fully covered with silica-based ceramics by fusion to the zirconia surface. In the laboratory, a zirconia frame with a large marginal or internal gap can be repaired with silica-based ceramics using the INT coating technique.

Therefore, the purpose of this study was to examine the tensile bond strength of two resin cements to zirconia ceramics pre-treated with or without the INT coating technique compared to that of silica-based ceramics. The null hypothesis was that the INT coating followed by silanization did not increase the bonding of resin cements to zirconia ceramics.

**MATERIALS AND METHODS**

**Materials used in this study**

The ceramic materials used in this study are shown in Table 1. Fifty-six zirconia ceramic specimens were fabricated from ingots (Cercon Base; Degudent, Hanau, Germany) according to the manufacturer’s instructions. The specimens had a diameter of 15 mm and a thickness of 2 mm. Twenty-eight silica-based ceramic specimens (GN-1, GN-1 Ceramic Block; GC, Tokyo, Japan) with a size of 13 × 17 × 21 mm were obtained from the manufacturer. GN-1 was used in order to investigate the effect of silane coupling agents on silica-based ceramics.

The resin cements and the primers for bonding ceramics used in this study are listed in Table 2.

For silanization treatment, a mixture of Clearfil

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Ceramic materials used in this study</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trade name</td>
<td>Batch No.</td>
</tr>
<tr>
<td>Cercon Base</td>
<td>18001459</td>
</tr>
<tr>
<td>Cercon Ceram Kiss (Shade: DA3)</td>
<td>34803</td>
</tr>
<tr>
<td>GN-1 Ceramic Block</td>
<td>0507121</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 2.a.</th>
<th>Resin cements used in this study</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Batch No.</td>
</tr>
<tr>
<td>Panavia F 2.0 (PF)</td>
<td>A-Paste: 0255AB</td>
</tr>
<tr>
<td></td>
<td>B-Paste: 0133AA</td>
</tr>
<tr>
<td>Superbond C&amp;B (SB)</td>
<td>Liquid: RR2</td>
</tr>
<tr>
<td></td>
<td>Powder: RK1</td>
</tr>
<tr>
<td></td>
<td>Catalyst: RR22</td>
</tr>
</tbody>
</table>

MDP, 10-methacryloyloxy decyl dihydrogen phosphate; MMA, methyl methacrylate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; PMMA, polymethyl methacrylate

<table>
<thead>
<tr>
<th>Table 2.b.</th>
<th>Primers used in this study</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Batch No.</td>
</tr>
<tr>
<td>Clearfil Porcelain Bond Activator</td>
<td>00207A</td>
</tr>
<tr>
<td>Clearfil SE Bond Primer</td>
<td>00755A</td>
</tr>
<tr>
<td>Porcelain Liner M</td>
<td>Liquid A: RL1</td>
</tr>
<tr>
<td></td>
<td>Liquid B: RL1</td>
</tr>
</tbody>
</table>

HEMA, 2-hydroxyethyl methacrylate
Table 3  Surface pretreatment protocols applied to each ceramic

<table>
<thead>
<tr>
<th>Ceramics</th>
<th>Abbreviation</th>
<th>Surface treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cercon Base</td>
<td>Zr</td>
<td>Polishing with #600 grit-SiC, Air-abrasion with alumina</td>
</tr>
<tr>
<td>Cercon Base + Cercon Ceram Kiss</td>
<td>INT</td>
<td>Polishing with #600 grit-SiC, Air-abrasion with alumina, Fusing Cercon Ceram Kiss onto the surface of Cercon Base followed by air-abrasion with alumina</td>
</tr>
<tr>
<td>GN-1 Ceramic Block</td>
<td>GN-1</td>
<td>Polishing with #600 grit-SiC, Air-abrasion with alumina</td>
</tr>
</tbody>
</table>

SE Bond Primer and Clearfil Porcelain Bond Activator (Kuraray Medical, Tokyo, Japan) was used for Panavia F 2.0 (PF, Kuraray Medical). Clearfil SE Bond Primer contains MDP and Clearfil Porcelain Bond Activator contains a silane coupling agent. Porcelain Liner M (Sun Medical, Moriyama, Japan) was used for Superbond C&B (SB, Sun Medical). Porcelain Liner M contains a silane coupling agent and 4-META in MMA.

