Influence of roughening procedures and priming agents on shear bond strength of CAD/CAM materials to zirconia frameworks

Futoshi KOMINE, Fumiaki KIMURA, Kei KUBOCHI, Ryoki TAKANO, Daishi NAKASE and Hideo MATSUMURA

Department of Fixed Prosthodontics, Nihon University School of Dentistry, 1-8-13, Kanda-Surugadai, Chiyoda-ku, Tokyo 101-8310, Japan

Corresponding author, Futoshi KOMINE; E-mail: komine.futoshi@nihon-u.ac.jp

This study investigated the influence of roughening procedures and application of primers on shear bond strengths of CAD/CAM composite resin material or ceramic material to zirconia frameworks. A CAD/CAM composite resin block (Katana Avencia Block; AVE) and lithium disilicate glass-ceramic block (IPS e.max CAD; IEC) were used as veneer materials. The veneers were divided into three surface treatment groups; HF, hydrofluoric acid etching; AB, airborne-particle abrasion; and CON, no surface treatment. Each veneer was primed with four agents: Clearfil Porcelain Bond Activator (ACT), Clearfil Photo Bond (CPB), Clearfil Photo Bond with Porcelain Bond Activator (CPB+ACT), and no priming (UP). The zirconia frameworks and AVE or IEC veneers were resin-bonded. In the AVE specimen, AB treatment showed significantly higher shear bond strength than the other treatments at 0 and 20,000 thermocycles, except for UP and CPB+ACT groups at 20,000 thermocycles. Airborne-particle abrasion is necessary for resin bonding to Avencia blocks.

**Keywords**: CAD/CAM, Ceramics, Composite resin, Dental veneer, Zirconia

---

**INTRODUCTION**

Zirconium dioxide (zirconia) ceramics have been employed for frameworks of single or multiple fixed dental prostheses (FDPs) in both anterior and posterior areas. Although the zirconia FDPs yield superior and durable clinical outcomes, many clinical studies showed relatively high levels of veneer chipping in zirconia FDPs\(^1,2\). To minimize veneer chipping, several digital approaches have been introduced into the fabrication of zirconia FDPs. Digital approaches such as CAD-on techniques and Rapid Layer Technology (RLT) involve a zirconia framework and a veneer, which are independently manufactured with computer-aided design/computer-aided manufacturing (CAD/CAM) techniques and combined using fusion ceramics or resin luting agents\(^3-6\). Another merit of this digital approach is the decrease in the number of firings of porcelain, which results in a reduced economical cost.

For the RLT approach, the zirconia framework and ceramic veneer are manufactured with CAD/CAM, followed by adhesive bonding with resin luting agents\(^7\). Since the firing process of porcelain is unnecessary, the RLT approach involves less thermal stress with firing, greater stability of the mechanical properties of the veneer, and a reduction in construction time. On the other hand, durable bonding performance between the zirconia framework and ceramic veneer is required for long-term service of the zirconia FDPs.

It is well known that the ceramic veneering materials provide excellent esthetic features, high fracture resistance, and low wear of the materials\(^7,8\). However, previous studies demonstrated high chipping and fracture rates, and high abrasiveness to opposing dental structures\(^9-12\). On the other hand, composite resin materials have drawbacks, including low wear resistance, loss of surface gloss, and insufficient color stability\(^12-14\). Conversely, the composite resin materials provide favorable esthetic results, and functional advantages in posterior areas due to the shock-absorbing capability of the materials\(^15\). Some studies indicated that the application of composite resin materials can be a promising alternative to ceramic veneers for zirconia FDPs\(^16,17\).

CAD/CAM composite resin materials containing a polymer matrix with at least 60% filler particles by weight have been recently introduced. Although long-term clinical results are unclear because of their recent introduction, several studies exhibited favorable clinical outcomes in short-term observation and superior physical properties of the materials\(^18-20\).

