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Shear bond strength between an indirect composite layering material and feldspathic porcelain-coated zirconia ceramics

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Abstract

Objectives This study aims to evaluate the effect of both feldspathic porcelain coating of zirconia frameworks and priming agents on shear bond strength between an indirect composite material and zirconia frameworks.

Materials and methods A total of 462 airborne-particleabraded zirconia disks were divided into three groups: untreated disks (ZR–AB), airborne-particle-abraded zirconia disks coated with feldspathic porcelain, (ZR–PO–AB), and hydrofluoric acid-etched zirconia disks coated with feldspathic porcelain (ZR–PO–HF). Indirect composite (Estenia C&B) was bonded to zirconia specimens with no (CON) or one of four priming agents—Clearfil Photo Bond (CPB), Clearfil Photo Bond with Clearfil Porcelain Bond Activator (CPB+activator), Estenia Opaque primer, or Porcelain Liner M Liquid B (PLB)—with or without an opaque material (Estenia C&B Opaque). All specimens were tested for shear bond strength before and after 20,000 thermocycles. The Steel–Dwass test and Mann–Whitney U test were used to compare shear bond strength.

Results In ZR–AB specimens, the initial bond strength of the CPB and CPB+Activator groups was significantly higher as compared with the other three groups (P<0.05),

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Department of Preventive and Restorative Sciences, Robert Schattner Center, University of Pennsylvania School of Dental Medicine, Philadelphia, USA whereas the PLB and CPB+Activator groups had the highest pre- and post-thermocycling bond strengths in ZR–PO– AB and ZR–PO–HF specimens. Among CON disks without opaque material, bond strength was significantly lower in ZR–AB specimens than in ZR–PO–AB and ZR–PO–HF specimens (P<0.05).

Conclusion Feldspathic porcelain coating of a Katana zirconia framework enhanced the bond strength of Estenia C&B indirect composite to zirconia independent of surface treatment. The use of a silane coupling agent and opaque material yields durable bond strength between the indirect composite and feldspathic-porcelain-coated zirconia.

Clinical relevance The results of the present study suggest that feldspathic porcelain coating of zirconia frameworks is an effective method to obtain clinically acceptable bond strengths of a layering indirect composite material to a zirconia framework.

Keywords Bond strength · Bonding agent · Indirect composite · Priming agent · Zirconia

Introduction

Several medium-term clinical trials demonstrate favorable performance and longevity of zirconium-dioxide (zirconia) ceramic-based restorations [1–7]. However, mechanical issues related to chipping of veneering porcelain still remain a concern [8]. Some causal factors for these mechanical issues have been identified in previous studies and include inappropriate framework design [9, 10], mismatched thermal properties of veneering porcelain and zirconia ceramics [10], and the use of incompatible veneering porcelains [11]. Consequently, inadequate bond of veneering porcelain to

the zirconia ceramic framework seems to be a relevant factor for veneer chipping [1, 2, 4, 6, 12, 13].

Several solutions to some of these issues have been investigated. For example, one clinical trial evaluated the clinical performance of posterior three-unit fixed partial dentures fabricated with the overpressing technique, in which a lithium-disilicate glass–ceramic material is pressed onto a zirconia framework [14]. The results of that study indicated excellent reliability with no veneer chippings after a follow-up period of 3 years [14]. In the attempt to decrease the incidence of cohesive porcelain fracture and other types of failure, customization of milled zirconia frameworks to provide even and controlled veneering porcelain thickness seems to be effective [15]. Typically, a full-contour wax-up is fabricated in conjunction with the dual-scanning technique to provide support for the veneering porcelain.

Recently, indirect composite materials have been introduced to zirconia-based restorations as an alternative to feldspathic porcelain for veneers [16, 17]. Kobayashi et al. [16] demonstrated that the bond strength of an indirect composite material to zirconia ceramics can be above the threshold for clinically acceptable composite-metal or ceramic bonds. In addition, it was concluded that indirect composite materials are considered promising alternatives as veneering materials for implant-supported zirconia restorations, which are exposed to higher occlusal stresses than tooth-supported restorations.

To achieve adequate adhesive resin bonds to zirconia-based restorations, coating of the intaglio surface with a silica-based ceramic material followed by silanization can be an effective tool [18, 19]. This efficacy can be attributed to the formation of siloxane bonds to the silica in the ceramic surface, as bond between silica-based ceramics and resin-composite materials with silane coupling agents is well established [20, 21]. In addition, Saito et al. [11] demonstrated promising bond strengths of layering porcelain to zirconia ceramics that were comparable to that of feldspathic porcelain to gold alloy and depended on the strength of the porcelain.

To obtain more durable bonds of indirect composite layering materials to zirconia ceramics, it may be advantageous to first fuse a feldspathic porcelain to the zirconia framework and then apply the composite with the respective silanization and bonding protocols. To date, however, there has not been scientific evidence on the validity, reliability, and longevity of this novel approach. Therefore, the purpose of the present study was to evaluate the effect of both feldspathic porcelain coating of zirconia frameworks and priming agents on the initial shear bond strength of an indirect composite material to zirconia ceramic frameworks, and the effect of artificial aging with thermocycling. The null hypothesis was that neither coating with feldspathic porcelain nor priming agents would affect the shear bond strength of an indirect composite material to zirconia ceramics, and artificial aging does not influence shear bond strength.

