Original

Effect of surface treatment on bond strength between an indirect composite material and a zirconia framework

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Abstract: The present study evaluated the effect of various surface treatments for zirconia ceramics on shear bond strength between an indirect composite material and zirconia ceramics. In addition, we investigated the durability of shear bond strength by using artificial aging (20,000 thermocycles). A total of 176 Katana zirconia disks were randomly divided into eight groups according to surface treatment, as follows: group CON (as-milled); group GRD (wet-ground with 600-grit silicon carbide abrasive paper); groups 0.05, 0.1, 0.2, 0.4, and 0.6 MPa (airborne-particle abrasion at 0.05, 0.1, 0.2, 0.4, and 0.6 MPa, respectively); and group HF (9.5% hydrofluoric acid etching). Shear bond strength was measured at 0 thermocycles in half the specimens after 24-h immersion. The remaining specimens were subjected to 20,000 thermocycles before shear bond strength testing. Among the eight groups, the 0.1, 0.2, 0.4, and 0.6 MPa airborne-particle abraded groups had significantly higher bond strengths before and after thermocycling. The Mann-Whitney *U*-test revealed no significant difference in shear bond strength between 0 and 20,000 thermocycles, except in the 0.2 MPa group (*P* = 0.013). From the results of this study, use of airborne-particle abrasion at a pressure of 0.1 MPa or higher increases initial and durable bond strength between an indirect composite material and zirconia ceramics. (J Oral Sci 54, 39-46, 2011)

Keywords: airborne-particle abrasion; indirect composite; shear bond strength; zirconia.

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Introduction

In recent years, increasing demand for all-ceramic restorations has led to development of ceramic materials with optimized mechanical properties, such as densely sintered aluminum oxide and zirconium dioxide (zirconia) ceramics. The inherent esthetic and functional advantages of these materials have broadened the range of clinical indications (1,2). With the maturation of computer-aided design/computer-assisted manufacture (CAD/CAM) technology, zirconia ceramics have been used as a superstructure for complete-coverage single crowns and fixed partial dentures (FPDs) (3). A number of clinical studies of zirconia-based all-ceramic restorations have confirmed their high stability as a framework material (4-8). However, high incidences of chipping of veneering porcelain and cohesive fracture of veneering ceramic have been reported (4-8).

Various explanations for the porcelain chipping and fracture have been offered, including mismatch of the thermal expansion coefficient between veneering porcelain and zirconia ceramics (9,10), the thickness of the veneering porcelain (11), surface defects or improper support by the framework (12), low fracture toughness

Table 1 Materials assessed in the present study

Material/Trade name	Manufacturer	Lot no.	Components
Zirconia ceramics			
Katana	Noritake Dental Supply Co., Ltd., Miyoshi, Japan		94.4% ZrO_2, 5.4% Y_2O_3
Indirect composite material			
Estenia C&B DA2	Kuraray Medical Inc., Tokyo, Japan	0040CA	UTMA, methacrylate, photoinitiator, pigment, 92% filler (glass, macro alumina)
Opaque material			
Estenia C&B OA2	Kuraray Medical Inc.	00043B	Bis-GMA, methacrylate, photoinitiator, pigment, filler (quartz, composite, others)
Alumina powder			
Hi-Aluminas	Shofu Inc., Kyoto, Japan		50-μm grains sized alumina
Hydrofluoric acid			
Porcelain Etch	Ultradent, South Jordan, UT, USA	B44V3	9.5% hydrofluoric acid gel
Priming agent			
Estenia Opaque Primer	Kuraray Medical Inc.	00157A	MDP, monomer solvent

UTMA: Urethane tetramethacrylate; Bis-GMA: bisphenol A-glycidyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate.

of the veneering porcelain (13), and low thermal conductivity of zirconia (11). Some attempts to address these deficiencies have been introduced. These include the over-pressing technique (in which heat-pressed ceramic is layered onto the zirconia framework) (13) and the use of an improved customized zirconia-coping design that adds bulk to the substructure to provide adequate support for the veneering porcelain (12). However, chipping of veneering porcelains continues to be reported in clinical studies of over-pressing technique for zirconia-based FPDs (14,15) and in investigations of zirconia-based FPDs with modified framework designs (16).