Specimen preparation for tensile bond test
The surface pretreatment protocols applied to each ceramic are shown in Table 3. The surfaces of the zirconia and silica-based ceramic specimens were ground with #600-grit silicon carbide paper using a polishing machine (Ecomet 4; Buehler, Lake Bluff, IL, USA) and then airborne-particle abraded using a sandblaster (Hi Blaster Ovaljet; Shofu, Kyoto, Japan) with 70 μm Al₂O₃ particles (Hi Aluminas; Shofu) at 0.5 MPa air-pressure for 5 seconds at a distance of 10 mm with circular movement in order to air-abrade the circular area approximately 7 mm in diameter.

Then the zirconia specimens were divided into two subgroups of 28 each according to the surface pretreatment as follows: no treatment (Zr); and the internal coating technique (INT); the surface of zirconia was coated with micro pearls of fusing porcelain (Cercon Ceram Kiss, Shade DA3; Degudent, Hanau, Germany), which is a ceramic veneering material designed exclusively for use with the Cercon Zirconia system. Two stainless steel plates with a thickness of 100 μm were placed on both ends of the zirconia specimen so that the middle of the specimen on which the porcelain would be coated was exposed. After that, the porcelain powder was stirred in an excess amount of distilled water and immediately painted on the exposed zirconia ceramic surfaces and then leveled using a spatula in order to standardize the thickness of the porcelain at 100 μm. The stainless steel plates were carefully removed and the porcelain was fired at 820°C for 1 minute in a vacuum to make a coating. After that, the surfaces of the INT specimens were air-abraded in the same way as mentioned above.

The specimens were ultrasonically cleaned in distilled water for 10 minutes and air-dried.

A piece of polyethylene tape with a circular hole 4.0 mm in diameter was positioned on the surface of the specimen to demarcate the area of bonding. Following this, the specimens in each group (n=7) were pretreated as follows; no pretreatment as a control (-) or conditioned with silane coupling agent (+).

Stainless steel rods were then bonded to the specimens with each resin cement and any excess was carefully removed with a brush. The bonded specimens were left at room temperature for 30 minutes and then stored in distilled water at 37°C for 24 hours.

The tensile bond strengths were measured using a universal testing machine (Autograph AGS-J; Shimadzu, Kyoto, Japan) at a crosshead speed of 1 mm/min.

Failure mode analysis
After the tensile test, the fractured interfaces of the specimens were examined with a light microscope (Olympus OCS 912042; Olympus, Tokyo, Japan) at 40 × magnification to examine the debonded area which was assigned to either adhesive failure between ceramics and resin cement (A) or a mixture of adhesive failure and cohesive failure in resin cements (M).

Topographic analysis of the conditioned ceramic surfaces
Ceramic surfaces airborne-particle abraded with 70 μm Al₂O₃, were examined with a SEM (JSM5310LV; JOEL, Tokyo, Japan) after sputtering using a gold alloy conductive layer of approximately 30 nm.

Surface roughness
In order to measure the surface roughness of the airborne-particle abraded surfaces of each ceramic, four specimens from each group were prepared in the same manner as described above. The surface roughness of each specimen was measured with a laser displacement meter (LC-2000; Keyence, Osaka, Japan) and was expressed as the average roughness (Ra) value as was used previously.

Statistical analysis
The tensile bond strengths were initially analyzed by three-way analysis of variance (ANOVA) to examine the effects of resin cement, ceramic substrate, and silanization. However, as there were significant interactions between all three factors, the data were analyzed by two-way ANOVA using the Bonferroni test to examine the effects of ceramic substrate, silanization, and the interaction between these two factors. The data for Ra values were analyzed by one-way ANOVA using the Dunnett’s T3 test. Significance for the above statistical tests was predetermined at a 95% confidence level, whilst the failure mode distributions were analyzed by chi-square test to a 99% confidence level.
RESULTS

