Shear bond strength between an indirect composite veneering material and zirconia ceramics after thermocycling

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Abstract: The present study evaluated the shear bond strength between an indirect composite material and zirconium dioxide (zirconia) ceramics after thermocycling. A total of 80 zirconia (Katana) discs were divided into five groups and primed with one of the following agents: All Bond 2 Primer B (ABB), Alloy Primer (ALP), AZ Primer (AZP), Estenia Opaque Primer (EOP), and Porcelain Liner M Liquid A (PLA). An indirect composite material (Estenia C&B) was then bonded to the primed zirconia. One-half of the specimens (n = 8) in each group were stored in distilled water at 37°C for 24 h, and the remaining eight specimens were thermocycled 5,000 times before shear bond strength testing. Mean bond strengths before thermocycling varied from 10.1 to 15.6 MPa; bond strengths after thermocycling ranged from 4.3 to 17.6 MPa. The ALP group had the highest strengths after thermocycling; there were no significant differences among the PLA, AZP, and EOP groups. The bond strength values for PLA, AZP, EOP, and ALP did not decrease with thermocycling. The application of an acidic functional monomer containing carboxylic anhydride (4-META), phosphonic acid (6-MHPA), or phosphate monomer (MDP) provided durable bond strength between Estenia C&B indirect composite and Katana zirconia. (J Oral Sci 51, 629-634, 2009)

Keywords: bond strength; functional monomer; indirect composite; thermocycling; zirconia.

Introduction

Esthetic restorative materials for crown and fixed partial denture (FPD) are changing from porcelain-fused-to-metal (PFM) restorations to all-ceramic systems. The superior mechanical properties of zirconium dioxide (zirconia) ceramics combined with CAD/CAM fabrication procedures allow for the production of large and complex restorations as a superstructure for tooth abutments and dental implants. Clinical studies have revealed that zirconia ceramics exhibit high stability as a framework material (1-6). Indeed, a success rate exceeding 97.8% has been reported for the zirconia framework (1-6). These studies also reported that fracture of the veneering porcelain was the most frequent reason for failure of zirconia-based restorations (1-6).

Although feldspathic porcelain is acceptable for use in a veneering zirconia framework, a highly loaded indirect composite resin is an alternative to this material. Çifçi and Canay (7) evaluated the effects of various veneering materials on stress distribution in implant-supported fixed partial dentures and found that the reduction in stress associated with the use of the composite resin material was 15% greater than that of porcelain or gold alloy. In a study of fracture resistance, there were no significant differences between the probability of failure between metal-ceramic
and highly filled resin-veneered implant-supported restorations (8).

Failure of a layered structure usually occurs in the weakest material or in the weakest interface of the unit. Kobayashi et al. (9) used acidic priming agents to evaluate the short-term bond strength between an indirect composite material and a zirconia ceramic material. They observed that the indirect composite material could be bonded to zirconia by application of either a hydrophobic phosphate monomer or a combination of silane and phosphate. However, there are no data on the durability of bonds between indirect composite materials and zirconia ceramics. The aim of the present study, therefore, was to evaluate the shear bond strength between an indirect composite material and zirconia ceramics after thermocycling.

**Materials and Methods**

The materials used in the present study are listed in Table 1. The 80 zirconia specimens to be used as a bonding substrate were made of zirconia ceramic material using CAD/CAM technology (Katana, Noritake Dental Supply Co. Ltd., Miyoshi, Aichi, Japan). The specimens were fabricated in the form of a disc that was 11.0 mm in diameter and 2.5 mm in thickness. Five commercial priming agents were evaluated as bonding promoters: All Bond 2 Primer B (ABB), Alloy Primer (ALP), AZ Primer (AZP), Estenia Opaque Primer (EOP), and Porcelain Liner M Liquid A (PLA) (Table 1). All priming agents contained at least one adhesive functional monomer. An indirect composite material (Estenia C&B Dentin DA2, Kuraray Medical Inc., Tokyo, Japan) was used as a veneering material for the zirconia ceramics, and an opaque material (Estenia C&B Body Opaque OA2, Kuraray Medical Inc.) was utilized as a high-flow bonding agent. The surface of all specimens was wet-ground with 600-grit silicon carbide abrasive paper and cleaned with acetone for 10 min in an ultrasonic bath (SUC-110, Shofu Inc., Kyoto, Japan). The surface of specimens was then airborne-particle abraded for 20 s using 50-µm-grain-sized alumina (Hi-Aluminas, Shofu Inc.) at a pressure of 0.2 MPa from a distance of 10 mm. The specimens were then ultrasonically cleaned in an acetone bath for 10 min.

