Abstract: This study investigated bond strength of CAD/CAM-manufactured composite resin and ceramic veneers to a zirconia framework and analyzed the effect of treatments of veneer surfaces. A CAD/CAM resin-based (AVE) composite or lithium disilicate ceramic (IEC) block was used as the veneer material. AVE and IEC specimens were assigned to receive one of three surface treatments (n = 22): no surface treatment, acid-etching with 9.5% hydrofluoric acid gel, and airborne-particle abrasion with alumina particles. Zirconia disks and AVE or IEC specimens in each group were bonded with a resin-based luting agent, and shear bond strength of the specimens was measured at 0 and 20,000 thermocycles. Significant differences were assessed by the Steel-Dwass test for multiple comparisons and Mann-Whitney U-test (α = 0.05). As compared with other surface treatments, bond strengths were significantly higher at 0 and 20,000 thermocycles in the airborne-particle abraded AVE and acid-etched IEC specimens. Airborne-particle abrasion of the surface of AVE specimens increased bond strength between AVE veneers and zirconia frameworks, while hydrofluoric acid treatment enhanced bond strength between IEC veneers and zirconia frameworks.

Keywords: bond strength; CAD/CAM; ceramics; composite resin; zirconia.

Introduction

Zirconium dioxide (zirconia) ceramics are widely used in frameworks for anterior and posterior tooth- and implant-supported prostheses. Previous studies reported that a major drawback of zirconia-based prostheses was minor fracture (chipping) of layering porcelain (1,2). Fortunately, several approaches have been introduced to address this limitation. These include layering a composite material on the zirconia framework (3), application of a monolithic structure for zirconia-based prostheses, and use of techniques in which a ceramic veneer manufactured with a CAD/CAM system is fused with specific glass ceramics or bonded with a resin-based luting agent onto the zirconia framework (4,5).

Several clinical studies found that monolithic zirconia restorations had high survival rates (>99%) and little or no chipping during short- and medium-term observation periods (6,7). Other advantages of monolithic zirconia restorations include the simple processing methods required, less-invasive preparation, and low production cost (8). However, the color of definitive restorations is limited by the low translucency of zirconia. Moreover, the chemical stability of the material and clinical longevity of monolithic zirconia restorations are not well understood.

Recent studies described a new CAD/CAM technique in which two segments of zirconia framework and veneer are manufactured with a CAD/CAM system and fused with fusion glass ceramics (i.e., CAD-on technique) or a resin-based luting agent (i.e., rapid layer technique) (4,5,9). These techniques have several advantages,
including less thermal stress attributable to porcelain firing and the stable mechanical properties of veneers manufactured with CAD/CAM systems. Indeed, when using a resin-based luting agent, no porcelain firing process is involved in bonding the two segments. Resin-based composite materials are an alternative to layering porcelain and have been used with zirconia-based restorations (3,10). However, long-term clinical use of resin-based composite materials is associated with inadequate restoration toughness and strength (11). In contrast, resin-based composite materials have highly favorable esthetic properties, absorb shocks from occlusal forces, are more repairable, and result in less antagonist wear (11). Evidence from some in vitro studies suggests that resin-based composite materials are suitable as layering materials in zirconia-based restorations (3,10). Recently, use of resin-based composite CAD/CAM blocks has increased because of the capabilities of the materials. A study of fracture resistance in implant-supported zirconia-based prostheses adhesively bonded with CAD/CAM-manufactured resin-based composite veneers concluded that this innovative procedure was a promising alternative as a layering method (12).

This study therefore investigated the bond strength of CAD/CAM-manufactured composite resin and ceramic veneers to a zirconia framework and the effects of different treatments for the veneer surfaces. The null hypotheses tested were that (1) there would be no difference in bond strength among the surface treatments, (2) thermocycling would not affect bond strength, and (3) bond strength would not differ between the veneer materials and zirconia.