Although feldspathic porcelain is acceptable for use in veneering for zirconia frameworks, a highly loaded indirect composite material is an alternative to feldspathic porcelain. Two in vitro studies investigated short-term bond strength between an indirect composite used as veneering material and zirconia ceramics in zirconia-based restorations (17,18). Kobayashi et al. (17) reported that application of priming agents containing MDP and an intermediate high-flow bonding agent resulted in superior bond strength between an indirect composite and zirconia ceramics. Komine et al. (18) proposed that the use of an acidic functional monomer containing carboxylic anhydride (4-META), phosphonic acid (6-MHPA), or phosphate monomer (MDP) can yield durable bond strength between an indirect composite and zirconia ceramics.

Bond strength between resin cements and ceramics might be improved by the presence of micromechanical retentions because creation of roughened ceramic surfaces may allow resin cement to penetrate and flow into such microretentions, thereby creating a stronger micromechanical interlock (19-22). Before the bonding procedure, one or more surface treatments of the intaglio surface of ceramic restorations are typically performed. In particular, airborne-particle abrasion with aluminum oxide particles is a common method for achieving strong adhesion. A number of studies have indicated that durable long-term bonds between resins and high-strength ceramic materials can be achieved by surface treatment with airborne-particle abrasion using aluminum oxide particles and the use of a modified priming and/or resin composite luting agent that contains MDP (23-26). However, airborne-particle abrasion with alumina has been reported to damage the surface integrity of high-strength ceramics (27,28). Therefore, reducing the pressure of airborne-particle abrasion or omitting airborne-particle abrasion entirely might improve the mechanical properties of zirconia ceramics. Indeed, Kern et al. (29) demonstrated that low-pressure airborne-particle abrasion at 0.05 MPa provided sufficient surface conditioning.

To date, few studies have assessed the effect of surface treatments on the bond strength of indirect composite material as a veneering material to a zirconia framework. Therefore, the present study evaluated the effect of various surface treatments for zirconia ceramics on shear bond strength between an indirect composite material and zirconia ceramics. In addition, we investigated the durability of shear bond strength after thermocycling (20,000 cycles). The following null hypotheses were tested: that there would be no difference in shear bond strength between an indirect composite material and zirconia ceramics after a variety of surface treatments, and that artificial aging with thermocycling would not affect bond strength.

Table 2 Shear bond strength (MPa) of an indirect composite material to zirconia ceramic after 0 and 20,000 thermocycles

Groups	0 thermocycle			20,000 thermocycles			Difference between 0 and 20,000 thermocycles**	*P* value
	Median	Mean (SD)	Category*	Median	Mean (SD)	Category*		
CON	0.3	0.4 (0.2)	a	0.3	0.3 (0.2)	d	Not significant	0.401
HF	0.1	0.1 (0.1)	a	0.1	0.2 (0.2)	d	Not significant	0.949
GRD	4.5	5.0 (2.3)	b	7.6	6.9 (3.1)	e	Not significant	0.080
0.05 MPa	7.4	7.3 (3.4)	b	9.9	9.9 (2.9)	e	Not significant	0.133
0.1 MPa	12.5	12.8 (2.8)	c	15.0	14.3 (2.2)	f	Not Significant	0.101
0.2 MPa	13.1	13.2 (2.8)	c	17.0	17.4 (4.4)	f	Significant	0.013
0.4 MPa	14.5	14.2 (2.7)	c	16.8	17.6 (4.6)	f	Not significant	0.116
0.6 MPa	13.8	14.2 (1.9)	c	15.9	16.7 (4.2)	f	Not significant	0.217

Group CON: as-milled; Group HF: acid-etched with 9.5% hydrofluoric acid gel; Group GRD: wet-ground with 600-grit silicon carbide abrasive paper; Groups 0.05, 0.1, 0.2, 0.4, and 0.6 MPa: airborne-particle abraded at 0.05, 0.1, 0.2, 0.4, and 0.6 MPa pressure, respectively.
*Identical letters indicate a nonsignificant difference (Steel-Dwass test; *P* > 0.05).
**Significant difference (*P* < 0.05) between 0 and 20,000 thermocycles on Mann-Whitney *U*-test.

