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Post-thermocycling shear bond strength of a gingiva-colored indirect composite layering material to three implant framework materials

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Abstract

Objective. To evaluate shear bond strength of a gingiva-colored indirect composite to three implant framework materials, before and after thermocycling, and verify the effect of surface pre-treatment for each framework. ***Materials and methods.*** Commercially pure titanium (CP-Ti), American Dental Association (ADA) type 4 casting gold alloy (Type IV) and zirconia ceramics (Zirconia) were assessed. For each substrate, 96 disks were divided into six groups and primed with one of the following primers: Alloy Primer (ALP), Clearfil Photo Bond (CPB), Clearfil Photo Bond with Clearfil Porcelain Bond Activator (CPB+Activator), Estenia Opaque Primer (EOP), Metal Link (MLP) and V-Primer (VPR). The specimens were then bonded to a gingiva-colored indirect composite (Ceramage Concentrate GUM-D). Shear bond strengths were measured at 0 and 20 000 thermocycles and data were analyzed with the Steel-Dwass test and Mann-Whitney U-test. ***Results.*** Shear bond strengths were significantly lower after thermocycling, with the exception of Type IV specimens primed with CPB (p = 0.092) or MLP (p = 0.112). For CP-Ti and Zirconia specimens, priming with CPB or CPB+Activator produced significantly higher bond strengths at 0 and 20 000 thermocycles, as compared with the other groups. For Type IV specimens, priming with ALP or MLP produced higher bond strengths at 0 and 20 000 thermocycles. ***Conclusions.*** Shear bond strength of a gingiva-colored indirect composite to CP-Ti, gold alloy and zirconia ceramics was generally lower after thermocycling. Application of a hydrophobic phosphate monomer and polymerization initiator was effective in maintaining bond strength of CP-Ti and zirconia ceramics. Combined use of a thione monomer and phosphoric monomer enhanced the durable bond strength of gold alloy.

Key Words: *bond strength, gingiva-colored, implant framework, indirect composite, in-vitro study*

Introduction

Osseointegrated dental implants are used to support fixed and removable prostheses for edentulous and partially edentulous patients. To ensure adequate stiffness, the framework of implant-supported prostheses is made of gold, cobalt–chromium or titanium alloys [1]. However, these metal alloys are heavy, often unwieldy and are thus frequently miscast. To overcome these issues, computer-aided design/computer-aided manufacturing (CAD/CAM) technology was developed and has gained acceptance in implant dentistry [2–4]. CAD/CAM technology makes it possible to machine-mill even large frameworks from pre-fabricated homogeneous blanks made of different materials. Recently, it became possible to fabricate large and complex frameworks for tooth- and implant-supported prostheses by machine-milling zirconium dioxide (zirconia) ceramic blocks [5]. Zirconia has excellent chemical and physical properties, good biocompatibility and resists plaque accumulation [5–7]. Thus, one-piece zirconia-based frameworks for implant-supported prostheses can now be fabricated owing to technological improvements in their design and manufacture [8,9]. Moreover, zirconia-based prostheses are esthetically superior due to the natural color of the framework, which eliminates the gray effect of metal frameworks.

Long-term esthetic and functional success of implant-supported prostheses requires optimal three-dimensional positioning of the implants. However, inadequate bone volume in edentulous jaws sometimes

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Table I. Materials assessed in the present study.

Material/Trade name	Abbreviation	Lot No.	Components	Manufacturer
CP-Titanium				
Ti Target	CP-Ti		Ti 99%	Japan Metal Service Co., Ltd., Saitama, Japan
Type 4 casting gold alloy				
Casting Gold M.C. Type IV	Type IV	1106031	Au 70%, Cu 16%, Ag 8%, Pt 2%, Pd 3%	GC Corp., Tokyo, Japan
Zirconia ceramics				
Katana Zirconia	Zirconia	200218	ZrO_2 94.4%, Y_2O_3 5.4%	Kuraray Noritake Dental Inc., Tokyo, Japan
Indirect composite material				
Ceramage Concentrate GUM-D		011008	UDMA	Shofu Inc., Kyoto, Japan
High-flow bonding agent				
Ceramage Pre-Opaque		021029	UDMA, HEMA	Shofu Inc.
Ceramage Opaque GUM-O		061008	UDMA, HEMA	
Priming agents				
Alloy Primer	ALP	00354A	MDP, VTD, acetone	Kuraray Noritake Dental Inc.
Clearfil Photo Bond	CPB	00423B	Catalyst; MDP, HEMA, bis-GMA Universal; accelerators, ethanol	Kuraray Noritake Dental Inc.
Clearfil Porcelain Bond Activator	Activator	2273	3-TMSPMA	Kuraray Noritake Dental Inc.
Estenia Opaque Primer	EOP	00172A	MDP, Solvent	Kuraray Noritake Dental Inc.
Metal Link	MLP	061046	MDDT, MHPA, Acetone	Shofu Inc.
V-Primer	VPR	TT1	VTD, Acetone	Sun Medical Co., Ltd., Moriyama, Japan

