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ORIGINAL ARTICLE

Durability of bond between an indirect composite veneering material and zirconium dioxide ceramics

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Abstract

Objective. The purpose of the present study was to evaluate the durability of bond strength between an indirect composite material and zirconia ceramics after thermocycling (100 000 cycles) and to assess the effect of various priming agents for zirconia surface treatments. ***Materials and methods.*** A CAD/CAM system (Katana, Noritake Dental Supply) was used to fabricate 96 zirconia disks as a bonding substrate. The specimens were randomly divided into six groups ($n = 16$) and treated with one of the following acidic priming agents: Alloy Primer (ALP, Kuraray), Clearfil Ceramic Primer (CCP, Kuraray), Clearfil Photo Bond (CPB, Kuraray), Clearfil Photo Bond with Clearfil Porcelain Bond Activator (CPB + Activator, Kuraray), Estenia Opaque Primer (EOP, Kuraray) and Porcelain Liner M Liquid A (PLA, Sun Medical). The specimens were bonded with an indirect composite material (Estenia C&B Dentin, Kuraray). Shear bond strengths were tested before and after 100 000 thermocycles and the data were analyzed by using the Steel-Dwass test and Mann-Whitney U-test. ***Results.*** After 100 000 thermocycles, the PLA group showed the lowest bond strength ($p = 0.010$), whereas the CPB + Activator (23.9 MPa; $p < 0.014$) and CPB (22.7 MPa; $p < 0.028$) groups had significantly higher bond strengths than the other groups. The Mann-Whitney U-test revealed that bond strengths did not significantly decrease after thermocycling, except for specimens in the PLA ($p = 0.038$) and CCP ($p = 0.028$) groups. ***Conclusions.*** Application of a combination of hydrophobic phosphate monomer (MDP) and initiator results in a durable long-term bond between Katana zirconia and Estenia C&B composite material.

Key Words: *bond strength, functional monomer, indirect composite, zirconia*

Introduction

Due to their excellent biocompatibility, increased strength and inherent esthetic properties, zirconium dioxide (zirconia) ceramics have been used as a framework material for tooth-supported or implant-supported all-ceramic restorations and implant abutments. A number of clinical studies of tooth-supported zirconia-based all-ceramic restorations have confirmed that zirconia ceramics exhibit high stability as a framework material [1–6]. However, a relatively high rate (6–25%) of chipping and minor fracture of veneering porcelain during a 5-year observation period was reported in several clinical studies of zirconia-based all-ceramic fixed partial dentures (FPDs) [1–4,6]. The zirconia–veneer interface is one of the weakest aspects of these restorations; thus; chipping of the porcelain veneer is a possibility [7]. Different factors can influence veneer chipping, such as differences in thermal expansion coefficients between zirconia and porcelain, firing shrinkage of porcelain, flaws in veneering and poor wetting by veneering on zirconia.

Feldspathic porcelain is processed onto the zirconia framework to fabricate zirconia-based all-ceramic restorations. Numerous studies have demonstrated that the risk of porcelain fracture for an implant-supported metal-ceramic restoration was greater than that for a tooth-supported restoration [8–11]. The main cause of such fractures might be the lack of neurologic feedback and the periodontal reflex mechanism due to the absence a periodontal ligament around the osseointegrated implant. Hämmerle et al. [12] reported that the threshold value for tactile perception for implants is more than 8-fold that of natural teeth.

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The use of layered indirect composite materials in implant-supported zirconia-based fixed restorations can be an intriguing option, as they avoid the porcelain chipping common to zirconia-based all-ceramic restorations. The effect of various veneering materials on stress distribution in implant-supported FPDs was evaluated in a previous study [13]. The results revealed that the reduction in stress associated with the use of the composite material was 15% greater than that of feldspathic porcelain or gold alloy [13]. Furthermore, with regard to fracture resistance, there was no significant difference in the likelihood of failure among metal-ceramic restorations and indirect composite material-veneered implant-supported restorations [14].