Materials and methods

The materials assessed in the present study and the experimental design is shown in Table 1 and Fig. 1, respectively. A

Material/Trade name	Abbreviation	Manufacturer	Lot no.	Components
Zirconia ceramic material				
Katana		Noritake Dental Supply Co., Ltd., Miyoshi, Japan		94.4% ZrO ₂ , 5.4% Y ₂ O ₃
Indirect composite material				
Estenia C&B		Kuraray Medical Inc., Tokyo, Japan		Dentin DA2, Opaque OA2
Feldspathic porcelain for zirconia				
Cerabien ZR		Noritake Dental Supply Co., Ltd.	4871	Shade base SBA2
Hydrofluoric acid				
Porcelain Etch		Ultradent, South Jordan, UT, USA		9.5% hydrofluoric acid gel
Priming agents				
Clearfil Photo Bond	CPB	Kuraray Medical Inc.	00423B	Catalyst; MDP, HEMA, bis-GMA
			00523A	Universal; accelerators, ethanol
Clearfil Porcelain Bond	Activator	Kuraray Medical Inc.	2273	3-TMSPMA
Activator				
Estenia Opaque Primer	EOP	Kuraray Medical Inc.	00157A	MDP, monomer solvent
Porcelain Liner M	PLB	Sun Medical Co., Ltd., Moriyama, Japan	RS1	3-TMSPMA
Liquid B				

 Table 1
 Materials assessed

MDP 10-methacrylogloxydecyl dihydrogen phosphate, 3-TMSPMA 3-trimethoxysilylpropyl methacrylate, HEMA 2-hydroxyethyl methacrylate, bis-GMA bisphenol A-glycidyl methacrylate



Fig. 1 Experimental design of the present study

total of 462 zirconia disk specimens (11 mm in diameter and 2.5 mm in thickness) were fabricated from zirconia ceramic materials (Katana, Noritake Dental Supply Co. Ltd., Miyoshi, Aichi, Japan) using computer-aided design and computer-aided manufacturing technology. All specimens were wet-ground flat up with 600-grit silicon-carbide paper, followed by airborne-particle abrasion with 50 μ m Al₂O₃ particles (Hi-Aluminas, Shofu Inc., Kyoto, Japan) at 0.2 MPa pressure from a distance of 10 mm for 20 s.

The zirconia specimens were divided at random into three groups (n=154) according to the surface pretreatment as follows: untreated disks (ZR–AB), airborne-particleabraded zirconia disks coated with feldspathic porcelain (ZR–PO–AB), and hydrofluoric acid-etched zirconia disks coated with feldspathic porcelain (ZR–PO–HF).

A piece of masking tape with a circular hole 5.0 mm in diameter was positioned on the surface of each zirconia disk to demarcate the area of feldspathic porcelain (Cerabien ZR SBA2, Noritake Dental Supply Co. Ltd.) coating. Porcelain powder was stirred in forming liquid (Noritake Dental Supply Co. Ltd.) and a thin layer of the feldspathic porcelain was immediately applied to the specimen surfaces. The masking tape was then carefully removed and the specimens were fired at 930°C for 1 min in a vacuum furnace (SingleMat Porcelain Furnace, Shofu Inc.) to create a porcelain coating on the surface of the zirconia specimens. In group ZR–PO–AB, the surfaces of half the coated specimens were airborne-particle abraded with Al_2O_3 particles (Hi-Aluminas, Shofu Inc.) for 2 s at a pressure of 0.2 MPa from a distance of 10 mm. The surfaces of the other half of the coated specimens in group ZR–PO–HF were acid etched with 9.5% hydrofluoric acid gel (Porcelain Etch, Ultradent, South Jordan, UT, USA) for 1 min, rinsed with distilled water for 20 s, ultrasonically cleaned in methanol, and dried with oil-free air spray (Air Duster AD400FL, Orientec Inc., Misato, Saitama, Japan).

Specimens from each surface pretreatment group were further randomly divided into five groups and assigned to one of the five following surface treatments: Clearfil Photo Bond (CPB, Kuraray Medical Inc., Tokyo, Japan), Clearfil Photo Bond with Clearfil Porcelain Bond Activator (CPB+ Activator, Kuraray Medical Inc.), Estenia Opaque primer (EOP, Kuraray Medical Inc.), Porcelain Liner M Liquid B (PLB, Sun Medical Co., Ltd., Moriyama, Japan), and no primer as control (CON). Clearfil Porcelain Bond Activator and PLB contain a silane coupling agent. CPB and EOP contain 10-methacryloyloxydecyl dihydrogen phosphate (MDP), an adhesive functional monomer. Next, the specimens of each group were divided into two groups (n=11), with or without application of an opaque material of indirect composite (Estenia C&B Body Opaque OA2, Kuraray Medical Inc.) on the specimens. Except for the EOP and control