To achieve adequate surface roughness of restorative materials, airborne-particle abrasion with alumina particles or hydrofluoric acid etching are preferable\(^21-24\). Airborne-particle abrasion provides the increased surface roughness and undercuts, which enable a strong micromechanical interlocking with resin luting agents\(^25,26\). However, airborne-particle abrasion induces surface flaws or defects of the materials, and consequently compromises the strength of the FDPs. It is known that hydrofluoric acid-etching has been recommended to obtain strong micromechanical interlocking for resin bonding to silica-based ceramics\(^21,22\). Hydrofluoric acid-etching produces selective dissolving of the glassy matrix and exposing crystalline structures. Kimura et al.\(^23\) evaluated the effect of different surface roughening procedures on the shear bond strength of a CAD/CAM composite resin material to a zirconia framework using a...
resin luting agent without priming. As a result, airborne-particle abrasion to the CAD/CAM composite resin material achieved a stable bond strength. However, the influence of chemical surface treatments on bond strength between CAD/CAM composite resin materials and zirconia frameworks has still not been clarified.

The objective of this current study was to evaluate the influence of roughening procedures and application of priming agents on shear bond strength of a CAD/CAM composite resin material or ceramic material to a zirconia framework. The null hypotheses were: (1) surface roughening producers do not affect bond strength between CAD/CAM composite resin materials or ceramic materials and zirconia materials, (2) priming agents do not influence bond strength, and (3) there would be no difference in shear-bond strength with and without thermocycles.

| Material/Trade name | Abbreviation | Components | Manufacturer |
|---------------------|--------------|------------|--------------|
| Zirconia ceramics   |              |            |              |
| Katana Zirconia HT  | ZR           | ZrO₂: 94.4%, Y₂O₃: 5.4% | Kuraray Noritake Dental, Tokyo, Japan |
| CAD/CAM composite resin block | | | |
| Katana Avencia block 12/ | AVE         | Al₂O₃ (20 nm), SiO₂ (40 nm): 62%, UDMA, TEGDMA | Kuraray Noritake Dental |
| Lithium disilicate glass-ceramic block | | | |
| IPS e.max CAD A2 C14 | IEC         | SiO₂: 57–80%, Li₂O: 11–19%, K₂O: 0–13%, P₂O₅: 0–11%, Zr₂O₃: 0–8%, ZnO: 0–8% | Ivoclar Vivadent, Schaan, Liechtenstein |
| Priming agent | | | |
| Clearfil Porcelain Bond Activator | ACT | 3-TMSPTA, hydrophobic aromatic, dimethacrylate, others | Kuraray Noritake Dental |
| Clearfil Photo Bond | CPB | Catalyst: MDP, HEMA, Bis-GMA Universal: Initiators, accelerators, ethanol | Kuraray Noritake Dental |
| Luting agent | | | |
| Panavia V5 | PV5 | A paste: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, fluoroalminsilicate glass filler, colloidal silica, accelerator, initiator B paste: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated aluminium oxide filler, accelerator, dl-camphorquinone, pigments | Kuraray Noritake Dental |

UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-GMA, bisphenol-A-diglycidyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; 3-TMSPTA, 3-trimethoxyxilpropyl methacrylate.
Aluminas, Shofu, Kyoto, Japan) (Fig. 1B). The bonding area of the zirconia disk was determined using single-sided tape (Star Traper-WD type P, Sakurai, Tokyo, Japan) with a circular hole of 5.0 mm. The bonding surfaces of the zirconia disks were treated with Clearfil Photo Bond (CPB; Kuraray Noritake Dental) (Fig. 1C).

The AVE and IEC blocks were sectioned with an IsoMet Low Speed saw (Buehler, Lake Bluff, IL, USA) to produce disks that were shaped with a 2.5 mm-thickness and 8.0 mm-diameter with a diamond bur (106RD, Shofu) under water cooling (Fig. 1D). The IEC veneers were then sintered in a porcelain furnace (Cerafusion Press, J. Morita, Suita, Japan) according to the manufacturer’s recommendations.

The surfaces of the AVE and IEC veneers were finished with 600-grit silicon carbide paper. The veneers were randomly assigned into three surface treatment groups ($n=88$); HF group, hydrofluoric acid etching (Porcelain Etch, Ultradent, South Jordan, UT, USA); AB group, airborne-particle abrasion with 50 µm alumina particles; and CON group, no further surface treatment (Fig. 1E). After the surface treatments, each veneer was primed with one of the following four agents ($n=22$); Clearfil Porcelain Bond Activator (ACT; Kuraray Noritake Dental), Clearfil Photo Bond (CPB; Kuraray Noritake Dental), Clearfil Photo Bond with Porcelain Bond Activator (CPB+ACT), and no priming (UP) (Fig. 1F).