Two *in vitro* studies investigated short-term bonding between indirect composite materials and zirconia ceramics in zirconia-based composite-veneered restorations [15,16]. Application of primers containing either the functional phosphate monomer MDP or an MDP-silane combination resulted in superior initial bonds between an indirect composite material and zirconia ceramics [15]. Komine et al. [16] demonstrated that the use of an acid functional monomer containing carboxylic anhydride (4-META), phosphonic acid (6-MHPA) or MDP yielded superior short-term bond strength between an indirect composite material and zirconia ceramics. Durable bonding between veneering materials and frameworks could be an important factor in the successful performance of framework/veneer bilayered restorations. The significant effects of thermocycling on the durability of resin bond to high-strength ceramic materials were confirmed in previous studies [17,18] and thermocycling can thus be used both to simulate intra-oral conditions and stress bonding interfaces. However, there has been very little study of long-term bond strength between an indirect composite as a veneering material and a zirconia ceramic framework. Therefore, the purpose of the present study was to evaluate the durability of bond strength between an indirect composite material and zirconia ceramics after thermocycling (100 000 cycles) and investigate the effect of various priming agents for zirconia surface treatments. The following hypotheses were tested: (1) artificial aging with thermocycling does not influence the bond strength between an indirect composite material and zirconia ceramics and (2) there is no difference in bond strength among various priming agents.

Materials and methods

Specimen preparation

The materials assessed in the present study are presented in Table I. Using a CAD/CAM system (Katana, Noritake Dental Supply Co. Ltd., Miyoshi, Japan), 96 zirconia disks (diameter, 11.0 mm; height, 2.5 mm) were industrially fabricated as a bonding substrate. The surface of all zirconia disks was finished by wet-grinding with 600-grit silicon carbide abrasive paper and cleaned with acetone for 10 min in an ultrasonic bath (SUC-110, Shofu Inc., Kyoto, Japan). Then, the surface of specimens was airborne-

Table I. Materials assessed in the present study.

Material/Trade name	Lot no.	Manufacturer	Components	Abbreviation
Zirconia ceramics		Noritake Dental Supply Co., Ltd.,		
Katana	200218	Miyoshi, Japan	94.4% ZrO_2, 5.4% Y_2O_3	
Indirect composite material				
Estenia C&B		Kuraray Medical Inc., Tokyo, Japan	Dentin DA2, Body Opaque OA2	
Treatment agents				
Alloy Primer	0303AA	Kuraray Medical Inc.	MDP, VTD, Acetone	ALP
Clearfil Ceramic Primer	0005DA	Kuraray Medical Inc.	MDP, γ-MPTS, Ethanol	CCP
Clearfil Photo Bond		Kuraray Medical Inc.		CPB
Catalyst	00414B		MDP, HEMA, Bis-GMA	
Universal	00515C		Initiators, Accelerators, Ethanol	
Cleafil Porcelain Bond Activator	0219AA	Kuraray Medical Inc.	γ-MPTS	Activator
Estenia Opaque Primer	0154AA	Kuraray Medical Inc.	MDP, Monomer solvent	EOP
Porcelain Liner M Liquid A	RW1	Sun Medical Co., Ltd., Moriyama, Japan	4-META, MMA	PLA

MDP, 10-methacryloyloxydecyl dihydrogen phosphate; VTD, 6-(4-vinylbenzyl-*n*-propyl)amino-1,3,5-triazine-2,4-dithione-dithiol tautomer; γ-MPTS, 3-trimethoxysilylpropyl methacrylate; HEMA, 2-hydroxyetheyl methacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; MMA, methyl methacylate.

particle abraded with 50-μm grain size Al_2O_3 particles at a pressure of 0.2 MPa from a distance of 10 mm for 20 s and ultrasonically cleaned in an acetone bath for 10 min.