Tensile bond strength
The results of the tensile bond strengths of PF and SB to each ceramic are summarized in Table 4 and 5, respectively. Statistically significant differences between resin cement, ceramic substrate, and silanization are indicated in the same Tables. Two-way ANOVAs revealed that the bond strengths of PF were influenced by both ceramic substrate ($F=4.502$, $p=0.018$) and silanization ($F=427.533$, $p<0.001$), and there was a significant interaction between the independent variables, ceramic substrate and silanization ($F=18.381$, $p<0.001$). It was also revealed that the bond strengths of SB were influenced by both ceramic substrate ($F=6.642$, $p=0.004$) and silanization ($F=89.229$, $p<0.001$), and there was a significant interaction between the independent variables, ceramic substrate and silanization ($F=31.137$, $p<0.001$).

For PF, the groups with silanization significantly improved the tensile bond strengths compared to the groups without silanization in each ceramic. INT/(+) showed significantly higher tensile bond strength than Zr/(+). On the other hand, there was no significant difference between INT/(+) and GN-1(+).

For SB, silanization significantly improved the bond strength compared to the groups without silanization in INT and GN-1, however, silanization was not effective for Zr. INT/(+) showed significantly higher bond strength than Zr/(+) and GN-1(+).

Failure mode analysis
The failure mode distributions are summarized in Table 6. None of the fracture occurred at the interface of the stainless steel rods. All the fractures after tensile testing occurred in 2 locations as follows: adhesive failure between the ceramic surface and the cement; and mixed failure involving adhesive failure and cohesive failure within the cement for both the silica-based ceramics and zirconia ceramics. Chi-square test indicated that there were no significant differences in failure mode between resin cement, ceramic substrate

### Table 4 Tensile bond strength of Panavia F 2.0 to Zr, INT and GN-1 (MPa)

<table>
<thead>
<tr>
<th>Silane coupling treatment</th>
<th>Zr</th>
<th>INT</th>
<th>GN-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>(-)</td>
<td>7.5±(1.5)</td>
<td>4.9±(0.9)</td>
<td>5.8±(0.5)</td>
</tr>
<tr>
<td>(+)</td>
<td>10.7±(1.2)</td>
<td>14.5±(1.1)</td>
<td>13.0±(1.2)</td>
</tr>
</tbody>
</table>

$n=7$. All values are mean (SD).
Within the same row, means with the same large superscript letter are not statistically different ($p>0.05$).
Within the same column, means with the same small superscript letter are not statistically different ($p>0.05$).

### Table 5 Tensile bond strength of Superbond C&B to Zr, INT and GN-1 (MPa)

<table>
<thead>
<tr>
<th>Silane coupling treatment</th>
<th>Zr</th>
<th>INT</th>
<th>GN-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>(-)</td>
<td>12.0±(1.7)</td>
<td>9.7±(1.4)</td>
<td>12.4±(1.4)</td>
</tr>
<tr>
<td>(+)</td>
<td>12.7±(1.5)</td>
<td>18.9±(1.4)</td>
<td>15.5±(1.4)</td>
</tr>
</tbody>
</table>

$n=7$. All values are mean (SD).
Within the same row, means with the same large superscript letter are not statistically different ($p>0.05$).
Within the same column, means with the same small superscript letter are not statistically different ($p>0.05$).

### Table 6 The failure mode distributins

<table>
<thead>
<tr>
<th>Ceramics</th>
<th>Resin cement</th>
<th>Silane</th>
<th>A</th>
<th>M</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zr</td>
<td>PF (-)</td>
<td>4</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(+)</td>
<td>0</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SB (-)</td>
<td>0</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(+)</td>
<td>0</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td>INT</td>
<td>PF (-)</td>
<td>4</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(+)</td>
<td>2</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SB (-)</td>
<td>2</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(+)</td>
<td>0</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td>GN-1</td>
<td>PF (-)</td>
<td>3</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(+)</td>
<td>0</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SB (-)</td>
<td>2</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(+)</td>
<td>0</td>
<td>7</td>
<td></td>
</tr>
</tbody>
</table>

A: Adhesive failure between the ceramic surface and the cement; M: Mix Failure involving adhesive failure and cohesive failure in the cement
Figure 1 shows the SEM images of the ceramic surfaces after airborne-particle abrasion. Airborne-particle abrasion with 70 μm Al₂O₃ altered the superficial ceramic layer and created sharp edges and grooves. Zr (Fig 1-a) exhibited a surface with small irregularities whereas INT (Fig 1-b) and GN-1 (Fig 1-c) showed similar surfaces with large irregularities.