The zirconia disks and AVE or IEC veneers were adhesively bonded using a dual-polymerizing resin luting agent (PV5; Panavia V5, Kuraray Noritake Dental) and light-activated polymerized from four directions for 10 s in each direction using a polymerization unit (Optilux501, Kerr, Orange, CA, USA) (Fig. 1G).

**Shear bond strength test**
Half of the specimens ($n=11$) were stored in distilled water at 37°C for 24 h (defined as 0 thermocycles). The remaining halves of the specimens in each group were then subjected to 20,000 cycles in a thermal-cycling device (Thermal Shock Tester TTS-1 LM, Thomas Kagaku, Tokyo, Japan) (Fig. 1H).

Each specimen was fixed in a steel mold and mounted in a shear testing jig (Tokyo Giken, Tokyo, Japan) according an ISO/TR 11405. Shear bond strength was determined using a universal testing machine (Type 5567, Instron, Canton, MA, USA) (Fig. 1I).

**Statistical analysis**
Data analyses were conducted with IBM SPSS Statistics, version 24.0 (IBM, Armonk, NY, USA). Although the Kolmogorov-Smirnov test showed normal distribution, the Levene test did not show dispersion for the data. Hence, all data were assessed using a nonparametric test (Kruskal-Wallis test) and a post-hoc Steel-Dwass test (Kyplot 5.0, KyensLab, Tokyo, Japan) for multiple comparisons. In addition, the Mann-Whitney U-test was performed for evaluation of the differences of shear bond strength values between 0 and 20,000 thermocycles. A significant difference was observed when the $p$ values were less than 0.05 in all analyses.

**Failure pattern analysis**
After the shear bond testing, the debonded surface of the specimens was observed using an optical microscope (Stemi DV4, Carl Zeiss, Jena, Germany) at $\times 32$ magnification. A failure pattern was classified into four categories, as shown in Fig. 2. Representative specimens, after the surface treatments and the shear bond testing,
were observed with a scanning electron microscope (SEM). The bonded specimens before shear bond testing were sectioned with a precision cutting device (IsoMet, Buehler) and were additionally observed following the same procedure.

The surfaces of the representative veneers before and after shear bond testing and addition of resin luting agents were examined with X-ray diffraction (XRD; MiniFlex, Rigaku, Tokyo, Japan) using CuKα radiation (tube voltage: 30 kV and tube current: 15 mA). X-ray diffractograms were acquired from 3° to 90° (2θ) with a scan speed of 2.0°/min. The obtained data were analyzed using X-ray analysis software (PDXL Ver 1.0 7.1, Rigaku).

The surfaces of the representative veneers before and after shear bond testing and addition of resin luting agents were examined with X-ray diffraction (XRD; MiniFlex, Rigaku, Tokyo, Japan) using CuKα radiation (tube voltage: 30 kV and tube current: 15 mA). X-ray diffractograms were acquired from 3° to 90° (2θ) with a scan speed of 2.0°/min. The obtained data were analyzed using X-ray analysis software (PDXL Ver 1.0 7.1, Rigaku).

Table 2 Shear bond strengths of the AVE veneer to zirconia at 0 and 20,000 thermocycles

|             | CON Median (IQR) | Category* | HF Median (IQR) | Category* | AB Median (IQR) | Category* |
|-------------|------------------|-----------|-----------------|-----------|-----------------|-----------|
| 0 thermocycles |                  |           |                 |           |                 |           |
| UP          | 8.9 (6.9; 10.5)  | a A       | 11.1 (8.1; 14.4)| b B       | 15.1 (12.7; 18.8)| c C       |
| ACT         | 10.7 (10.1; 11.6)| a D       | 10.8 (8.3; 13.2)| b D       | 16.7 (13.2; 19.9)| c E       |
| CPB         | 11.2 (9.4; 13.3) | a F       | 13.0 (10.9; 16.3)| b G       | 17.3 (14.3; 20.0)| c H       |
| CPB+ACT     | 11.7 (10.1; 13.6)| a I       | 13.2 (10.9; 16.2)| b J       | 18.9 (13.8; 23.4)| c K       |
| 20,000 thermocycles |     |           |                 |           |                 |           |
| UP          | 0.8 (0.6; 1.0)   | b A       | 6.4 (3.7; 9.3)  | d B       | 10.8 (8.4; 13.8)| f B       |
| ACT         | 0.4 (0.4; 0.5)   | a C       | 9.3 (7.7; 10.5) | d, e D    | 14.4 (12.2; 16.4)| f, g E    |
| CPB         | 1.8 (0.8; 2.8)   | b, c F    | 11.3 (9.8; 13.0)| e G       | 15.4 (13.6; 16.1)| f, g H    |
| CPB+ACT     | 2.3 (1.0; 3.0)   | c I       | 11.8 (8.5; 15.0)| e J       | 15.8 (12.8; 17.5)| g J       |