The specimens were randomly divided into six groups (n = 16) and assigned one of the following priming agents: Alloy Primer (ALP, Kuraray Medical Inc., Tokyo, Japan), Clearfil Ceramic Primer (CCP, Kuraray Medical Inc.), Clearfil Photo Bond (CPB, Kuraray Medical Inc.), Clearfil Photo Bond with Clearfil Porcelain Bond Activator (CPB + Activator, Kuraray Medical Inc.), Estenia Opaque Primer (EOP, Kuraray Medical Inc.), and Porcelain Liner M Liquid A (PLA, Sun Medical Co., Ltd., Moriyama, Japan) (Table I). All agents contained at least one adhesive functional monomer in the solvent. The functional monomers were 6-(4-vinylbenzyl-*n*-propyl)amino-1,3,5-triazine-2,4-dithione, -dithiol tautomer (VTD) for ALP, 4-methacryloyloxyethyl trimellitate anhydride (4-META) for PLA and 10-methacryloyloxydecyl dihydrogen phosphate (MDP) for ALP, CCP, CPB and EOP. Three agents (ALP, EOP and PLA) were developed for priming base metal alloys, two priming agents (CCP and CPB + Activator) are marketed for the treatment of silica-based ceramics and CPB was originally designed for enamel bonding. An indirect composite material (Estenia C&B Dentin DA2, Kuraray Medical Inc.) was used as a veneering material for the zirconia ceramics and an opaque material (Estenia C&B Body Opaque OA2, Kuraray Medical Inc.) was used as a high-flow bonding agent.

A piece of double-stick tape with a circular hole 5.0 mm in diameter was placed on the surface of the specimens to define the bonding area. The surface of the zirconia specimens was treated with one of the six priming agents according to the manufacturer's recommendations. For all specimens in each group, a thin layer of opaque material was applied and polymerized for 90 s using a laboratory light-polymerization unit (α-Light II, J. Morita Corp., Suita, 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 brass ring with a force of 5 N. The specimen was then polymerized with the polymerization vessel for 5 min and finally heat-polymerized at 110°C for 15 min in an oven (KL-310, J. Morita Corp.). Half the specimens (n = 8) in each group were stored in distilled water and kept at 37°C for 24 h. This state was defined as 0 thermocycles. The remaining eight specimens from each group were then placed in a thermocycling machine (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co. Ltd., Tokyo, Japan) for 100 000 cycles at 5°C and 55°C for 60 s each.

Shear bond testing

Before shear bond testing, specimens were embedded in an acrylic resin (Unifast III, GC Corp., Tokyo, Japan) mold and seated in an ISO/TR 11405 shear testing jig. Shear bond strengths were tested with a universal testing device (Type 5567, Instron Corp., Canton, MA) at a crosshead speed of 0.5 mm/min. Shear bond strengths were applied to the adhesive interface until fracture occurred.

Statistics

For multiple group comparisons, homogeneity of variance was primarily analyzed by using the Levene test (SPSS ver. 15.0, SPSS Inc., Chicago, IL). When homoscedasticity was not shown by the Levene test, the Steel-Dwass test (Kyplot 5.0, KyensLab Inc., Tokyo, Japan) was performed. The Mann-Whitney U-test was used for comparisons between groups of specimens with and without thermocycling. The level of statistical significance was set at 5%.

Table II. Shear bond strengths (MPa) of indirect composite material to zirconia ceramics at 0 and 100 000 thermocycles.

	0 thermocycle			100 000 thermocycles				Difference between	
Group	Median	Mean	SD	Median	Mean	SD	Category**	0 and 100 000 thermocycles	*p*-value***
PLA	10.6*	11.9*	2.8*	9.4	9.4	1.6	a	Significant	0.038
EOP	14.1*	14.7*	1.7*	16.8	16.3	2.5	b	Not significant	0.234
ALP	15.3*	15.6*	1.6*	15.7	16.2	2.0	b	Not significant	0.645
CCP	19.7*	19.8*	2.7*	17.2	15.9	2.6	b	Significant	0.028
CPB	21.6*	21.6*	2.9*	23.2	22.7	3.2	c	Not significant	0.505
CPB + Activator	24.1*	24.2*	3.2*	25.1	23.9	3.2	c	Not significant	0.645

Refer to legend of Table I and the main text for definitions of abbreviations.
*Data are quoted from Kobayashi et al. [15].
**Identical letters indicate that values are not significantly different (Steel-Dwass test of multiple groups; $p > 0.05$) of bond strengths at 100 000 thermocycles.
***Statistically significant difference between storage conditions, i.e. 0 and 100 000 thermocycles (Mann-Whitney U-test; $p < 0.05$).

Table III. Failure modes after shear bond testing.