**Table 7** Surface roughness values (Ra) of each ceramic airborne-particle abraded with 70 μm Al₂O₃

<table>
<thead>
<tr>
<th>Ceramics</th>
<th>Surface roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zr</td>
<td>5.4A (0.5)</td>
</tr>
<tr>
<td>INT</td>
<td>21.3B (3.6)</td>
</tr>
<tr>
<td>GN-1</td>
<td>17.9B (5.4)</td>
</tr>
</tbody>
</table>

Means with the same large superscript letter are not statistically different (p>0.05). n=4. All values are mean (SD).

and silanization (p>0.01).

**Topographic analysis of the conditioned ceramic surfaces**

Figure 1 shows the SEM images of the ceramic surfaces after airborne-particle abrasion. Airborne-particle abrasion with 70 μm Al₂O₃ altered the superficial ceramic layer and created sharp edges and grooves. Zr (Fig 1-a) exhibited a surface with small irregularities whereas INT (Fig 1-b) and GN-1 (Fig 1-c) showed similar surfaces with large irregularities.

**Surface roughness**

Means and standard deviations of the surface roughness (Ra) for each ceramic airborne-particle abraded with 70 μm Al₂O₃ are shown in Table 7. The surface roughness of INT and GN-1 was significantly higher than that of Zr (p<0.05), whereas there was no difference between INT and GN-1 (p>0.05).

**DISCUSSION**

Tetragonal stabilized zirconia ceramic is the most recently introduced dental all-ceramic material. It exhibits much higher strength and toughness than all the other commercially available dental ceramics.²⁶,²⁷ The bond between silica-based ceramics and resin cements is well-established with the application of a silane coupling agent. The use of a silane coupling agent on silica-based ceramics forms a siloxane network with the silica in the ceramic surface. However, establishing a strong and stable bond with zirconia has proven to be difficult, as the material is acid resistant and does not respond to common etching and silanization procedures.¹

Airborne-particle abrasion with Al₂O₃ is the preferred surface treatment method for high-strength ceramic materials, such as alumina and zirconia ceramics,¹,³¹,²₈⁻³¹ which creates high surface energy and promotes micro-retention. However, the results of the surface roughness and SEM images showed the air-abraded surfaces of Zr to have smaller irregularities compared to those of GN-1 and INT. Airborne-particle abrasion increased the mean flexural strength and the
monoclinic phase of TZP\textsuperscript{32,33}. Some manufacturers recommend heat treatment for zirconia after airborne-particle abrasion in order to cause the reverse monoclinic (m)→ tetragonal (t) phase transformation. However, heat treatment was not applied according to the manufacturer’s instructions in this study.

Previous studies have reported that tribochemical silica coating on zirconia ceramics improved bonding to the zirconia surface\textsuperscript{21-23}. In this technique, the surfaces are air-abraded with silica-coated alumina particles\textsuperscript{21,22}. The blasting pressure results in the embedding of silica particles on the ceramic surface, rendering the silica-modified surface chemically more reactive to the resin through silane coupling agents. However, de Oyagüe \textit{et al.}\textsuperscript{34} has recently reported that tribochemical silica coating followed by silanization was not effective for zirconia with a phosphate monomer-containing cement nor a conventional Bis-GMA resin cement.