Groups	ZR–AB			ZR-PO-A	ΔB			ZR-PO-F	ZR–PO–HF					
	Median	Mean (SD)			Median	Mean (SD)			Median	Mean (SD)				
CON	0.1	0.1 (0.1)	а	А	3.8	3.0 (2.5)	d	В	4.8	3.8 (2.6)	g	В		
EOP	7.7	7.3 (1.3)	b	С	10.7	11.4 (2.5)	e	D	9.0	8.8 (1.7)	h	C, D		
PLB	0.1	0.2 (0.2)	а	Е	15.4	14.5 (1.9)	e, f	F	13.7	14.0 (1.5)	h, i	F		
CPB CPB+	13.0	13.1 (2.0)	с	G	12.9	12.6 (3.1)	e	G	11.4	11.1 (3.0)	h	G		
Activator	13.3	13.3 (2.7)	c	Н	17.0	17.9 (2.7)	f	Ι	15.7	16.2 (2.1)	i	Ι		

Table 2 Shear bond strength (MPa) of Estenia C&B composite to Katana zirconia (without opaque material)

Identical uppercase letters in the same row indicate that the values are not statistically different (Steel–Dwass test, P>0.05)

Identical lowercase letters in the same column indicate that the values are not statistically different (Steel–Dwass test, P > 0.05)

groups, each group underwent EOP application before bonding of the opaque material to the zirconia disks. After surface preparation of the specimens, a piece of doublecoated tape with a circular hole 5.0 mm in diameter was positioned on each zirconia disk to define the area of bonding. A thin layer of opaque material was placed on the specimen surface and exposed to light for 90 s in a laboratory light-polymerization unit (α -Light II, J. Morita Corp., Suita, Japan). An additional layer was applied on top of the primary opaque material in the same manner.

A stainless ring (6.0 mm inner diameter, 2.0 mm length) was placed around the opaque material. The ring was filled with a dentin shade of composite material (Estenia C&B Dentin DA2, Kuraray Medical Inc.) at a standardized force of 5 N. The specimen was then light cured in the polymerization unit (α -Light II, J. Morita Corp.) for 5 min and polymerized in a heat oven (KL-310, J. Morita Corp.) at 110°C for 15 min. All specimens were stored in distilled water at 37°C for 24 h after 30 min of specimen preparation.

To evaluate the effect of simulated aging, the specimens with application of the opaque material that achieved higher initial shear bond strength were duplicated to undergo simulated aging. The specimens were subjected to thermocycling for 20,000 cycles between 5°C and 55°C with a 1-min

dwell time using a thermocycling apparatus (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co. Ltd., Tokyo, Japan). All specimens that did not debond with the thermocycling were subjected to shear bond testing.

Each specimen was embedded in a steel mold and seated in a shear testing jig (ISO TR 11405, Tokyo Giken Inc., Tokyo, Japan). Shear bond strengths were determined using a mechanical testing machine (Type 5567, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/min. Loads were converted to MPa by dividing the failure load (N) by the bonding area (mm²).

The results of shear bond strength were analyzed using software for statistical analysis (SPSS version 15.0, SPSS, Inc., Chicago, IL, USA). Equality of variance in bond strengths was primarily analyzed by using the Levene test. When the Levene test did not show equality of variances, the Steel–Dwass test (KyPlot 5.0, Kyens Lab, Tokyo, Japan) was performed. The Mann–Whitney U test was used for comparisons between groups of specimens with and without application of opaque material, and between pre- and post-thermocycling. P values less than 0.05 were considered to indicate statistical significance.

After shear bond testing, the fractured interfaces of specimens were examined with a stereomicroscope (StemiDV4,

Groups	ZR-AB		ZR-PO-A	В			ZR-PO-HF							
	Median	Mean (SD)	Mean (SD)			Median Mean (SD)				n Mean (SD)				
CON	3.5	3.6 (2.5)	а	А	6.9	6.8 (2.0)	d	В	13.6	13.8 (1.4)	g	С		
EOP	12.6	13.1 (2.2)	b	D, E	10.7	11.3 (2.7)	e	D	15.2	14.5 (1.7)	g, h	Е		
PLB	14.8	14.2 (2.3)	b	F	19.4	19.9 (3.3)	f	G	17.1	17.3 (3.0)	h, i	F, G		
CPB	20.4	20.0 (1.8)	c	Н	10.5	10.9 (1.3)	e	Ι	14.2	14.6 (2.6)	g, h, i	J		
CPB+														
Activator	19.7	19.5 (1.9)	c	K	19.6	19.5 (2.6)	f	Κ	18.4	18.2 (3.0)	i	Κ		

 Table 3
 Shear bond strength (MPa) of Estenia C&B composite to Katana zirconia (with opaque material)

Identical uppercase letters in the same row indicate that the values are not statistically different (Steel–Dwass test, P>0.05)

Identical lowercase letters in the same column indicate that the values are not statistically different (Steel–Dwass test, P>0.05)