	0 thermocycle			100 000 thermocycles		
Groups	A	CA	C	A	CA	C
PLA	8*	0*	0*	8	0	0
EOP	8*	0*	0*	8	0	0
ALP	6*	2*	0*	8	0	0
CCP	6*	2*	0*	6	2	0
CPB	4*	4*	0*	7	1	0
CPB + Activator	5*	3*	0*	7	1	0

Refer to legend of Table I and the main text for definitions of abbreviations.
*Data are quoted from Kobayashi et al. [15].
A, Adhesive failure at zirconia interface; AC, Combined cohesive/adhesive failure; C, Cohesive failure within indirect composite material.

Observation of debonded surfaces

To determine mode of failure, debonded surfaces were observed with a stereomicroscope (Stemi DV4, Carl Zeiss Co., Ltd., Jena, Germany) at an original magnification × 32. Mode of failure was classified as (A) adhesive failure between the indirect composite material and zirconia, (C) cohesive failure within the indirect composite material or (AC) combined adhesive/cohesive failure. All specimens were sputter-coated with osmium (HPC-IS, Vacuum Device, Mito, Japan) for 30 s and observed with a scanning electron microscope (S-4300, Hitachi High-Technologies Co. Ltd., Tokyo, Japan) operated at 15 kV.

Results

Shear bond strength

The results of shear bond strength testing are presented in Table II. After 100 000 thermocycles, mean bond strength varied from 9.4 to 23.9 MPa. The PLA group showed the lowest bond strength ($p = 0.010$), whereas the CPB + Activator (23.9 MPa; $p < 0.014$) and CPB (22.7 MPa; $p < 0.028$) groups exhibited significantly higher bond strengths than the other groups.

The Mann-Whitney U-test revealed that bond strengths did not significantly decrease after 100 000 thermocycles, except for those of the PLA ($p = 0.038$) and CCP ($p = 0.028$) groups (see Table II). Before thermocycling, the mean bond strengths of the CPB and CPB + Activator groups were high (> 21 MPa); after thermocycling, bond strength remained stable in those groups (> 23 MPa).

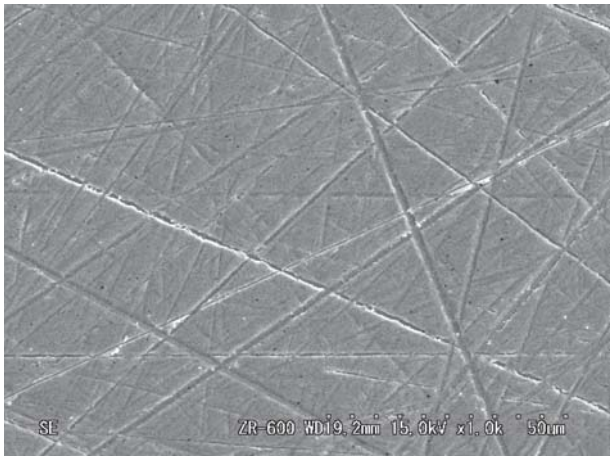

Figure 1. SEM image of zirconia surface ground with 600-grit silicon carbide paper (original magnification ×1000).

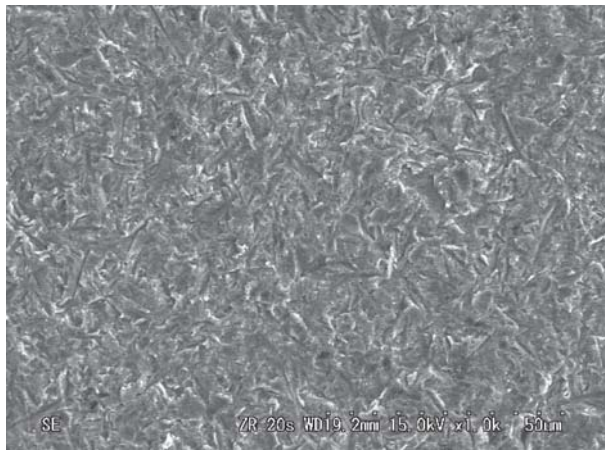

Figure 2. SEM image of airborne-particle-abraded zirconia surface (original magnification ×1000).