*Different uppercase letters in the same row exhibit statistically significant differences among the surface treatments (Steel-Dwass test; p<0.05).
*Different lowercase letters in the same line exhibit statistically significant differences among the priming agents (Steel-Dwass test; p<0.05).
IQR: interquartile range

RESULTS

Descriptive shear bond strengths and statistical analysis results of the AVE and IEC specimens are displayed in Tables 2 and 3, respectively. In the AVE specimen, the AB treatment showed significantly higher shear bond strengths than the other surface treatments at 0 and 20,000 thermocycles, except for the UP and CPB+ACT groups at 20,000 thermocycles. Among the priming groups, there was no significant difference in shear bond strength at 0 thermocycles (p>0.05) for all surface treatments. In addition, no significant differences were detected among the priming groups for the HF and AB treatments, except for the UP group.

For the IEC specimen, the shear bond strengths of the HF treatment were significantly higher than those of the CON and AB treatments at both 0 and 20,000 thermocycles. The ACT and CPB+ACT groups exhibited significantly higher bond strengths than the other priming groups at 0 thermocycles in all surface treatments. After 20,000 thermocycles, the bond strengths of the CPB+ACT group were significantly highest in all priming groups, except for the ACT group receiving HF treatment.

The reduction rates and the results of the Mann-Whitney U-test for pre- and post-thermocycling in each veneering material are showed Tables 4 and 5, respectively. In the AVE specimen, the bond strength of the ACT, CPB, and CPB +ACT priming groups for the HF and AB surface treatments did not significantly decrease with thermal cycling (Table 4). Although significant differences were found for the reduction rate of shear bond strength with thermal cycling in the IEC
Table 3  Shear bond strengths of the IEC veneer to zirconia at 0 and 20,000 thermocycles

|           | CON     | HF       | AB       |
|-----------|---------|----------|----------|
|           | Median (IQR) | Category* | Median (IQR) | Category* | Median (IQR) | Category* |
| 0 thermocycles |         |          |          |          |          |          |
| UP        | 13.6 (10.0; 16.9) | a A       | 34.2 (29.4; 38.8) | d C       | 23.3 (18.9; 29.2) | f B       |
| ACT       | 36.1 (29.0; 43.8) | c D       | 48.5 (43.9; 51.0) | e E       | 40.6 (35.0; 47.9) | g D, E    |
| CPB       | 20.9 (18.9; 23.6) | b F       | 35.3 (32.9; 38.9) | d G       | 24.1 (18.9; 29.3) | f F       |
| CPB+ACT   | 35.8 (30.9; 41.4) | c H       | 43.2 (39.3; 47.7) | e J       | 42.1 (37.9; 48.1) | g I       |
| 20,000 thermocycles | 0.8 (0.6; 1.0) | a A       | 14.1 (12.4; 17.6) | d B       | 0.9 (0.7; 1.0) | f A       |
| ACT       | 1.2 (1.1; 1.3)  | b C       | 32.7 (29.2; 38.4) | e E       | 1.7 (1.5; 2.0)  | h D       |
| CPB       | 1.1 (0.9; 1.3)  | b F       | 13.8 (9.6; 16.0)  | d H       | 1.2 (1.2; 1.4)  | g F       |
| CPB+ACT   | 2.0 (1.5; 1.8)  | c I       | 32.4 (25.5; 38.7) | e K       | 2.8 (2.4; 3.3)  | i J       |