Table 4 P values for comparison of shear bond strengths in specimens

 with and without application of opaque material

Groups	ZR–AB	ZR-PO-AB	ZR–PO–HI				
CON	0.000*	0.002*	0.000*				
EOP	0.000*	1.000	0.000*				
PLB	0.000*	0.000*	0.004*				
CPB	0.000*	0.151	0.014*				
CPB+							
Activator	0.000*	0.200	0.088				

*P<0.05, significant difference between specimens with and without application of opaque material, Mann–Whitney U test

Carl Zeiss Co., Ltd., Jena, Germany) at an original magnification×32 to determine the mode of failure. The failure modes for the ZR-AB specimens were A (adhesive failure at the zirconia-composite material interface) and B (combined adhesive failure and cohesive failure within the composite material). The failure modes for the ZR-PO-AB and ZR-PO-HF specimens were C (adhesive failure at the porcelaincomposite material interface), D (adhesive failure at the porcelain–composite material interface with cohesive failure within the porcelain), and E (cohesive failure within the porcelain). After surface preparation and shear bond testing, representative specimens were sputtered with osmium and observed with a scanning electron microscope (SEM, S-4300, Hitachi high-Technologies Co. Ltd., Tokyo, Japan) operated at 15 kV.

X-ray diffraction (XRD) with Cu K α radiation was used to analyze airborne-particle-abraded zirconia surfaces, the porcelain-coated surfaces of zirconia that had been airborne-particle abraded or acid etched with 9.5% hydrofluoric acid gel feldspathic, and each subgroup of zirconia specimens after shear bond testing. The specimens were placed in the holder of an X-ray diffractometer (MiniFlex, Rigaku Corp., Tokyo, Japan) and, using flat-plate geometry, data were collected at 30 kV (15 mA) with a scanning range of 15–29°.

Results

Table 2 shows the shear bond strengths of Estenia C&B composite to Katana zirconia material, without application of opaque material. Among ZR–AB specimens, the bond strengths of the CPB and CPB+Activator groups were significantly higher than those of the other three groups. There was no significant difference between the CON and PLB groups. Among ZR–PO–AB and ZR–PO–HF specimens, the PLB and CPB+Activator groups had the highest bond strengths. In the CON group, the bond strength of ZR–AB specimens was significantly lower than those of ZR–PO–AB and ZR–PO–HF specimens.

Table 3 shows the shear bond strengths of Estenia C&B composite to zirconia material, with application of opaque material. Among ZR–AB specimens, the bond strengths of the CPB and CPB+Activator groups were significantly higher than those of the other three groups. Among ZR–PO–AB specimens, the PLB and CPB+Activator groups had significantly higher bond strengths. Among ZR–PO–HF specimens, the PLB, CPB, and CPB+Activator groups had the highest bond strengths, ranging from 14.6 to 18.2 MPa. In the CON group, the bond strength of ZR–PO–HF specimens was significantly higher than those of the other two subgroups.

As shown in Table 4, the Mann–Whitney U test revealed that bond strengths increased significantly with the use of the opaque material, except for the EOP (P=1.000), CPB (P=0.151), and CPB+Activator (P=0.200) groups in ZR–PO–AB specimens and the CPB+Activator (P=0.088) group in ZR–PO–HF specimens.

Table 5 presents the shear bond strengths after 20,000 thermocycles for the groups with application of opaque material, and Table 6 shows the comparison of bond strength between pre- and post-thermocycling. For ZR–PO–AB specimens, the EOP or CPB group displayed a significant reduction of shear bond strength after thermocycling; whereas the PLB or CPB+Activator group exhibited no reduction of that. The ZR–PO–HF specimens showed

Groups	ZR–AB		ZR-PO-A	B		ZR–PO–HF						
	Median	Mean (SD)			Median	Mean (SD)			Median	Mean (SD)		
ЕОР	23.9	23.2 (2.1)	а	А	0.8	1.2 (1.1)	b	В	15.7	16.2 (2.1)	d	С
PLB	22.4	22.2 (5.0)	а	D	19.5	20.1 (5.4)	с	D	20.7	21.0 (4.1)	e	D
CPB	23.2	22.7 (5.0)	а	Е	1.6	2.0 (1.5)	b	F	16.2	15.8 (4.3)	d	G
CPB+												
Activator	24.2	24.3 (2.8)	а	Н	25.0	25.9 (4.1)	с	Н	22.9	23.2 (3.6)	e	Н

Table 5 Shear bond strength (MPa) of Estenia C&B composite to Katana zirconia (with opaque material) after 20,000 thermocycles

Identical uppercase letters in the same row indicate that the values are not statistically different (Steel–Dwass test, P > 0.05)

Identical lowercase letters in the same column indicate that the values are not statistically different (Steel–Dwass test, P > 0.05)

Groups	ZR-AB (reduction)	ZR-PO-AB (reduction)	ZR-PO-HF (reduction)
EOP	0.000* (-77.1)	0.000* (89.4)	0.047* (-11.7)
PLB	0.000* (-56.3)	0.898 (-1.0)	0.034* (-21.4)
CPB	0.332 (-13.5)	0.000* (81.7)	0.519 (-8.2)
CPB+			
Activator	0.000* (-24.6)	0.001* (-32.8)	0.005 (-27.4)