Failure mode

The distribution of failure modes after shear bond testing is shown in Table III. In the PLA and EOP groups, all failures were adhesive, regardless of

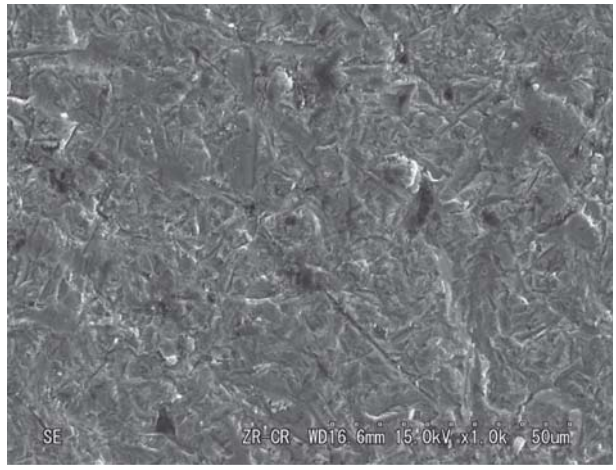

Figure 3. SEM image of the zirconia surface of a PLA group specimen after failure due to shear bond testing with 100 000 thermocycles (original magnification ×1000). This image shows total adhesive failure.

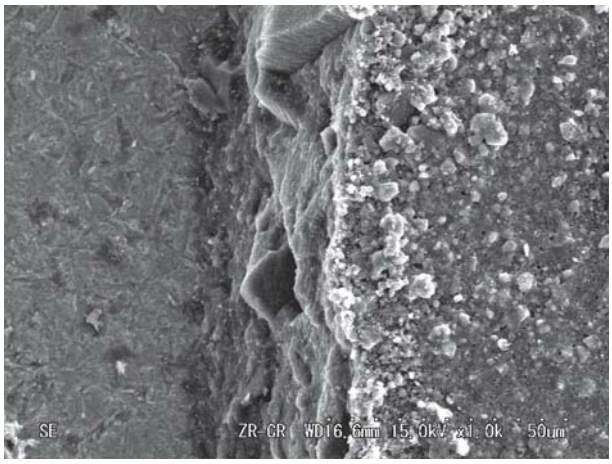

Figure 4. SEM image of the zirconia surface of a CPB + Activator group specimen after failure due to shear bond testing with 100 000 thermocycles (original magnification ×1000). In this example of combined cohesive/adhesive failure, both the original zirconia surface and remnants of adhered material can be seen.

thermal cycling. In the ALP group, although combined cohesive/adhesive failure was observed in two specimens without thermocycling, adhesive failure was observed in all specimens after thermocycling. Without thermocycling, the CCP, CPB and CPB + Activator groups exhibited either adhesive failure or combined cohesive/adhesive failure, whereas adhesive failure was predominant after thermocycling.

SEM observation

An SEM image of a zirconia surface wet-ground with 600-grit silicon carbide paper showed a smooth surface and zirconia grains (Figure 1). A representative SEM image of the zirconia surface after airborne-particle abrasion revealed sharp edges and undercuts (Figure 2). Figures 3 and 4 show two representative SEM images of fractured zirconia surfaces after shear bond testing. The representative SEM image of adhesive failure (Figure 3) was similar in appearance to the image after airborne-particle abrasion in Figure 2. Figure 4 shows a representative SEM image of combined failures and shows the remnants of indirect composite material on the specimen surface.

Discussion

To assess the possible clinical application of composite veneered zirconia-based restorations, the present study evaluated the long-term shear bond strength of an indirect composite to a zirconia framework material. There was no significant difference in shear bond strength between specimens exposed to 0 and 100 000 thermocycles, except in the PLA and CCP groups. Thus, the results of the present study partially confirm the hypothesis that artificial aging by means of thermocycling does not influence the bond strength between an indirect composite material and zirconia ceramics. Because the bond strength of an indirect composite to zirconia ceramics was influenced by the type of priming agent, we reject the second hypothesis, i.e. that there is no difference in bond strength among the priming agents.