UDMA, 1,6-bis(2-methacryloyl-oxyethoxycarbonylamino)-2,4,4-trimethylhexane; HEMA, 2-hydroxyetheyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; VTD, 6-(4-vinylbenzyl-*n*-propyl)amino-1,3,5-triazine-2,4-dithione-dithiol tautomer; bis-GMA, adduct of bisphenol A and glycidyl methacrylate; 3-TMSPMA, 3-trimethoxysilylpropyl methacrylate; MDDT, 10-methacryloxydecyl 6,8-dithiooctanoate; MHPA, 6-methacryloxyhexyl phosphonoacetate.

restricts implant placement [10,11]. Surgical reconstructive procedures can improve hard- and soft-tissue defects before implant placement [10,11]; however, such procedures can be painful and unpredictable, may increase cost and healing time and may not be feasible for all patients [12]. In addition, preservation of reconstructed soft architecture around implants remains challenging. If a patient does not desire additional surgical treatment, conservative prosthodontic treatment options must be considered. Techniques to replicate soft-tissue defects around implant-supported prostheses include using a tissue-colored facade of autopolymerizing acrylic resin [13,14], flexible silicone material [15] or gingiva-colored porcelain [16,17]. These gingival prostheses have several advantages, e.g. they can provide lip support, ensure gingival symmetry and are more predictable [17].

Gingiva-colored porcelain is widely used for replicating soft-tissue defects, as it provides superior esthetics and maintains continuity of materials within the prosthesis [17]. However, gingiva-colored, porcelain-layered, zirconia-based, implant-supported prostheses have several limitations, including minor fracture of layering porcelain, weight, limited reparability and unsatisfactory long-term clinical outcomes. In recent years, a gingiva-colored indirect composite material has been used to replace lost tissue architecture [17,18]. Koizuka et al. [19] evaluated the bond strength of a gingiva-colored indirect composite to three different implant framework materials (gold alloy, titanium and zirconia ceramics) and the effect of surface pre-treatment with four priming agents. The results showed that bond strength for each framework material was influenced by the type of priming agent used. However, there are limited data on the durability of bonds between gingiva-colored indirect composite and implant framework materials.

The purpose of the present study was to evaluate the shear bond strength of a gingiva-colored indirect composite material to three different implant framework materials (titanium, gold alloy and zirconia ceramics) after thermocycling (20 000 thermocycles) and verify

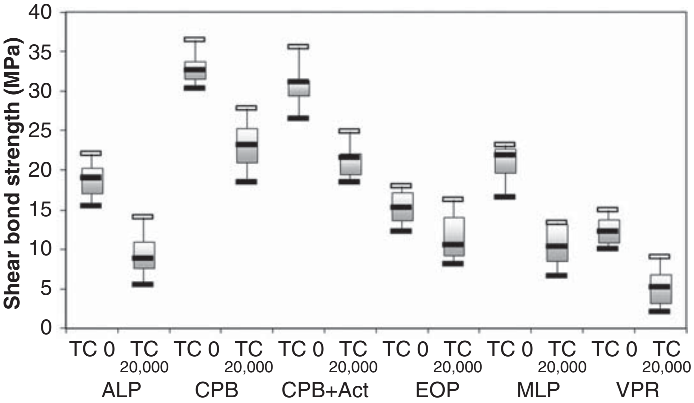

Figure 1. Box plots of shear bond strength of gingiva-colored indirect composite to CP-Ti. Line in each box represents median value. TC 0: 0 thermocycles. TC 20 000: 20 000 thermocycles.

the effect of surface pre-treatment for each framework material. The following null hypotheses were tested: (1) artificial aging with thermocycling does not influence shear bond strength of a gingiva-colored indirect composite to titanium, gold-alloy or zirconia ceramics, (2) there is no difference in bond strength among the six priming agents and (3) the bond strength of implant framework materials is equal.