*Different uppercase letters in the same row exhibit statistically significant differences among the surface treatments (Steel-Dwass test; *p* < 0.05).
*Different lowercase letters in the same line exhibit statistically significant differences among the priming agents (Steel-Dwass test; *p* < 0.05).
IQR: interquartile range

Table 4  Reduction rates and the results of the Mann-Whitney *U*-test in shear bond strength with 20,000 thermocycles for the AVE specimens

| Surface treatment | Priming group | Reduction rate (%) | *p*-value |
|-------------------|---------------|--------------------|-----------|
| CON               | UP            | 91.0               | <0.0001*  |
|                   | ACT           | 96.2               | <0.0001*  |
|                   | CPB           | 83.9               | <0.0001*  |
|                   | CPB+ACT       | 80.1               | <0.0001*  |
| HF                | UP            | 42.3               | 0.023*    |
|                   | ACT           | 13.9               | 0.519     |
|                   | CPB           | 12.4               | 0.243     |
|                   | CPB+ACT       | 10.6               | 0.332     |
| AB                | UP            | 28.5               | 0.023*    |
|                   | ACT           | 13.8               | 0.116     |
|                   | CPB           | 11.0               | 0.056     |
|                   | CPB+ACT       | 16.4               | 0.171     |

*Statistically significant difference between 0 and 20,000 thermocycles (Mann-Whitney *U*-test; *p*<0.05).

specimens, the reduction rate of the HF treatment was relatively low compared with that of the CON and AB treatments (Table 5).

For the AVE specimens, the number of combination failures of adhesive/cohesive failures within the resin luting agent receiving AB treatment was more than that of the CON and HF treatments at both 0 and 20,000 thermocycles (Table 6). Table 7 shows that adhesive failure at resin luting agent/zirconia materials was observed in the ACT and CPB+ACT groups for the IEC specimen at 0 thermocycles.

Figures 3 and 4 show the SEM images of the surfaces of the AVE and IEC veneers after each surface treatment, respectively. For the AVE veneers, the surface of the HF treatment showed limited porosity compared with that of the CON treatment (Figs. 3A and B). The AB treatment formed sharp edges and an irregular surface (Fig. 3C). For the IEC veneers, the surface of the HF treatment exhibited irregular and porous structures (Fig. 4B). On the other hand, sharp edges were observed...
Table 5  Reduction rate and the result of the Mann-Whitney U-test in shear bond strength with 20,000 thermocycles for the IEC specimens

| Surface treatment | Priming group | Reduction rate (%) | p-value    |
|-------------------|---------------|--------------------|------------|
| CON               | UP            | 94.1               | <0.0001*   |
|                   | ACT           | 96.7               | <0.0001*   |
|                   | CPB           | 94.7               | <0.0001*   |
|                   | CPB+ACT       | 94.4               | <0.0001*   |
| HF                | UP            | 58.8               | <0.0001*   |
|                   | ACT           | 32.6               | <0.0001*   |
|                   | CPB           | 52.4               | <0.0001*   |
|                   | CPB+ACT       | 25.0               | 0.002*     |
| AB                | UP            | 96.1               | <0.0001*   |
|                   | ACT           | 95.8               | <0.0001*   |
|                   | CPB           | 95.0               | <0.0001*   |
|                   | CPB+ACT       | 93.3               | <0.0001*   |

*Statistically significant difference between 0 and 20,000 thermocycles (Mann-Whitney U-test; p<0.05).