Thermocycling [17–19] and long-term water storage [20] significantly influence resin bond to ceramics, especially high-strength ceramics, and these methods are thus commonly used for artificial aging in *in vitro* studies. Wegner et al. [18] suggested that thermocycling has a much greater impact than water storage at a constant temperature on the durability of resin bond strength to zirconia ceramics. Thermocycling might be effective in evaluating durability against water penetration of the adhesive interface in resin-to-ceramic-bonded specimens. In the present study, there were significant differences in the PLA group with regard to shear bond strength between specimens that were and were not exposed to 100 000 cycles of thermocycling and bond strengths were lowest in this group under both experimental conditions. Specific monomers, namely acidic monomers, were used for priming of the zirconia surfaces in the present study. This finding of the present study demonstrates that application of Porcelain Liner M Liquid A, which contains trimellitate anhydride (4-META), to zirconia ceramics did not result in a durable bond of Estenia C&B composite to Katana zirconia, after exposure to 100 000 thermocycles. In addition, bond strength in PLA group specimens was significantly lower than that of other groups, with and without [15] thermocycling. These results suggest that this carboxylic monomer is ineffective in creating a chemical bond with zirconia ceramics, which agrees with findings from previous studies [15,21].

In contrast, as compared with primer containing carboxylic monomer (4-META), application of primers containing hydrophobic phosphate monomer (MDP) resulted in higher bond strengths after thermocycling. These findings can be attributed to a difference in bonding ability between MDP monomer and 4-META. The effectiveness of MDP monomer in bonding dental base metal alloys has been studied [22,23]. The surfaces of base metal alloys as well as zirconia ceramics are typically covered with a metal oxide layer. Certain reactions could have occurred at the interface between the adhesive monomer and zirconium dioxide layer on the surface of primed zirconia [24,25]. It is likely that the MDP monomer was effective for bonding zirconia because zirconia is a dioxide of zirconium. In addition, thermocycling did not decrease the bond strength in specimens of zirconia ceramics surface-treated with agents containing MDP, with the exception of the CCP group. These results indicate that priming agents containing hydrophobic MDP monomer improve bond durability between an indirect composite material and a zirconia framework.

Silanes did not enhance the durability of bonds between indirect composite and zirconia ceramics, as there were no significant differences in bond strengths between the EOP and CCP groups or the CPB and CPB + Activator groups. These findings might be due to a missing silica phase in the Katana zirconia and agree with the results of previous studies indicating that a silane alone did not result in long-term durable bonding to zirconia [24,26].

Among the six groups, the CPB + Activator and CPB groups had significantly higher bond strengths after exposure to 100 000 thermocycles, which suggests that application of MDP and initiators promotes a water-resistant chemical bond between zirconia and an indirect composite material. More specifically, application of a combination of hydrophobic phosphate monomer (MDP) and initiators can yield superior long-term bonding strength between Katana zirconia and Estenia C&B composite. The results of other studies evaluating short- or long-term bond strength were similar to those of the present study [15,19]. Analysis of debonded surfaces (Table III) showed different failure mode distributions between CPB + Activator and CPB specimens that were and were not exposed to thermocycling. Although shear bond strength was not changed by aging in the CPB + Activator and CPB groups, the rate of adhesive failure increased after thermocycling, which suggests that the integrity of thermocycled specimens at the interface between the indirect composite and zirconia might be inferior to that of non-thermocycled specimens. In the literature, there is no consensus regarding the best regimen for artificial aging. Gale and Darvell [27] concluded that 10 000 thermocycles corresponded to approximately 1 year of *in vivo* function. The number of cycles in the present study was arbitrarily set at 100 000, so as to roughly correspond to 10 years *in vivo*. Although all *in vitro* studies have limitations, they are indispensable in evaluating new ideas and treatment methods before clinical use. Additional *in vitro* studies and randomized clinical trials are necessary before investigators can make detailed recommendations regarding methods of layering indirect composite materials to zirconia frameworks.

Within the limitations of the present study, it can be concluded that application of a hydrophobic phosphate monomer (MDP)/initiator combination results in durable long-term bonding between Katana zirconia and Estenia C&B composite material. In addition, the effect of artificial aging with thermocycling on the bond strength between zirconia and indirect composite material varies by priming agent.

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