Materials and methods

Specimen preparation

The materials assessed in the present study are listed in Table I. Three different framework materials for implant-supported restorations were used as adherend materials: a grade 2 commercially pure titanium (CP-Ti; Ti Target, Japan Metal Service Co. Ltd., Saitama, Japan), an American Dental Association (ADA) type 4 casting gold alloy (Type IV; Casting Gold M.C. Type IV, GC Corp., Tokyo, Japan) and zirconia ceramics (Zirconia; Katana Zirconia, Kuraray Noritake Dental Inc., Tokyo, Japan).

Six priming agents were evaluated as bonding promoters: Alloy Primer (ALP; Kuraray Noritake Dental Inc.), Clearfil Photo Bond (CPB; Kuraray Noritake Dental Inc.), Clearfil Photo Bond with Clearfil Porcelain Bond Activator (CPB+Activator; Kuraray Noritake Dental Inc.), Estenia Opaque Primer (EOP; Kuraray Noritake Dental Inc.), Metal Link (MLP; Shofu Inc., Kyoto, Japan) and V-Primer (VPR; Sun Medical Co. Ltd., Moriyama, Japan) (Table I). All priming agents contained at least one adhesive functional monomer. The functional monomers were 10-methacryloyloxydecyl dihydrogen phosphate (MDP) for ALP, CPB and EOP; 10-methacryloxydecyl 6,8-dithiooctanoate (MDDT) with 6-methacryloxyhexyl phosphonoacetate (MHPA) for MLP; and 6-(4-vinylbenzyl-*n*-propyl)amino-1,3,5-triazine-2,4-dithione-dithiol tautomer (VTD) for ALP and VPR. VPR was developed for conditioning noble metal alloys, whereas ALP, EOP and MLP were designed for priming base metal alloys. CPB+Activator is marketed for treating silica-based ceramics and CPB was originally designed for bonding enamel. A gingiva-colored indirect composite material (Ceramage Concentrate GUM-D, Shofu Inc.) was used as the layering material. Two opaque materials (Ceramage Pre-Opaque, Shofu Inc. and Ceramage Opaque GUM-O, Shofu Inc.) were used as high-flow bonding agents.

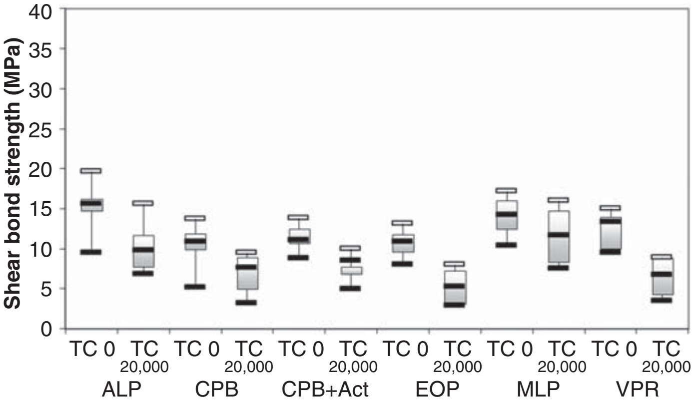

Figure 2. Box plots of shear bond strengths of gingiva-colored indirect composite to Type IV. Line in each box represents median value. TC 0: 0 thermocycles. TC 20 000: 20 000 thermocycles.

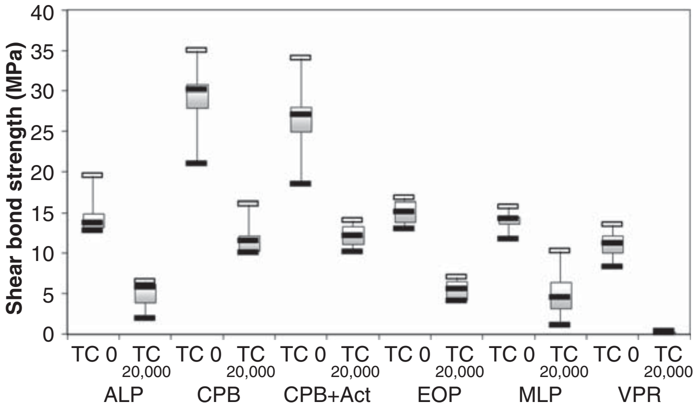

Figure 3. Box plots of shear bond strengths of gingiva-colored indirect composite to Zirconia. Line in each box represents median value. TC 0: 0 thermocycles. TC 20 000: 20 000 thermocycles.