Table 6 P values and reduction (%) for comparison of shear bond strengths in groups at 0 and 20,000 thermocycles

*P<0.05, Statistically significant difference between two thermocycling conditions (0 and 20,000 thermocycles); Mann–Whitney U test

significantly higher shear bond strength than ZR–PO–AB specimens in EOP and CPB groups. In addition, there was significant difference between the EOP or CPB group and PLB or CPB+Activator group for ZR–PO–HF specimens. For ZR–AB specimens, all groups exhibited stable shear bond strength after 20,000 thermocycles.

Table 7 shows the failure modes observed by optical microscopy after shear bond testing. For ZR–AB specimens, the failure mode in all groups without application of opaque material was adhesive failure at the zirconia-composite material interface. For two groups (CPB, CPB+Activator), combined cohesive failure within the composite material and adhesive failure was observed in specimens with opaque material. However, for ZR–PO–AB and ZR–PO–

HF specimens, most debonded specimens showed adhesive failure at the porcelain-composite material interface with cohesive failure within the porcelain, regardless of whether opaque material had been applied. The CPB+Activator and PLB groups exhibited cohesive failure within the porcelain with and without application of the opaque material. The unprimed control group showed adhesive failure at the porcelain–composite interface. After 20,000 thermocycles, adhesive failure at the porcelain–composite interface was observed in the EOP and CPB groups for ZR–PO–AB specimens.

SEM images of zirconia surface prepared for ZR-AB, ZR-PO-AB, and ZR-PO-HF specimens before bonding are showed in Fig. 2. Airborne-particle abrasion with 50um Al₂O₃ altered the superficial ceramic layer and created a roughened pattern that is characteristic of a microscopically treated surface. The surface of ZR-AB specimens exhibited small surface irregularities (Fig. 2a), whereas large irregularities were visible on the surface of ZR-PO-AB specimens (Fig. 2b). In contrast, ZR-PO-HF specimens showed complicated microporosity with marked undercuts (Fig. 2c). Figures 3 and 4 are representative SEM images of debonded ZR-AB surfaces. Figure 3 illustrates areas of adhesive failure, which can have exactly the same appearance as a surface before priming. Figure 4 shows combined cohesive and adhesive failure; both the original zirconia surface and remnants of adhered material are visible. Figures 5 and 6 are

Table 7 Failure modes after shear bond testing			Without opaque material				With opaque material										
								The	Thermocycle 0				The	ermoc	cycles	20,00)0
	Ceramics	Primer	А	В	С	D	Е	A	В	С	D	Е	A	В	С	D	E
	ZR-AB	CON	11	0				11	0				-	-			
		EOP	11	0				11	0				8	3			
		PLB	11	0				10	0				9	2			
		CPB	11	0				4	7				8	3			
		CPB+															
		Activator	11	0				6	5				7	4			
	ZR-PO-AB	CON			11	0	0			9	2	0			-	-	-
A adhesive failure at		EOP			0	11	0			0	11	0			11	0	0
zirconia-composite material		PLB			0	11	0			0	8	3			0	4	7
interface, B combination		CPB			0	11	0			0	11	0			11	0	0
adhesive failure at zirconia-		CPB+															
cohesive failure within		Activator			0	8	3			0	7	4			0	9	2
composite material, <i>C</i> adhesive failure at porcelain-composite material interface, <i>D</i> adhesive failure at porcelain-composite material interface with cohesive failure within the porcelain, <i>E</i>	ZR-PO-HF	CON			11	0	0			7	4	0			-	-	-
		EOP			0	11	0			0	10	1			1	9	1
		PLB			0	8	3			0	9	2			0	4	7
		CPB			0	11	0			0	11	0			2	7	2
		CPB+															
cohesive failure within the porcelain		Activator			0	8	3			0	9	2			0	5	6





Fig. 2 SEM images of **a** zirconia surface after airborne-particle abrasion (ZR–AB), **b** feldspathic porcelain-coated zirconia surface after airborne-particle abrasion (ZR–PO–AB), and **c** feldspathic porcelain-coated zirconia surface after hydrofluoric acid etching (ZR–PO–HF; original magnification,×1,000)

typical SEM images of ZR–PO–AB and ZR–PO–HF specimens after shear bond testing. The surfaces of the specimens were completely covered with porcelain material. The



Fig. 3 SEM image of debonded ZR-AB surface of PLB specimen without application of opaque material (original magnification, ×1,000)

surface shown in Fig. 5 differs from that of a surface before priming, whereas the surface shown in Fig. 6 shows partially resembles a surface before priming.