Table 6  Failure mode of the AVE specimens after shear bond strength test

|              | CON | HF | AB |
|--------------|-----|----|----|
|              | Av  | Az | AC | C  | Av  | Az | AC | C  | Av  | Az | AC | C  |
| 0 thermocycles |     |    |    |    |     |    |    |    |     |    |    |    |
| UP            | 11  | 0  | 0  | 0  | 8   | 0  | 3  | 0  | 4   | 0  | 7  | 0  |
| ACT           | 11  | 0  | 0  | 0  | 8   | 0  | 3  | 0  | 6   | 0  | 5  | 0  |
| CPB           | 10  | 0  | 1  | 0  | 4   | 0  | 7  | 0  | 3   | 0  | 8  | 0  |
| CPB+ACT       | 9   | 0  | 2  | 0  | 3   | 0  | 8  | 0  | 3   | 0  | 8  | 0  |
| 20,000 thermocycles |     |    |    |    |     |    |    |    |     |    |    |    |
| UP            | 11  | 0  | 0  | 0  | 11  | 0  | 0  | 0  | 10  | 0  | 1  | 0  |
| ACT           | 11  | 0  | 0  | 0  | 9   | 0  | 2  | 0  | 7   | 0  | 4  | 0  |
| CPB           | 11  | 0  | 0  | 0  | 9   | 0  | 2  | 0  | 7   | 0  | 4  | 0  |
| CPB+ACT       | 11  | 0  | 0  | 0  | 8   | 0  | 3  | 0  | 7   | 0  | 4  | 0  |

Av: adhesive failure at the veneering material/resin luting agent interface
Az: adhesive failure at the resin luting agent/zirconia material interface
AC: combination failure of adhesive/cohesive failure within resin luting agent
C: cohesive failure within resin luting agent

For the surface of the AB treatment (Fig. 4C).

Figure 5 presents the representative SEM images of combination failures for the AVE and IEC specimens after the shear bond test. Residual materials were seen on the surfaces of the AVE and the IEC specimens (Figs. 5A and B). Representative SEM images of vertical cross-sections in the AVE and IEC specimens before shear bond testing are shown in Figs. 6 and 7, respectively. The interface between the veneering material (AVE) and resin luting agent of the AB treatment showed irregularity compared to that of the HF treatment (Figs. 6A and B). For the IEC specimen, microcracks within the veneer material was observed in the AB treatment (Fig. 7A). On the other hand, no microcracks were found at the veneering material/resin luting agent interface in the HF treatment for the IEC specimen (Fig. 7B).

The XRD patterns of represented specimens of AVE and IEC are shown in Figs. 8 and 9, respectively. The AVE specimen of combination failures of adhesive/cohesive failures in the resin luting agent were both peaks for quartz and alumina (Fig. 8C). Both peaks of quartz and lithium disilicate were seen in the IEC specimen of combination failures and adhesive/cohesive failures in resin luting agents (Fig. 9C).
Table 7  Failure mode of the IEC specimens after shear bond strength test

|       | CON Av | CON Az | CON AC | CON C | HF Av | HF Az | HF AC | HF C | AB Av | AB Az | AB AC | AB C |
|-------|--------|--------|--------|-------|-------|--------|--------|-------|-------|--------|--------|-------|-------|
| 0 thermocycles |       |        |        |       |       |        |        |       |       |        |        |       |       |
| UP    | 8      | 0      | 3      | 0     | 0     | 0      | 11     | 0     | 0     | 0      | 11     | 0     |       |
| ACT   | 0      | 2      | 9      | 0     | 0     | 4      | 7      | 0     | 0     | 2      | 9      | 0     |       |
| CPB   | 5      | 0      | 6      | 0     | 0     | 0      | 11     | 0     | 0     | 0      | 11     | 0     |       |
| CPB+ACT | 0    | 1      | 10     | 0     | 0     | 3      | 8      | 0     | 0     | 2      | 9      | 0     |       |
| 20,000 thermocycles |       |        |        |       |       |        |        |       |       |        |        |       |       |
| UP    | 11     | 0      | 0      | 0     | 10    | 0      | 1      | 0     | 11    | 0      | 0      | 0     |       |
| ACT   | 11     | 0      | 0      | 0     | 5     | 0      | 6      | 0     | 11    | 0      | 0      | 0     |       |
| CPB   | 11     | 0      | 0      | 0     | 9     | 0      | 2      | 0     | 11    | 0      | 0      | 0     |       |
| CPB+ACT | 11   | 0      | 0      | 0     | 6     | 0      | 5      | 0     | 11    | 0      | 0      | 0     |       |

Av: adhesive failure at the veneering material/resin luting agent interface
Az: adhesive failure at the resin luting agent/zirconia material interface
AC: combination failure of adhesive/cohesive failure within resin luting agent
C: cohesive failure within resin luting agent

DISCUSSION

This study investigated the influence of roughening procedures and the application of priming agents on shear bond strength of a CAD/CAM composite resin material or ceramic material to a zirconia framework. Based on the present results, the airborne-particle abrasion and hydrofluoric acid etching enhanced the bonding strength...
Fig. 5 Representative SEM images (×1,000) of debonded surface for combination failure in the AVE and IEC specimens after shear bond test. A, AVE specimen; B, IEC specimen. AVE, Katana Avencia block; IEC, IPS e.max CAD; PV5, Panavia V5.