A total of 96 disk specimens were fabricated in each framework material. CP-Ti disk specimens (10 × 2.5 mm) were fabricated from commercially pure titanium (Ti Target) by the manufacturer. Type IV specimens (10 × 2.5 mm) were made from type 4 casting gold alloy (Casting Gold M.C. Type IV). Plastic disks were cut from acrylic resin sticks (Kanki Kakouzai Corp, Tokyo, Japan), using a precision saw (IsoMet low-speed saw; Buehler Ltd, Lake Bluff, IL). The plastic patterns were invested in cristobalite investment material (Surevest, GC Corp.) and cast using a centrifugal casting apparatus (Argoncaster-C, Shofu Inc.). Zirconia disk specimens (11 × 2.5 mm) were fabricated with the Katana system (Kuraray Noritake Dental Inc.) by the manufacturer. The Zirconia disks were milled with a machine (Katana H-18; Kuraray Noritake Dental Inc.) and sintered to full density in a heat furnace (Katana F-1; Kuraray Noritake Dental Inc.) at 1 400°C for 90 min.

The surfaces of all specimens were wet-ground with 600-grit silicon carbide abrasive paper (Tri-M-ite Wetordry sheets, 3M ESPE, St. Paul, MN) and ultrasonically (SUC-110, Shofu Inc.) cleaned in acetone for 10 min. CP-Ti and Type IV specimens were abraded with airborne 50-μm aluminum oxide particles (Hi-Alumina, Shofu Inc.) at a pressure of 0.5 MPa from a distance of 1.0 cm for 10 s using a fine-airborne-particle abrasion unit (Jet Blast II, J. Morita Corp., Suita, Japan). The surfaces of Zirconia specimens were airborne-particle–abraded at a pressure of 0.2 MPa from a distance of 1.0 cm for 10 s.

All specimens in each group were randomly subdivided into six groups (n = 16) and assigned a priming agent, i.e. ALP, CPB, CPB+Activator, EOP, MLP or VPR. A piece of double-faced tape with an inner diameter of 5 mm (Star Traper-GP, Sakurai Co., Tokyo, Japan) was placed on each specimen to define the bonding area. The six primers were then applied to specimen surfaces according to the manufacturers' instructions. The specimens were thinly layered with an opaque material (Ceramage Pre-Opaque) and light-cured for 60 s using a laboratory light-polymerization unit (Solidilite, Shofu Inc.). Another opaque material (Ceramage Opaque GUM-O) was additionally applied on the primary layered opaque material and light-cured with the same light-polymerization unit for 3 min. Stainless steel rings (SUS303, Nagata Industry Co. Ltd., Sagamihara, Japan) with an inner diameter of 6 mm and a height of 2 mm were mounted so that they surrounded the opaque layered surface in all specimens. The rings were filled with a gingiva-colored indirect composite material (Ceramage Concentrate GUM-D) at a standardized force of 5 N. The specimens were then light-polymerized (Solidilite) for 5 min. After 30 min, the specimens were stored in distilled water at 37°C for 24 h. This state was defined as 0 thermocycles. Half (n = 8) of the specimens were subjected to shear bond testing at this stage. The remaining eight specimens per group were tested after they were artificially aged by thermocycling with 20 000 cycles at 5°C and 55°C, at a dwell time of 1 min per bath (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co. Ltd., Tokyo, Japan).

Shear bond testing

Each specimen was placed in a shear-bond–testing jig (ISO/TR 11405, Tokyo Giken Inc., Tokyo, Japan). A universal testing machine (Instron model 5567, Instron Corp., Norwood, MA) was used to determine shear bond strength with a 5-kN load cell at a cross-head speed of 0.5 mm/min. To convert load values into megapascals (MPa), failure load (N) was divided by bonding area (mm^2).

Statistical analysis

The data were primarily analyzed with the Levene test (SPSS version 15.0, SPSS, Inc., Chicago, IL) to evaluate equality of variance. The results were then analyzed with the Kruskal-Wallis test (SPSS version 15.0, SPSS, Inc.) followed by the Steel-Dwass test for multiple comparisons (Kyplot 5.0, KyensLab, Tokyo, Japan) to compare differences among the six priming groups before and after thermocycling. Differences between pre- and post-thermocycling bond strengths for the same priming condition were analyzed with the Mann-Whitney U-test (SPSS version 15.0, SPSS, Inc.). The significance level was set at 0.05 for all analyses.