The XRD patterns of selected specimens are presented in Figs. 7 and 8. The XRD pattern of zirconia disks after airborne-particle abrasion (Fig. 7a) conforms to that observed after adhesive failure at the zirconia-composite material interface after shear bond testing in ZR–AB specimens (Fig. 7b). The XRD pattern of combined adhesive failure at the zirconia-composite interface and cohesive failure within the indirect composite material after shear bond testing in ZR–AB specimens (Fig. 7c) was similar to that of the indirect composite material (Fig. 7d), which showed peaks corresponding to SiO₂ (2 θ =21.2°, 23.6°, 26.4°). The XRD patterns of the surface of feldspathic porcelain-coated zirconia (Fig. 8a) and the surface of ZR–PO–AB and ZR–PO–HF specimens after shear bond testing (Fig. 8b–d) were very



Fig. 4 SEM image of debonded ZR–AB surface of CPB+Activator specimen with application of opaque material (original magnification, $\times 1,000$)



Fig. 5 SEM image of debonded ZR–PO–AB surface of CPB+Activator specimen with application of opaque material after shear bond testing (original magnification, ×1,000)

similar $(2\theta=15-29^{\circ})$. These XRD patterns showed amorphous glass, and their main peaks were at $2\theta=21.82^{\circ}$, 27.04° .

Discussion

The present study evaluated the effect of both feldspathic porcelain coating of zirconia frameworks and priming agents on the initial or artificially aged shear bond strength between an indirect composite material and modified zirconia frameworks. The results of the present study support rejection of the first part of the null hypothesis, namely, that feldspathic porcelain coating of zirconia ceramics does not affect the shear bond strength of an indirect composite material to the coated zirconia ceramics. However, there was no significant difference in shear bond strength between priming agents. Therefore, the results do not support rejection of the null hypothesis for this variable. Significant differences in shear bond strength were found among the priming agents assessed in this study. Thus, the second part of the null hypothesis, i.e., that the priming agents would not influence the shear bond strength of an indirect composite material to the zirconia ceramics, was rejected. Based on the results after thermocycling obtained, the third part of the hypothesis that artificial aging does not influence shear bond strength was disproved. The shear bond strength after thermocycling was significantly decreased in group EOP and CPB for ZR-PO-AB specimens.

In the CON group, ZR–PO–AB and ZR–PO–HF specimens revealed significantly higher bond strengths as compared with ZR–AB specimens, without application of opaque material. The results indicate that feldspathic porcelain coating of the zirconia framework enhances the initial bond strength between the indirect composite material and the zirconia framework. Our SEM images indicate that airborne-particle abrasion of the feldspathic-porcelain-coated zirconia creates a rougher surface as compared with airborne-particle abrasion of the noncoated zirconia. ZR–PO–HF specimens exhibit numerous honeycombed defects and relief patterns, suggesting increased retentive characteristics of the etched surface. These findings indicate that feldspathic-porcelain-coated zirconia, treated with either airborne-particle abrasion or hydrofluoric acid etching, is able to provide a more retentive surface than airborne-particle abraded noncoated zirconia.

Initial bond strengths of ZR-PO-HF specimens were the highest among the other subgroups with application of opaque material in the CON group. Therefore, the opaque composite material seems to further promote adhesive bonds to the etched surface of feldspathic-porcelain-coated zirconia. In addition, the ZR-PO-HF specimens showed durable shear bond strength compared with ZR-PO-AB specimens in EOP and CPB groups with application of opaque material after thermocycling. The effectiveness of hydrofluoric acid etching to achieve high and durable resin bonds to feldspathic porcelain has been shown in previous studies. [21-23]. It is possible that the hydrofluoric acid treatment combined with a high-flow bonding agent provides better bonding due to increased wettability and, therefore, improved micromechanical interlocking. On the other hand, the shear bond strength after thermocycling was significantly decreased in group EOP and CPB for ZR-PO-AB specimens. It can be speculated that the airborne-particle abrasion to porcelain-coated zirconia surface does not enhance the micromechanical interlocking.

For ZR-AB specimens, the CPB and CPB+Activator groups had significantly higher shear bond strength among



Fig. 6 SEM image of debonded ZR–PO–HF surface of EOP specimen with application of opaque material after shear bond testing (original magnification, \times 1,000)





Fig. 7 XRD patterns of a zirconia ceramic surface after airborneparticle abrasion, b adhesive failure at zirconia-composite material interface after shear bond testing in a ZR-AB specimen, c combined

the five groups, with and without application of opaque material. Failure modes of all specimens without opaque material application for ZR–AB specimens were adhesive at the zirconia–composite interface. However, combination adhesive failures at the zirconia–composite interface and cohesive failure within the composite material were observed in CPB and CPB+Activater groups bonded with the bonding agent and opaque material. XRD analysis in



Fig. 8 XRD patterns of **a** the surface of feldspathic porcelain-coated zirconia, **b** adhesive failure at the porcelain-composite material interface after shear bond testing in a ZR–PO–AB specimen, **c** cohesive failure within the porcelain after shear bond testing in a ZR–PO–HF specimen, and **d** adhesive failure at the porcelain–composite material interface with cohesive failure within the porcelain after shear bond testing in a ZR–PO–HF specimen, and **d** adhesive failure within the porcelain after shear bond testing in a ZR–PO–HF specimen.