The specimens were divided at random into five groups \((n = 16)\) and assigned one of the following priming agents: ABB, ALP, AZP, EOP, and PLA. A piece of double-stick tape with a circular hole 5.0 mm in diameter was positioned on the surface of zirconia discs to define the bonding area. The surface of the specimens was then treated with one of the five priming agents according to the manufacturer’s instructions. For all of the specimens in each group, a thin layer of opaque material was applied and light cured for 90 s using a laboratory light-polymerization unit (α-Light II, J. Morita Corp., Sita, Japan). A brass ring (6.0 mm in inner diameter and 2.0 mm in height) was placed on the treated surface of all specimens. The indirect composite material was packed into the ring with a force of 5 N. The specimen was then light cured with the polymerization unit for 5 min, and finally heat-polymerized at 110°C for 15 min in a heat oven (KL-100, J. Morita Corp.).

Half of the specimens \((n = 8)\) in each group were stored in distilled water at 37°C for 24 h, and then shear bond testing was performed. Before shear bond testing, specimens were embedded in an acrylic resin (Unifast III, GC Corp., Tokyo, Japan) mould and mounted in the jig (ISO TR 11405, Tokyo Giken Inc., Tokyo, Japan) of a mechanical testing machine (Type 5567, Instron Corp., Canton, MA, USA). The shear force was applied to the
adhesive interface until fracture occurred. The specimens were loaded at a crosshead speed of 0.5 mm min$^{-1}$. The remaining eight specimens from each group were subjected to thermocycling for 5,000 cycles between 5°C and 55°C with a one-minute dwell time using a thermocycling apparatus (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co. Ltd., Tokyo, Japan). All specimens that did not debond during the thermocycling were subjected to shear bond testing.

For multiple group comparisons, homogeneity of variance was primarily analyzed by Levene’s test (SPSS ver. 15.0, SPSS Inc., Chicago, IL, USA). When homoscedasticity was not shown by Levene’s test, the Steel – Dwass test (Kyplot 5.0, KyensLab Inc., Tokyo, Japan) was performed. The level of statistical significance was set at 5%.

After the shear bond testing, the fractured interfaces were observed with a stereomicroscope (Stemi DV4, Carl Zeiss Co., Ltd., Jena, Germany) at original magnification ×32 to determine the mode of failure. Mode of failure was classified as A – adhesive failure between the indirect composite material and zirconia, C – cohesive failure within the indirect composite material, and AC – combined adhesive and cohesive failure. Selected typical specimens were osmium-coated with a sputter coater (HPC-IS, Vacuum Device, Mito, Japan) for 30 s and examined using a scanning electron microscope (S-4300, Hitachi High-Technologies Co. Ltd., Tokyo, Japan) operated at 15 kV.

The surfaces of airborne-particle abraded zirconia, the indirect composite material, and the zirconia after shear bond testing were analyzed using X-ray diffraction (XRD) with Cu Kα radiation. The specimens were placed in the holder of an X-ray diffractometer (MiniFlex II, Rigaku Corp., Tokyo, Japan) and, using flat plate geometry, data were collected at 30 kV (15 mA) with a scanning range of 20° to 30°.

**Results**

The shear bond strength values for the five priming agent groups and the two different storage conditions (0 and 5,000 thermocycles) are summarized in Table 2. For the groups without thermocycling, mean bond strength ranged from 10.1 MPa for the ABB group to 15.6 MPa for the ALP group. Significant differences were not observed among the ABB, PLA, and ALP groups (category a). The ABB group had a significantly lower bond strength than the other four groups (category b). After 5,000 thermocycles, the ABB group had the lowest mean shear bond strength (4.3 MPa), which was significantly lower than that of the other groups (category c). The highest values were observed in the ALP group (17.6 MPa); there were no significant differences among the PLA, AZP, and EOP groups (category c). The bond strength values for PLA, AZP, EOP, and ALP did not decrease with thermocycling.

Table 3 shows the modes of failure after shear bond testing. Most of the debonded specimens failed at the indirect composite material-zirconia interface regardless of thermal cycling. Cohesive failure within the indirect composite material was not observed in any of the specimens in the present study.

Figures 1 and 2 show SEM images of the zirconia ceramic surface before and after airborne-particle abrasion, respectively. Observation of the surface topography of zirconia ground with 600-grit silicon carbide abrasive paper reveals a smooth surface with small scars; no microretentive pattern can be seen (Fig. 1). As illustrated in Fig. 2, airborne-particle abrasion altered the surface of zirconia ceramics and created sharp edges and undercuts. Figures 3 and 4 show typical SEM fractographs after shear bond testing. For the specimens treated with AZP and 0 thermocycles, the small number of filler particles retained on the zirconia surface suggested failure primarily of the adhesive bond (Fig. 3). An SEM image of the fracture interface for an ALP group specimen treated with 5,000 thermocycles shows combined adhesive and cohesive failure (Fig. 4).