Observation of failure

To assess mode of failure, specimen surfaces were examined with an optical microscope (Stemi DV4, Carl Zeiss MicroImaging GmbH, Göttingen, Germany) at ×32 magnification after shear bond testing. Failure mode was classified into three categories: (A) adhesive failure at the interface of the indirect composite material and substrate material, (AC) combined adhesive/cohesive failure and (C) cohesive failure within the indirect composite material. Randomly selected specimens were sputter-coated with osmium (HPC-IS, Vacuum Device Inc., Mito, Japan) for 30 s and observed under a scanning electron microscope (SEM; S-4300, Hitachi High-Technologies Co. Ltd., Tokyo, Japan) at an accelerating voltage of 15 kV.

Results

The results of shear bond strength testing before and after thermocycling are summarized in Table II and Figures 1, 2, and 3. For the CP-Ti and Zirconia substrate materials, specimens primed with CPB or CPB+Activator had significantly higher bond strengths ($p < 0.05$) than the other groups at 0 and 20 000 thermocycles. There was no significant difference between the CPB and CPB+Activator primers for the CP-Ti and Zirconia substrate materials. In contrast, the VPR group had the lowest bond strengths ($p < 0.01$) at 0 and 20 000 thermocycles for the CP-Ti and Zirconia substrate materials. For the Type IV substrate material, specimens primed with ALP or MLP had significantly higher bond strengths ($p < 0.05$) than the other groups before and after thermocycling. The post-thermocycling shear bond strengths of CP-Ti substrate were significantly higher ($p < 0.01$) than those of the other two substrate materials for specimens primed with CPB, CPB +Activator or EOP. For the CP-Ti and Type IV substrates, specimens primed with ALP, MLP or VPR had significantly higher post-thermocycling bond strengths ($p < 0.05$) as compared with Zirconia substrate.

The results of the Mann-Whitney U-test for intragroup comparison of pre- and post-thermocycling values are shown in Table III. Bond strength was significantly ($p < 0.05$) lower after thermocycling, except in Type IV specimens primed with CPB ($p = 0.092$) or MLP ($p = 0.112$).

The results of failure mode assessment after shear bond testing are summarized in Table IV. The frequency of combined adhesive/cohesive failure was

Table II. Shear bond strengths (MPa) of gingiva-colored indirect composite to three materials at 0 and 20 000 thermocycles.

	CP-Ti			Type IV			Zirconia		
Substrate	Median	Mean (SD)	Category[a,b]	Median	Mean (SD)	Category[a,b]	Median	Mean (SD)	Category[a,b]
0 thermocycle									
ALP	19.0*	18.6 (2.2)*	a, A	15.6*	15.3 (2.8)*	d, B	13.7*	14.5 (2.3)*	f, B
CPB	32.6	32.8 (2.0)	b, C	10.9	10.6 (2.6)	e, D	30.1	29.1 (2.8)	g, C
CPB+Activator	31.1	30.4 (2.6)	b, E	11.0	11.5 (1.6)	e, F	27.1	26.5 (3.2)	g, E
EOP	15.3*	15.3 (2.2)*	a, G	10.9*	10.6 (1.8)*	e, H	15.1*	15.1 (1.4)*	f, G
MLP	21.8*	20.8 (2.4)*	a, I	14.2*	14.0 (2.4)*	d, J	14.2*	13.9 (1.4)*	f, J
VPR	12.2*	12.3 (1.7)*	c, K	13.3*	12.5 (2.0)*	e, K	11.1*	11.1 (1.5)*	h, K
20 000 thermocycles									
ALP	8.8	9.4 (3.5)	i, L	9.8	10.3 (3.3)	l, L	5.7	5.0 (1.8)	n, M
CPB	23.1	23.1 (3.2)	j, N	7.6	6.5 (2.6)	m, O	11.5	11.9 (2.0)	o, P
CPB+Activator	21.5	21.3 (1.9)	j, Q	7.7	7.3 (1.5)	m, R	12.1	12.1 (1.5)	o, S
EOP	10.5	11.6 (3.1)	i, T	5.2	5.7 (2.3)	m, U	5.5	5.2 (1.3)	n, U
MLP	10.3	10.3 (2.5)	i, V	11.6	11.3 (3.6)	l, V	4.5	4.9 (2.9)	n, W
VPR	5.2	5.1 (2.5)	k, X	6.7	6.5 (2.4)	m, X	0.3	0.2 (0.1)	p, Y

See Table I and text for description of group abbreviations.