adhesive failure at zirconia-composite material interface and cohesive failure within indirect composite material after shear bond testing in a ZR-AB specimen, and **d** indirect composite material

the present study validated these findings as remnants of the composite elements such as silica were detected. As previously indicated in other studies [16, 17], our results demonstrate that application of both a hydrophobic phosphate monomer (MDP) and a polymerization initiator is effective for short-term bonding between the Katana zirconia and the Estenia composite. Kobayashi et al. [16] reported that application of a priming agent containing MDP as well as a high-flow bonding agent (opaque composite) yielded superior bond strength between zirconia ceramic and indirect composite. Furthermore, the primers containing a phosphate monomer (MDP) enhanced the durable bond strength after thermocycling in the present study, which is agreement with the study [17].

The present study demonstrated that application of a silane coupling agent to the pretreated ceramic surface results in satisfactory bonds between the indirect composite material and the porcelain-coated zirconia. For both the ZR-PO-AB and ZR-PO-HF specimens, PLB and CPB+Activator groups showed superior initial as well as durable shear bond strengths as compared to the other groups. The most probable reason for this finding is the formation strong chemical bonds through a siloxane network between the silane and the silica in the feldspathic porcelain [20, 21]. This reasoning is further supported by the prevalence of cohesive failures within the porcelain in all three groups bonded with silane coupling agent and bonding agent or opaque composite material for the ZR-PO-AB and ZR-PO-HF specimens. Therefore, silane coupling agents and opaque composite materials greatly improve bond strength of composite to porcelaincoated zirconia.

With regard to the effect of silanization on the bond strength of indirect composite material to zirconia ceramics, the results of the present study demonstrated that silane application without porcelain coating does not enhance bond strengths. No significant differences were found between CON and PLB groups as well as between CPB and CPB+activator groups for ZR-AB specimens. These findings are consistent with previous studies, which showed that a silane coupling agent is ineffective as a priming agent to zirconia due to the lack of a silica phase in high-purity oxide ceramics [24, 25]. However, once that surface is coated with silica, for example through air-particle abrasion with silica-containing particles, a silane coupling agent functions as a reliable promoter of resin–zirconia bonds, as shown by Blatz and coworkers [26].

Interestingly, there were no adhesive failures between the feldspathic-porcelain coat and the zirconia for the ZR–PO–AB and ZR–PO–HF specimens. XRD analysis also confirmed that the feldspathic porcelain remained on the zirconia surface, indicating no adhesive failure at that interface. These findings imply that the bond of the porcelain to the zirconia exceeds the bond between the porcelain and the indirect composite, therefore, demonstrates that porcelain coating is a valid method to improve adhesive resin bonds to zirconia. These results confirm the findings of a previous in vitro study [11], where shear bond strength of feldspathic porcelain to zirconia was dependent on the strength of the layering porcelain.

The bond strength of the composite material to zirconia ceramics increased with thermocycling in all groups, except for the EOP and CPB groups in the ZR–PO–AB specimens. These findings are consist with the study [17], which can be presumed that polymerization of the Estenia composite material was still progressing at 37°C for 24 h, and that thermal stress at 55°C with 20,000 thermocycles resulted in additional polymerization of the composite material.

It is not clear at which threshold bond strengths of layering materials to zirconia frameworks can be deemed "clinically acceptable". However, several researchers have suggested that the clinically acceptable range of bond strengths for composite-ceramics and composite-metal bonds ranges between 10 and 13 MPa [27, 28]. Because the bond strengths for some combinations of materials in the present study were approximately 20 MPa, coating of zirconia frameworks with feldspathic porcelain seems to be an effective method to obtain clinically acceptable bond strengths between a layering indirect composite material and a zirconia framework.

While the results of this study are encouraging, they have to be interpreted with caution. One of the shortcomings of this study is the specimen design. The simplified shear bond strength test and specimen design allow for some basic bond evaluations under standardized and controlled circumstances but fail to simulate the complex interaction between the threedimensionally shaped frameworks and separately applied veneering materials. The validity of using indirect composites as veneering materials to zirconia should ultimately be verified in randomized clinical trials.

Conclusion

Feldspathic porcelain coating of a Katana zirconia framework enhances the bond strength of Estenia C&B indirect composite to zirconia independent of surface treatment. In addition, the application of a high-flow bonding agent (Estenia C&B Opaque) improves bonding of indirect composite to the porcelain-coated and non-coated zirconia. The use of a silane coupling agentand opaque composite material yields superior durable bond strength between the indirect composite and feldspathic-porcelain-coated zirconia.