### Materials and Methods

#### Specimen preparation

A zirconia framework material (Katana Zirconia HT) for CAD/CAM-manufactured restorations was used as the bonding substrate (Table 1). A CAD/CAM resin-based composite (AVE; Katana Avencia Block) or lithium disilicate ceramic (IEC; IPS e.max CAD) block was used as the veneer material (Table 1).

In total, 132 zirconia disks (11.0 mm in diameter ¥ 2.5 mm in thickness) were manufactured with the CAD/CAM system (Fig. 1A). The surface of the zirconia disks was polished with 600-grit silicon carbide paper (Tri-M-ite Wetordry sheets, 3M, St. Paul, MN, USA) and airborne-particle abraded with 50-µm alumina particles (Hi-Aluminas, Shofu, Kyoto, Japan), as shown in Figs 1B and 1D. To define the bonding area, a single-sided

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**Table 1 Materials assessed in the present study**

| Material/Trade name         | Abbreviation | Component                                      | Manufacturer                  |
|-----------------------------|--------------|------------------------------------------------|-------------------------------|
| Zirconia ceramics          | ZR           | ZrO$_2$: 94.4 wt%, Y$_2$O$_3$: 5.4 wt%          | Kuraray Noritake Dental, Tokyo, Japan |
| Katana Zirconia HT          |              |                                                |                               |
| CAD/CAM resin block         | AVE          | Al$_2$O$_3$ (20 nm), SiO$_2$ (40 nm): 62 mass%, UDMA, TEGDMA | Kuraray Noritake Dental |
| Katana Avencia Block 12/A2 LT |              |                                                |                               |
| Lithium disilicate glass-ceramics | IEC       | SiO$_2$: 57-80 wt%, Li$_2$O: 11-19 wt%, K$_2$O: 0-13 wt%, P$_2$O$_5$: 0-11 wt%, ZrO$_2$: 0-8 wt%, ZnO: 0-8 wt% | Ivoclar Vivadent, Schaan, Liechtenstein |
| IPS e.max CAD A2 C14        |              |                                                |                               |
| Hydrofluoric acid           | HF           | 9.5% hydrofluoric acid                         | Ultradent Products, South Jordan, UT, USA |
| Porcelain Etch              |              |                                                |                               |
| Resin-based luting agent     | PV5          | A paste: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, fluoroaluminosilicate glass filler, colloidal silica, accelerator, initiator | Kuraray Noritake Dental |
| Panavia V5                  |              |                                                |                               |
| Priming agent               | CPB          | Catalyst: MDP, HEMA, Bis-GMA                   | Kuraray Noritake Dental |
| Clearfil Photo Bond         |              | Universal: initiators, accelerators, ethanol   |                               |

UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; Bis-GMA: bisphenol-A-diglycidyl methacrylate; MDP: 10-methacryloxydecyldihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate.
tape (Star Traper-WD type P, Sakurai, Tokyo, Japan) with a circular 5.0-mm hole was taped on the zirconia specimens. The bonding area of the specimens was primed with Clearfil Photo Bond (CPB) (Table 1 and Fig. 1B).

For preparation of AVE and IEC specimens, the blocks were cut with a precision saw (IsoMet Low Speed Saw, Buehler, Lake Bluff, IL, USA) and precision cutting device (IsoMet Diamond Wafering Blades, Buehler) to a thickness of 2.5 mm and a diameter of 8.0 mm with diamond burs (Bur No. 106RD, Shofu), using a diamond rotary instrument (Presto Aqua, Nakanishi, Kanuma, Japan) under application of water spray (Fig. 1C). A total of 132 disk specimens were manufactured for veneers. After manufacture of the disks, the IEC specimens were sintered in a porcelain furnace (Cerafusion Press, Morita, Suita, Japan). The surfaces of the AVE and IEC specimens were polished with 600-grit silicon carbide paper and then divided into three surface treatments \( n = 22 \), namely, no surface treatment (CON); acid etching with 9.5% hydrofluoric acid gel (Porcelain Etch), rinsing in distilled water for 20 s, and cleaning in methanol in an ultrasonic bath (Ultrasonic cleaner UT-106, Sharp, Osaka, Japan) (HF); or airborne-particle abrasion (AB) (Table 1 and Fig. 1D).