* Data are from Koizuka et al. [19].

[a] Identical lower-case letters indicate that the values do not significantly differ with respect to the primer used ($p > 0.05$; Steel-Dwass test).

[b] Identical upper-case letters indicate that the values do not significantly differ with respect to the substrate material used ($p > 0.05$; Steel-Dwass test).

Table III. Results of analysis with the Mann-Whitney U-test.

Substrate		p-value	Difference between 0 and 20 000 thermocycles
CP-Ti	ALP	0.004	Significant
	CPB	0.001	Significant
	CPB+Activator	0.001	Significant
	EOP	0.021	Significant
	MLP	0.001	Significant
	VPR	0.001	Significant
Type IV	ALP	0.007	Significant
	CPB	0.092	Not significant
	CPB+Activator	0.001	Significant
	EOP	0.001	Significant
	MLP	0.112	Not significant
	VPR	0.001	Significant
Zirconia	ALP	0.001	Significant
	CPB	0.001	Significant
	CPB+Activator	0.001	Significant
	EOP	0.001	Significant
	MLP	0.001	Significant
	VPR	0.000	Significant

See Table I and text for description of group abbreviations. Significant difference is defined as $p < 0.05$.

substantially lower after thermocycling. After 20 000 thermocycles, such failures were seen in CP-Ti and Zirconia materials primed with CPB, CPB+Activator or EOP. Complete cohesive failure within the indirect composite material was not observed in any specimen.

Figures 4, 5, and 6 show representative SEM images of specimen surfaces of the three substrate materials after airborne-particle abrasion. Surfaces of CP-Ti (Figure 4) and Type IV (Figure 5) specimens were rougher and had sharper edges and undercuts than the zirconia surface (Figure 6). Figures 7 and 8 show representative SEM images of the fracture interface after shear bond testing. At 0 thermocycles, a Zirconia specimen primed with VPR shows adhesive failure (Figure 7). At 20 000 thermocycles, a CP-Ti specimen primed with CPB shows combined adhesive/cohesive failure (Figure 8).

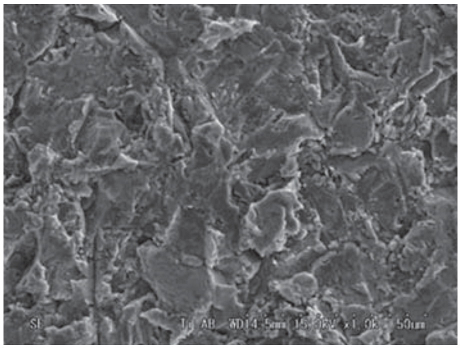

Figure 4. SEM image of CP-Ti surface airborne-particle abraded with airborne Al_2O_3 particles (original magnification ×1000). This figure is reproduced with permission from Koizuka et al. [19].

Discussion

This *in vitro* study assessed shear bond strength of a gingiva-colored indirect composite to three implant framework materials. Artificial aging by thermocycling resulted in lower shear bond strengths as compared with no thermocycling. Thus, the first null hypothesis, i.e. that artificial aging with thermocycling does not influence shear bond strength of gingiva-colored indirect composite to the three implant framework materials, was substantially rejected. The second and third null hypotheses are rejected, as there were differences in shear bond strength among the six priming agents and three implant framework materials.