Fig. 6 Representative SEM images (×600) of the vertical cross-section of the AVE specimen before shear bond test. A, AB treatment group; B, HF treatment group. AVE, Katana Avencia block; PV5, Panavia V5; ZR, Katana Zirconia.

Fig. 7 Representative SEM images (×600) of the vertical cross-section of the IEC specimen before shear bond test. A, AB treatment group; B, HF treatment group. IEC, IPS e.max CAD; PV5, Panavia V5; ZR, Katana Zirconia. The white arrow in Fig. 7A shows microcracks in the IEC material.

Fig. 8 XRD patterns of specimens. (A) quartz of the resin luting agent, (B) alumina of the AVE specimens, and (C) surface of combination failure for the AVE specimens after shear bond test.

of the CAD/CAM composite resin material and lithium disilicate glass-ceramics to a zirconia framework, respectively. Hence, the first hypothesis, that surface roughening procedures do not affect bond strength between a CAD/CAM composite resin material or ceramic material and a zirconia framework, was disproven. The second hypothesis, that priming agents do not influence bond strength, was also disproven, because the ACT and CPB+ACT groups displayed significantly higher bond strength than the other priming groups at 0 thermocycles for the IEC specimens. The thermocycles significantly decreased the bond strength of both the AVE and IEC specimens. The third hypothesis, that there would be no difference in shear-bond strength with and without
thermocycles, was rejected.

As shown in Fig. 3, the AB treatment provided rougher surfaces compared with the other treatments for AVE veneers. This indicated that the airborne-particle abrasion yielded durable bond strength between the Avencia block and zirconia frameworks. Meanwhile, the HF treatment did not roughen the surface of the AVE veneer. This finding suggests that hydrofluoric acid cannot react with the silica phase in the Avencia block, which consists of alumina and silica nanofillers (62 wt%). Hence, airborne-particle abrasion should be recommended for roughening the surface of Avencia veneers.

For the AVE veneer, the silane and phosphate functional monomer (MDP) did not play a positive role in the bonding to zirconia frameworks. The AVE block contains nano-particle size fillers (20 and 40 nm) (Table 1)\textsuperscript{26}. As the filler particle size decreases, the surface area of the fillers increases, and the matrix components escalate in the composite resin\textsuperscript{27}. The EDX analysis of a previous study\textsuperscript{28} revealed that the alumina fillers were only detected in the AVE block. In addition, the fillers were embedded in the surrounding resin matrix\textsuperscript{28}. Therefore, it can be speculated that the silane and phosphate functional monomers (MDP) were ineffective for bonding to alumina and silica fillers of the AVE block.

A number of studies reported that hydrofluoric acid etching to lithium disilicate glass-ceramics creates honeycomb-like features, and achieves strong and durable resin bonding\textsuperscript{29}. In addition, airborne-particle abrasion also enhances the bond strength of composite resin materials to lithium disilicate glass-ceramics. In the current study, although the AB and HF treatments roughened the surface of the IEC veneer, the AB treatments exhibited inferior and unstable bond strength for resin bonding to the IEC veneers before and after thermocycles. The results can be explained in Fig. 7, which shows microcracks within the IEC veneer for the AB treatment. These findings are consistent with those of previous studies\textsuperscript{29,30}. It is assumed that the microcracks caused by airborne-particle abrasion destabilize the bonding between the IEC veneer and resin luting agent.

For the IEC specimens, the CPB+ACT groups showed significantly higher bond strength at 0 and 20,000 thermocycles. It is known that silane plays a prominent role in bonding with SiO\textsubscript{2} through a siloxane network. In addition, the phosphate functional monomer (MDP) contained in CPB can promote formation of siloxane bonds\textsuperscript{31}. Moreover, the initiators contained in CPB have a positive effect on the bond between lithium disilicate ceramics and resin luting agents\textsuperscript{32}. Therefore, a concurrent use of the phosphate functional monomer, silane, and initiators (CPB+ACT groups) yielded durable bonds of resin luting agents to lithium disilicate ceramics (e.max).