The XRD patterns of selected specimens are presented in Fig. 5. Figure 5A shows a representative XRD pattern.
Table 3  Modes of failure after shear bond testing

<table>
<thead>
<tr>
<th>Groups</th>
<th>0 thermocycles</th>
<th>5,000 thermocycles</th>
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<td>A</td>
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<td>ALP</td>
<td>6</td>
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See Table 1 and text for description of group abbreviations.
(A) Adhesive failure at zirconia interface; (AC) Combination of cohesive and adhesive failure; (C) Cohesive failure within the indirect composite material.

Fig. 1  SEM image (original magnification ×2,000) of a zirconia ceramic surface ground with 600-grit silicon carbide abrasive paper.

Fig. 2  SEM image (original magnification ×2,000) of a zirconia ceramic surface airborne-particle abraded with 50-µm alumina particles.

Fig. 3  SEM image (original magnification ×2,000) of a zirconia ceramic surface of a specimen from the AZP group after shear bond testing without thermocycling.

Fig. 4  SEM image (original magnification ×2,000) of a zirconia ceramic surface of a specimen from the ALP group after shear bond testing and 5,000 thermocycles.
for zirconia ceramics after airborne-particle abrasion. An XRD pattern for adhesive failure after shear bond testing is illustrated in Fig. 5B. This pattern is almost identical to that shown in Fig. 5A. Figure 5C illustrates a typical XRD pattern for combined adhesive and cohesive failure; the diffraction peak of SiO$_2$ ($2\theta = 26.5^\circ$) can be seen. Figure 5D shows that the peak of SiO$_2$ was detected in the XRD pattern of the indirect composite material.

Discussion

In the present study, the strength of the bond between an indirect composite material and zirconia ceramics before and after exposure to thermocycling was measured, in order to assess the clinical durability of composite-veneered restorations with a zirconia framework.

The results of the present study show that the bond strength of the indirect composite material to the zirconia ceramics was influenced by both the type of priming agent and the application of thermocycling. Although the ABB group had the lowest bond strength without thermocycling, there were no significant differences between the PLA and AZP groups. In addition, after 5,000 thermocycles, mean bond strength in the ABB group decreased by 57.7% and was significantly lower (4.3 MPa) than that of the other four groups. These results indicate that application of All Bond 2 Primer B containing BPDM to the zirconia ceramics does not result in a stable bond of the Estenia composite to the Katana zirconia, after exposure to thermocycling. Moreover, after 5,000 thermocycles, there was a significant difference between the ABB and PLA groups, which contain different carboxylic monomers. It can be presumed from these findings that the All Bond 2 Primer B, which contains aromatic univalent carboxylic acid (BPDM), is ineffective for durable bonding between the composite material and zirconia ceramics, as compared with the Porcelain Liner M Liquid A, which contains trimellitate anhydride (4-META).

The bond strength between the composite material and zirconia ceramics increased slightly with thermocycling in all groups except the ABB group, which indicates that the application of Porcelain Liner M Liquid A containing carboxylic anhydride (4-META), AZ Primer containing phosphonic acid (6-MHPA), and Estenia Opaque Primer or Alloy Primer containing phosphate monomer (MDP) can achieve stable shear bond strength after thermocycling. Furthermore, it should be pointed out that polymerization of the Estenia composite material was still progressing at 24 h and that thermal stress at 55°C resulted in additional polymerization of the composite material.

There was no significant difference in bond strength between the ALP and EOP groups, regardless of thermocycling. One interpretation of this finding is that the

![Fig. 5 XRD patterns of (A) a zirconia ceramic surface after airborne-particle abrasion with 50-μm alumina particles, (B) a zirconia ceramic surface of an AZP group specimen after shear bond testing without thermocycling, (C) zirconia ceramic surface of an ALP group specimen after shear bond testing and 5,000 thermocycles, and (D) indirect composite material.](image)
thione compound (VBATDT), which was originally designed for conditioning noble metal alloys, did not interfere with the effectiveness of the MDP monomer with respect to durable bonding between the Estenia composite and the Katana zirconia (9,10).

Thermocycling can be very useful in simulating intraoral conditions and stressing the bonding interface (11). As shown in Table 3, the mode of failure was predominantly adhesive failure at the zirconia ceramic surface, regardless of thermocycling. A SEM image of a zirconia ceramic surface that had experienced adhesive failure revealed the presence of a small number of residual filler particles on the zirconia (Fig. 3). However, XRD analysis of the zirconia-composite interface revealed that composite elements, such as silica, could not diffuse into the zirconia surface. On the other hand, XRD analysis showed that specimens that had experienced combined adhesive and cohesive failure did have typical composite elements, such as silica. These results show that XRD analysis can be a useful supplement to optical and SEM observations, and that it can be effective in the examination of debonded surfaces.

In conclusion, the present study demonstrated that the use of an acidic functional monomer containing carboxylic anhydride (4-META), phosphonic acid (6-MHPA), or phosphate monomer (MDP) can yield durable bond strength between Estenia C&B indirect composite and Katana zirconia.

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References