Thermocycling and water storage are frequently used in dental research as clinically relevant conditions for testing durability of adhesion [20]. In addition to thermal stress, a variety of factors have a clinical effect on bond durability, such as mechanical stress, hydrolytic stability, water diffusion into the bonded interface and corrosion of substrate materials [21,22]. Therefore, thermocycling was used in the present study to investigate hydrolytic stability and thermal stress on bonding between indirect composite and zirconia ceramics. Shear bond strength was significantly reduced with thermocycling, except in Type IV specimens primed with CPB or MLP. These results confirm the findings of a previous study, which found a decrease in bond strength of composite materials to titanium and gold alloy after thermocycling [23–26]. A few *in vitro* studies evaluated the durability of bond strength of indirect composite to zirconia ceramics [27]. Komine et al. [27] reported that thermocycling did not decrease bond strength in specimens of zirconia ceramics surface-treated with priming agents containing MDP. These conflicting results are mainly due to differences in the indirect composite materials that were assessed in the two studies; Estenia C&B (Kuraray Noritake Dental Inc.) was evaluated in the earlier study [27]. The authors speculated that polymerization of Estenia composite material was ongoing at 24 h and that thermal stress at 55°C resulted in additional polymerization of the composite material [27]. Thus, different types of indirect composites may have varying effects on the durability of bond strength to zirconia ceramic frameworks.

Failure mode analysis (Table IV) showed that most specimens suffered adhesive failure after thermocycling. Indeed, the fact that bond strength was lower and adhesive failure was more frequent after

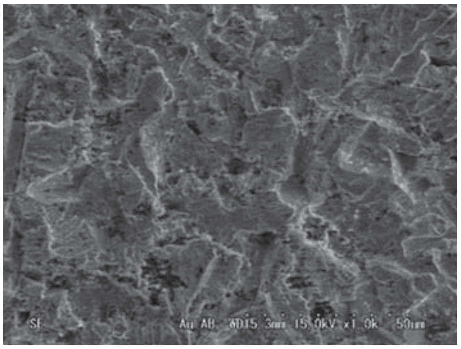

Figure 5. SEM image of Type IV surface airborne-particle abraded with airborne Al_2O_3 particles (original magnification ×1000). This figure is reproduced with permission from Koizuka et al. [19].

thermocycling suggests that the bonding interface was negatively affected by thermocycling. Therefore, thermocycling was clearly an effective means to evaluate adhesive durability of the gingiva-colored composite material to the substrate material-bonded specimens.

For the CP-Ti and Zirconia substrate materials, specimens primed with CPB or CPB+Activator had significantly higher bond strengths than the other groups, before and after thermocycling. These results indicate that application of hydrophobic phosphate monomer (MDP) and polymerization initiator is effective with respect to initial and durable bond strength of gingiva-colored indirect composite to CP-Ti and Zirconia substrate materials. Our findings agree with those of previous studies that investigated the efficacy of hydrophobic phosphate monomer (MDP) and polymerization initiators bond between titanium [23] or zirconia [27,28] and composite material. For the Type IV substrate material, specimens primed with ALP or MLP had significantly higher bond strengths than the other groups, before and after thermocycling. The combined use of a thione monomer and phosphoric monomer appears to have a synergetic effect when layering the gingiva-colored indirect composite over type IV gold alloy, due to the subsistence of pure metal elements and metal oxides on the surface [29]. In the present study, in Type IV specimens, the combined use of a thione monomer (VTD or MDDT) and phosphoric monomer (MDP or MHPA) resulted in greater bond strength as compared with the use of only MDP or VTD, which is consistent with the findings of an earlier study [29].

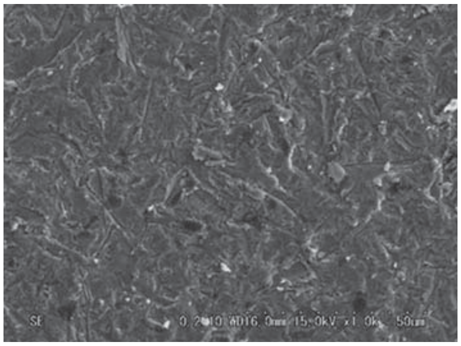

Figure 6. SEM image of Zirconia surface airborne-particle abraded with airborne Al_2O_3 particles (original magnification ×1000). This figure is reproduced with permission from Koizuka et al. [19].

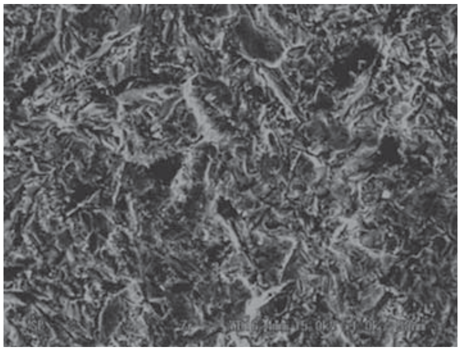

Figure 7. Pre-thermocycling SEM image after shear bond testing of Zirconia surface from VPR group (original magnification ×1000).