Although an in vitro study provides informative results as a relevant preclinical test, the study setup of this in vitro study did not reflect the actual clinical conditions. In addition, only one CAD/CAM composite resin was assessed in the current study, it would be interesting to compare the results with different CAD/CAM composite resin materials from other manufacturers. Thus, further laboratory studies and clinical trials should be conducted in advance of clinical use. Within the limitations of the current study, it can be concluded that airborne-particle abrasion is necessary for resin bonding to Avencia blocks. Silane and phosphate functional monomers (MDP) could not play a positive role in the resin bonding of Avencia blocks to zirconia frameworks. A concurrent use of the phosphate functional monomer, silane, and initiators...
yielded durable bonds of lithium disilicate ceramics to zirconia material using a resin luting agent.

ACKNOWLEDGMENTS
This study was supported in part by JSPS KAKENHI Grant Number 19K10217 and a Grant from the Sato Fund, Nihon University School of Dentistry (2019 and 2020).

CONFLICTS OF INTEREST
No potential conflict of interest was reported by the authors.

REFERENCES
1) Pjetursson BE, Valente NA, Strasding M, Zwahlen M, Liu S, Sailer I. A systematic review of the survival and complication rates of zirconia-ceramic and metal-ceramic single crowns. Clin Oral Implants Res 2018; 29: 199-214.
2) Spies BC, Balmer M, Jung RE, Sailer I, Vach K, Kohal RJ. All-ceramic single crowns supported by zirconia implants: 5-year results of a prospective multicenter study. Clin Oral Implants Res 2019; 30: 466-475.
3) Kurbad A. Digital veneering-manufacturing computer-generated veneers with the CAD-on technique. Int J Comput Dent 2011; 14: 235-271.
4) Wiedhahn K. The impression-free Cerec multilayer bridge with the CAD-on method. Int J Comput Dent 2011; 14: 33-45.
5) Schmitter M, Mueller D, Rues S. Chipping behaviour of all-ceramic crowns with zirconia framework and CAD/CAM manufactured veneer. J Dent 2012; 40: 154-162.
6) Saedi Pour R, Edelhoff D, Rafael CF, Prandtner O, Frei S, Maziero Volpato CA, et al. Combining esthetic layering and lithium disilicate sintering technique on zirconia frameworks: a veneering option to prevent ceramic chipping. Int J Periodontics Restorative Dent 2017; 37: 561-569.
7) Holand W, Schweiger M, Watzke R, Peschke A, Kappert H. Fracture resistance of dental CAD/CAM restorative materials: part I—procedures and results. Dent Mater 2014; 30: e99-e111.
8) Kern M, Strub JR, Lu XY. Wear of composite resin veneering materials in a dual-axis chewing simulator. J Oral Rehabil 1999; 26: 372-378.
9) Quinn GD, Giuseppetti AA, Hoffman KH. Chipping fracture resistance of dental CAD/CAM restorative materials: part I—procedures and results. Dent Mater 2014; 30: e99-e111.
10) Kern M, Strub JR, Lu XY. Wear of composite resin veneering materials in a dual-axis chewing simulator. J Oral Rehabil 1999; 26: 372-378.
11) Lehmann F, Spiegl K, Eickemeyer G, Rammelsberg P. Adhesively luted, metal-free composite crowns after five years. J Adhes Dent 2009; 11: 493-498.
12) Vanoorbeek S, Vandamme K, Lijnen I, Naert I. Computer-aided designed/computer-assisted manufactured composite resin versus ceramic single-tooth restorations: a 3-year clinical study. Int J Prosthodont 2010; 23: 223-230.
13) Lehmann F, Spiegl K, Eickemeyer G, Rammelsberg P. Adhesively luted, metal-free composite crowns after five years. J Adhes Dent 2009; 11: 493-498.
14) Vanoorbeek S, Vandamme K, Lijnen I, Naert I. Computer-aided designed/computer-assisted manufactured composite resin versus ceramic single-tooth restorations: a 3-year clinical study. Int J Prosthodont 2010; 23: 223-230.