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References

- Vult von Steyern P (2005) All-ceramic fixed partial dentures. Studies on aluminum oxide- and zirconium dioxide-based ceramic systems. Swed Dent J Suppl 173:1–69
- Raigrodski AJ, Chiche GJ, Potiket N, Hochstedler JL, Mohamed SE, Billiot S, Mercante DE (2006) The efficacy of posterior threeunit zirconium-oxide-based ceramic fixed partial dental prostheses: a prospective clinical pilot study. J Prosthet Dent 96:237–244
- Sailer I, Fehér A, Filser F, Lüthy H, Gauckler LJ, Schärer P, Franz Hämmerle CH (2006) Prospective clinical study of zirconia posterior fixed partial dentures: 3-year follow-up. Quintessence Int 37:685–693
- Sailer I, Fehér A, Filser F, Gauckler LJ, Lüthy H, Hämmerle CH (2007) Five-year clinical results of zirconia frameworks for posterior fixed partial dentures. Int J Prosthodont 20:383–388
- Edelhoff D, Florian B, Florian W, Johnen C (2008) HIP zirconia fixed partial dentures—clinical results after 3years of clinical service. Quintessence Int 39:459–471
- Molin MK, Karlsson SL (2008) Five-year clinical prospective evaluation of zirconia-based Denzir 3-unit FPDs. Int J Prosthodont 21:223–227
- Roediger M, Gersdorff N, Huels A, Rinke S (2010) Prospective evaluation of zirconia posterior fixed partial dentures: four-year clinical results. Int J Prosthodont 23:141–148
- Komine F, Blatz MB, Matsumura H (2010) Current status of zirconia-based fixed restorations. J Oral Sci 52:531–539
- Taskonak B, Yan J, Mecholsky JJ Jr, Sertgoz A, Kocak A (2008) Fractographic analyses of zirconia-based fixed partial dentures. Dent Mater 24:1077–1082
- Fischer J, Stawarzcyk B, Trottmann A, Hämmerle CH (2009) Impact of thermal misfit on shear strength of veneering ceramic/ zirconia composites. Dent Mater 25:419–423
- Saito A, Komine F, Blatz MB, Matsumura H (2010) A comparison of bond strength of layered veneering porcelains to zirconia and metal. J Prosthet Dent 104:247–257

- Tinschert J, Natt G, Mautsch W, Augthun M, Spiekermann H (2001) Fracture resistance of lithium disilicate-, alumina-, and zirconia-based three-unit fixed partial dentures: a laboratory study. Int J Prosthodont 14:231–238
- Aboushelib MN, Kleverlaan CJ, Feilzer AJ (2006) Microtensile bond strength of different components of core veneered all-ceramic restorations. Part II: zirconia veneering ceramics. Dent Mater 22:857–863
- Beuer F, Edelhoff D, Gernet W, Sorensen JA (2009) Threeyear clinical prospective evaluation of zirconia-based posterior fixed dental prostheses (FDPs). Clin Oral Investig 13:445–451
- Marchack BW, Futatsuki Y, Marchack CB, White SN (2008) Customization of milled zirconia copings for all-ceramic crowns: a clinical report. J Prosthet Dent 99:169–173
- 16. Kobayashi K, Komine F, Blatz MB, Saito A, Koizumi H, Matsumura H (2009) Influence of priming agents on the short-term bond strength of an indirect composite veneering material to zirconium dioxide ceramic. Quintessence Int 40:545–551
- Komine F, Kobayashi K, Saito A, Fushiki R, Koizumi H, Matsumura H (2009) Shear bond strength between an indirect composite veneering material and zirconia ceramics after thermocycling. J Oral Sci 51:629–634
- Kitayama S, Nikaido T, Maruoka R, Zhu L, Ikeda M, Watanabe A, Foxton RM, Miura H, Tagami J (2009) Effect of an internal coating technique on tensile bond strengths of resin cements to zirconia ceramics. Dent Mater J 28:446–453

- Kitayama S, Nikaido T, Ikeda M, Alireza S, Miura H, Tagami J (2010) Internal coating of zirconia restoration with silica-based ceramic improves bonding of resin cement to dental zirconia ceramic. Biomed Mater Eng 20:77–87
- Horn HR (1983) Porcelain laminate veneers bonded to etched enamel. Dent Clin North Am 27:671–684
- Blatz MB, Sadan A, Kern M (2003) Resin-ceramic bonding: a review of the literature. J Prosthet Dent 89:268–274
- Chen JH, Matsumura H, Atsuta M (1998) Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain. J Dent 26:53–58
- Kato H, Matsumura H, Atsuta M (2000) Effect of etching and sandblasting on bond strength to sintered porcelain of unfilled resin. J Oral Rehabil 27:103–110
- Kern M, Wegner SM (1998) Bonding to zirconia ceramic: adhesion methods and their durability. Dent Mater 14:64–71
- Wegner SM, Kern M (2000) Long-term resin bond strength to zirconia ceramic. J Adhes Dent 2:139–147
- Blatz MB, Chiche G, Holst S, Sadan A (2007) Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. Quintessence Int 38:745–753
- Thurmond JW, Barkmeier WW, Wilwerding TM (1994) Effect of porcelain surface treatments on bond strengths of composite resin bonded to porcelain. J Prosthet Dent 72:355–359
- Matsumura H, Yanagida H, Tanoue N, Atsuta M, Shimoe S (2001) Shear bond strength of resin composite veneering material to gold alloy with varying metal surface preparations. J Prosthet Dent 86:315–319

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