In the present study, the bond strengths of PF and SB to INT were significantly higher than those to Zr after 24 hours water storage. For the INT coating groups, silanization significantly increased the tensile bond strengths in both PF and SB. This indicates that a chemical bond resulting from the formation of a siloxane network was facilitated between the resin cement and the surface of the INT coating. In addition, it was reported that the silane coupling agents improve the wettability of ceramic surface\textsuperscript{35}. The INT coating technique followed by silanization showed equivalent bond strengths to GN-1. In the INT coating group, there were no adhesive failures at the interface between zirconia and veneering porcelain, nor cohesive failures in veneering porcelain, which indicates that the bonding of veneering porcelain to zirconia exceeded that of resin cements to veneering porcelain. Previous studies\textsuperscript{46,57} have reported that the bond strength between zirconia and veneering porcelain ranged between 23 MPa and 33 MPa, depending on the material, test method and surface treatment of zirconia. In the present study, the failure modes didn’t always correspond to that of tensile bond strengths. This might have been related to the difference in the mechanical properties of the substrates among Zr, INT and GN-1 and also between the two resin cements.

The tensile bond strength of PF was lower than that of SB in each group. PF is a dual-cured resin cement. The resin cement was polymerized without additional light as it is questionable if the light can reach to cements through zirconia frame in clinical situations. Therefore, PF may not have been polymerized in the best condition, which might explain the lower tensile bond strength of PF compared to that of SB. SB is a MMA-based resin cement. The elastic modulus of MMA-based cement is lower than that of dimethacrylate-based cement\textsuperscript{30}. Kitsaksa \textit{et al.}\textsuperscript{39} suggested that the shear bond strengths of resin cements may be influenced by their material properties. The monomer of SB might have penetrated the air-abraded zirconia surface more easily than that of PF because of the smaller molecular size of MMA\textsuperscript{40}.

For the Zr groups, applying a mixture of Clearfil SE Bond Primer and Clearfil Porcelain Bond Activator significantly increased the tensile bond strength in PF. Clearfil SE Bond Primer contains a phosphate ester monomer of MDP, which has been demonstrated to bond directly to metal oxides\textsuperscript{41}. Previous studies have shown that the application of an MDP-containing bonding/silane coupling agent mixture to zirconia ceramic restorations yielded superior shear bond strength\textsuperscript{18,20,42}.

Porcelain Liner M contains a carboxylic acid monomer, 4-META, which is supposed to have a chemical affinity to metal oxides\textsuperscript{43,44}. However, the present study showed that the application of Porcelain Liner M did not improve the bond strength. Ozcan \textit{et al.}\textsuperscript{45} reported that all the zirconia specimens bonded with Superbond debonded after thermal cycling. On the contrary, Derand \textit{et al.}\textsuperscript{46} reported that the bond strength of Superbond to zirconia did not decrease after 2 months water storage compared to that after one day.

Reuter \textit{et al.}\textsuperscript{47} reported that silanized interfaces appear to be unstable in humid conditions and the silane bond was found to deteriorate in moisture. Since the resins are permeable to water, the bond between silane and resin composite is expected to deteriorate by hydrolysis over time.

Beuer \textit{et al.}\textsuperscript{47} reported that three CAD/CAM systems of zirconia fabrication showed marginal gaps below 120 μm which were considered clinically acceptable. On the other hand, Reisch \textit{et al.}\textsuperscript{48} reported that the marginal gaps and internal fitness of zirconia fabricated FPDs varied between 8 μm and 272 μm, and between 39 μm and 502 μm, respectively. In clinical situations, the coating should be thin when the gap is small. On the other hand, after sintering or adjusting by burs, a zirconia frame with a large marginal or internal gap can be repaired fully or partially with silica-based ceramics using the INT coating in the laboratory. The porcelain coating of 100 μm was made experimentally to standardize the thickness by a dental technician in the present study. We are currently investigating the internal fitness and the marginal adaptation of the zirconia frame using the INT coating with different thickness and techniques.

Therefore, further studies should be carried out to confirm the hydrolytic stability of the bond of resin cement to a zirconia surface using the INT coating technique \textit{in vitro}, and also the application of the INT coating technique in the laboratory should be improved. Moreover, clinical evaluations of zirconia restorations are required to establish reliable application methods.

**CONCLUSIONS**

Within the limitations of this study, the following conclusions were drawn:

Surface treatment of zirconia using the INT coating technique followed by silanization can successfully increase the bond strength of the resin cements to
zirconia ceramics.

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