The zirconia disks and AVE or IEC specimens in each group were bonded by using a dual-curing resin-based luting agent (Panavia V5) under a load of 5 N, without priming (Table 1). The specimens were light-cured from four directions for 10 s in each direction with a quartz-tungsten halogen curing unit (Optilux 501, Kerr, Orange, CA, USA) (Fig. 1E).

Shear bond test
Half the specimens \( n = 11 \) were stored in distilled water at 37°C for 24 h; the other specimens were subjected to thermocycling with a thermocycling device (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co, Ltd., Tokyo, Japan) at the following conditions: 20,000 cycles between 5°C and 55°C and a dwell time of 1 min (Fig. 1F and 1G). The specimens were placed on a shear testing jig (Tokyo Giken, Tokyo, Japan). Measurement of shear bond strength was performed with a mechanical testing machine (Type 5567, Instron, Canton, MA, USA) at a crosshead speed of 0.5 mm/min (13) (Fig. 1H).

Statistical analysis
Data were first analyzed with the Kolmogorov-Smirnov test and Levene test (IBM SPSS 24.0, IBM, Armonk, NY, USA). When the Levene test did not show homoscedasticity, the Kruskal-Wallis test and post-hoc Steel-Dwass test were used to analyze shear bond strength values for each veneering material. The Mann-Whitney U-test was used for comparisons of values at 0 and 20,000 thermocycles and those in the AVE and IEC groups. Statistical significance was defined as \( \alpha = 0.05 \).

Observation of fracture mode
Fracture mode of the debonded specimens was observed by using a previously described method (13) and classified as adhesive failure at the veneer material/resin-based luting agent interface (Av), adhesive failure at the resin-based luting agent/zirconia interface (Az), combined adhesive/cohesive failure within the resin-based luting agent (AC), and cohesive failure within the resin-based luting agent (C).
Debonded specimens were observed with a scanning electron microscope (SEM) and underwent X-ray diffraction (XRD) analysis, as described previously (13). In addition, the cross-sectional surfaces of representative specimens were inspected by using SEM.

**Results**

Table 2 shows the results of shear bond strength testing and statistical analysis. For AVE specimens, bond strength was significantly higher in the AB group than in the other two groups at 0 and 20,000 thermocycles. For IEC specimens, bond strength was significantly higher in the HF group than in the CON and AB groups at 0 and 20,000 thermocycles. In the HF group, shear bond strength was higher for IEC specimens than for AVE specimens at 0 and 20,000 thermocycles. Although pre-thermocycling bond strength in the AB group did not significantly differ between AVE and IEC specimens ($P = 0.151$), bond strength

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

|             | AVE |               | IEC |               |
|-------------|-----|---------------|-----|---------------|
|             | Median (IQR) | Category | Median (IQR) | Category | $P$-value |
| 0 thermocycles |     |             |     |             |           |
| CON         | 9.5 (6.9; 10.5) a |             | 13.8 (10.0; 16.9) c |             | 0.133     |
| HF          | 10.6 (8.1; 14.3) a |             | 34.0 (29.3; 38.8) e |             | <0.001**  |
| AB          | 14.2 (12.7; 18.8) b |             | 22.6 (18.8; 29.1) d |             | 0.151     |
| 20,000 thermocycles |     |             |     |             |           |
| CON         | 0.7 (0.6; 1.0) f  |             | 0.8 (0.6; 1.0) i  |             | 1.000     |
| HF          | 4.6 (3.7; 9.3) g  |             | 13.8 (12.4; 17.6) j |             | <0.001**  |
| AB          | 11.0 (8.4; 13.7) h |             | 0.9 (0.7; 1.0) i  |             | <0.001**  |

* Significant difference in shear bond strength among surface treatments (Steel-Dwass test; $P < 0.05$).
** Significant difference between two veneer materials (Mann-Whitney $U$-test; $P < 0.05$).
IQR: interquartile range.
See Table 1 and text for descriptions of abbreviations.