Few studies have compared the bond strength of commercially pure titanium, type IV casting gold alloy and zirconia ceramics to composite material. After thermocycling, shear bond strengths with the CP-Ti substrate were significantly higher than those with Type IV and Zirconia substrate materials, when primed with CPB, CPB+Activator or EOP. This indicates that, as compared with the other substrates, the CP-Ti substrate may result in more-durable bonds to gingiva-colored composite when the substrate surface is treated with a hydrophobic phosphate monomer (MDP). As mentioned above, application of a hydrophobic phosphate monomer (MDP) and polymerization initiator resulted in better bond strengths between the gingiva-colored indirect composite and the CP-Ti and Zirconia substrate materials. Therefore, we speculate that bonding between the phosphoric monomer and oxides on the surface of the CP-Ti substrate is stronger than that between the phosphoric monomer and oxides of the Zirconia substrate.

For the Zirconia substrate, priming with CPB or CPB+Activator resulted in post-thermocycling shear bond strengths > 10 MPa, which is the suggested

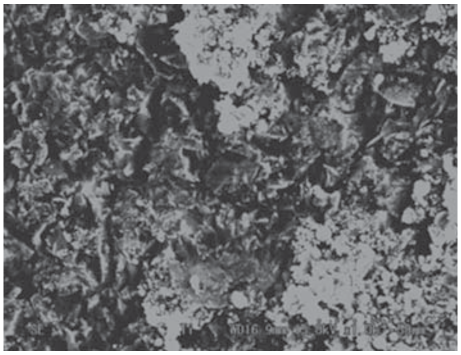

Figure 8. Post-thermocycling SEM image after shear bond testing of CP-Ti surface from CPB group (original magnification ×1000).

Table IV. Failure modes after shear bond testing.

		0 thermocycle			20 000 thermocycles		
Substrate	Primer	A	AC	C	A	AC	C
CP-Ti	ALP	7	2	0	9	0	0
	CPB	6	3	0	7	2	0
	CPB+Activator	6	3	0	8	1	0
	EOP	9	0	0	7	2	0
	MLP	7	2	0	9	0	0
	VPR	9	0	0	9	0	0
Type IV	ALP	7	2	0	9	0	0
	CPB	9	0	0	9	0	0
	CPB+Activator	9	0	0	9	0	0
	EOP	9	0	0	9	0	0
	MLP	9	0	0	9	0	0
	VPR	9	0	0	9	0	0
Zirconia	ALP	9	0	0	9	0	0
	CPB	6	3	0	7	2	0
	CPB+Activator	8	1	0	7	2	0
	EOP	9	0	0	9	0	0
	MLP	9	0	0	9	0	0
	VPR	9	0	0	9	0	0

See Table I and text for description of group abbreviations.
A, Adhesive failure at interface of indirect composite material and substrate material; AC, combined adhesive/cohesive failure; C, cohesive failure within the indirect composite material.

threshold for clinically successful composite–ceramic bonds [30,31]. An indirect composite veneered restoration is exposed to occlusal forces that induce shear stresses. However, a gingival prosthesis to replace gingival defects would not be directly exposed to such forces. Therefore, the gingiva-colored indirect composite material might be a promising alternative layering material for zirconia-based, implant-supported restorations.

In clinical procedures, mechanical retention of the layered surface is ensured by airborne-particle abrasion and by placement of retention beads before indirect composite materials are layered. However, retention beads cannot be placed on the surface of zirconia ceramics. Therefore, comparison and assessment of shear bond strength of CP-Ti, Type IV and Zirconia substrates are limited by the present experimental conditions. In addition, it should be noted that the clinical long-term stability of bonding between gingiva-colored indirect composite and these three framework materials is not known. Further clinical study of these materials is necessary.

In conclusion, the shear bond strength of a gingiva-colored indirect composite to commercially pure titanium, type IV casting gold alloy and zirconia ceramics in implant framework materials was generally lower after thermocycling. Application of a hydrophobic phosphate monomer (MDP) and polymerization initiator was effective with respect to durable bond strength of the gingiva-colored indirect composite to commercially pure titanium and zirconia ceramics. For type IV casting gold alloy, combined use of a thione monomer (VTD or MDDT) and phosphoric monomer (MDP or MHPA) resulted in better durable bond strengths than did use of only MDP or VTD. Bond strength varied according to the framework material used.

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