### Table 3 Reduction in shear bond strength at 20,000 thermocycles

| Group | $P$-value | Reduction |
|-------|-----------|-----------|
| AVE   |            |           |
| CON   | <0.001*   | 91.0%     |
| HF    | 0.007*    | 34.0%     |
| AB    | 0.023*    | 28.5%     |
| IEC   |            |           |
| CON   | <0.001*   | 93.2%     |
| HF    | <0.001*   | 58.8%     |
| AB    | <0.001*   | 94.9%     |

* Significant difference between 0 and 20,000 thermocycles (Mann-Whitney $U$-test; $P < 0.05$).
See Table 1 and text for descriptions of abbreviations.

![SEM images](image)

**Fig. 2** SEM images of surface of veneer materials after surface treatment (original magnification ×1,000). A, B, and C: AVE specimens (A: CON, B: HF, and C: AB groups); D, E, and F: IEC specimens (D: CON, E: HF, and F: AB groups). AVE: Katana Avencia Block; IEC: IPS e.max CAD; CON: wet-grinding; HF: acid-etching; AB: airborne-particle abrasion.
strength after thermocycling was significantly higher for AVE specimens than for IEC specimens \((P < 0.001)\). In AVE and IEC specimens, bond strength in all groups was significantly lower after 20,000 thermocycles (Table 3).

The failure modes of the tested specimens are summarized in Table 4. No specimen developed adhesive failure at the resin-based luting agent/zirconia interface. However, adhesive failure at the veneer material/resin-based luting agent interface was noted in almost all AVE and IEC specimens after thermocycling.

In AVE specimens, SEM images collected after grinding showed scratches (Fig. 2A). SEM images of HF group specimens (Fig. 2B) showed that surfaces were smoother than those in AB group (Fig. 2C). SEM images of IEC specimens revealed no imperfections after grinding (Fig. 2D). In IEC specimens, the surface was rougher after HF treatment (Fig. 2E) than after AB treatment (Fig. 2F). In the HF group, the surface was smoother for AVE specimens (Fig. 2B) than for IEC specimens, which exhibited a characteristic honeycomb-like structure (Fig. 2E). SEM observation of AB group indicated that the surface of AVE specimens (Fig. 2C) exhibited greater irregularity and sharper edges and undercuts than that of IEC specimens (Fig. 2F).

Figure 3 shows representative SEM images of the surfaces of debonded specimens. SEM images of adhesive failure at the veneer material/resin-based luting agent interface of AVE specimens (Fig. 2A) and IEC specimens (Fig. 2B) were similar to the images for each substrate treatment. SEM images of combined adhesive/cohesive failure within the resin-based luting agent for AVE specimens (Fig. 2C) and IEC specimens (Fig. 2D) showed both the veneer material and residual resin-based luting material.

Figures 4A and 4B show cross-sectional surfaces of airborne-particle abraded AVE and IEC specimens, respectively. No surface defects were observed in the AVE-AB specimen (Fig. 4A), but microcracks were present in the IEC-AB specimen (Fig. 4B).

Figures 5A and 6A show the XRD pattern of the
resin-based luting agent, which exhibited a quartz peak ($2\theta = 21.8^\circ$, 24.2$^\circ$, and 27.0$^\circ$). The XRD pattern of an AVE specimen after grinding exhibited a quartz peak ($2\theta = 21.5^\circ$, 23.9$^\circ$, and 26.7$^\circ$) and alumina peak ($2\theta = 37.6^\circ$, 44.6$^\circ$, and 77.9$^\circ$) (Fig. 5B). The XRD pattern for combined adhesive/cohesive failure (Fig. 5C) conformed to those for the resin-based luting agent (Fig. 5A) and ground AVE specimen (Fig. 5B). The XRD pattern for an IEC specimen after grinding exhibited peaks for quartz ($2\theta = 21.4^\circ$ and 24.4$^\circ$) and lithium silicon oxide ($2\theta = 24.9^\circ$, 38.1$^\circ$, and 61.0$^\circ$) (Fig. 6B). Combined adhesive/cohesive failure within the resin-based luting agent (Fig. 6C) coincided with an XRD pattern showing resin-based luting agent (Fig. 6A) and the ground IEC specimen (Fig. 6B).

**Discussion**

This study investigated the bond strength of CAD/CAM-manufactured composite resin and ceramic veneers with a zirconia framework. Bond strength was significantly higher in the AB group of AVE specimens and in the HF group of IEC specimens. Hence, the first null hypothesis—that bond strength would not differ in relation to surface treatment—was rejected. In AVE and IEC specimens, bond strength was significantly lower after 20,000 thermocycles for all surface treatments. Therefore, the second null hypothesis—that thermocycling would not affect bond strength—was also disproved. The third null hypothesis, too, was rejected, since bond strength significantly differed between AVE and IEC specimens for each surface treatment.

Airborne-particle abrasion cleans the substrate surface and increases the roughness of bonding surfaces. It also enhances the resin bond to indirect composite resins (14,15). At 0 and 20,000 thermocycles, the AB group of AVE specimens had the highest bond strength among the tested groups. This result is consistent with those of past studies, which found that airborne-particle abrasion had a greater positive effect than hydrofluoric acid treatment on resin bonding to indirect composite resins (16). AVE blocks mainly consist of alumina and silica fillers (Table 1). Previous studies found that HF treatment was ineffective for bonding to alumina fillers (17,18), which explains why hydrofluoric acid treatment yielded a microretentive surface in AVE specimens and resulted in lower and less stable bond strengths. The present results thus suggest that airborne-particle abrasion to AVE specimens results in superior bond strengths between AVE veneers and a zirconia framework.

Among IEC specimens, bond strength was significantly
higher for the HF group. Hydrofluoric acid etching is the first choice for achieving micromechanical interlocking of silica-based ceramics to resin bond. Hydrofluoric acid selectively dissolves the glass matrix, exposes the crystalline structures, and roughens ceramic surfaces (18-20). Several studies reported that hydrofluoric acid treatment provided strong, durable resin bonding to a lithium disilicate ceramic material (16,21,22), which supports the present findings. Thus, hydrofluoric acid treatment is recommended for enhancing bond strength of IEC veneers to zirconia frameworks.

The bond strength of AB specimens in IEC group was markedly lower (0.9 MPa) after thermocycling. In addition, although pre-thermocycling bond strengths of AB specimens did not significantly differ between the AVE and IEC groups, post-thermocycling bond strength was lower in the IEC group, perhaps because of microcrack propagation from airborne-particle abraded surfaces of IEC specimens (23). An SEM image of the cross-section of an IEC specimen (Fig. 4B) revealed microcracks originating on the specimen surface and therefore supports this hypothesis. Airborne-particle abrasion leads to microcracks at the ceramic surface, lowers bond strength, and is more likely to result in premature failure (24). In addition, microcracks caused by airborne-particle abrasion might propagate along fillers along with repetitive expansion and contraction during thermocycling (25). Thus, the bond between IEC veneers and zirconia frameworks was not durable in this study.

This study had limitations that should be mentioned. The effect of chemical bonding, such as priming or bonding, should be investigated in future studies. Furthermore, clinical studies are necessary to validate the clinical effectiveness of restorations in which CAD/CAM-manufactured composite resin or ceramic veneers are adhesively bonded to a zirconia framework.

Under the conditions of this in vitro study, airborne-particle abrasion of the surface of AVE specimens achieved superior bond strengths between AVE veneers and zirconia frameworks but did not result in a durable bond between IEC veneers and zirconia frameworks. Hydrofluoric acid treatment likely enhances bond strengths between IEC veneers and zirconia frameworks.

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**Conflict of interest**

The authors declare no conflict of interest.

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