Materials and Methods

The materials assessed in the present study are listed in Table 1. A total of 176 zirconia disks (11 mm in diameter × 2.5 mm in thickness) were fabricated with Katana Zirconia (Noritake Dental Supply Co. Ltd., Miyoshi, Japan) as substrates for bonding. An indirect composite material (Estenia C&B Dentin DA2, Kuraray Medical Inc., Tokyo, Japan) was applied as a veneering material to the zirconia, and an opaque material (Estenia C&B Opaque OA2, Kuraray Medical Inc.) was used as a high-flow bonding agent. Estenia Opaque Primer (EOP; Kuraray Medical Inc.) was applied as a bonding promoter. The zirconia specimens were randomly divided into eight groups ($n = 22$) according to surface treatment, as follows: Group CON (specimens with the original as-milled ceramic surface, ie, without surface treatment), Group GRD (specimens that were wet-ground with 600-grit silicon carbide abrasive paper), Groups 0.05, 0.1, 0.2, 0.4, and 0.6 MPa (specimens that were airborne-particle abraded using 50-μm alumina (Hi-Aluminas, Shofu Inc., Kyoto, Japan) at 0.05, 0.1, 0.2, 0.4, and 0.6 MPa pressure, respectively, for 20 s at a distance of 10 mm from the surface), and Group HF (specimens that were acid-etched with 9.5% hydrofluoric acid gel (Porcelain Etch, Ultradent, South Jordan, UT, USA) on the milled surface 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, Japan)).

After surface preparation of the specimens, a piece of double-coated tape with a circular hole 5.0 mm in diameter was positioned on each zirconia disk to define the area of bonding. EOP was then applied to the surface of the specimens, according to the manufacturer's instruction, before bonding of opaque material to the zirconia disks. For all specimens in each group, a thin layer of opaque material (Estenia C&B Opaque OA2) was applied and light-polymerized for 90 s using 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 steel ring (6.0 mm in inside diameter, 2.0 mm in depth) was then positioned to surround the opaque resin layers. The ring was filled with a dentin shade of indirect composite material (Estenia C&B Dentin DA2) at a standardized force of 5 N. The specimen was light-polymerized in the polymerization unit for 5 min and finalized in a heat oven (KL-310, J. Morita Corp.) at 110°C for 15 min. All specimens were immersed in 37°C water for 24 h.

After 24-h immersion, shear bond strength was measured at 0 thermocycle in half the specimens. The remaining specimens were placed in a thermocycler (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co. Ltd., Tokyo, Japan) and thermocycled between 5°C and 55°C water with a dwell time of 1 min per bath for 20,000 cycles. Each specimen was positioned in a steel mold, and shear bond strength was measured using a mechanical testing device (Type 5567, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/min. The data were primarily analyzed by using Levene's test for equality of variance (SPSS version 15.0, SPSS, Inc., Chicago, IL, USA). Because homoscedasticity was not confirmed by Levene's test, the Steel-Dwass comparison (Kyplot 5.0, KyensLab, Tokyo, Japan) was performed. The Mann-Whitney *U*-test was used to evaluate the difference in bond strengths between pre- and post-thermocycling groups. The significance level was set at 0.05 for all analyses.

After shear bond testing, the fractured interfaces of

Table 3 Failure modes after shear bond testing

	0 thermocycle			20,000 thermocycles		
	A	AC	C	A	AC	C
CON	11	0	0	11	0	0
HF	11	0	0	11	0	0
GRD	11	0	0	10	1	0
0.05 MPa	11	0	0	10	1	0
0.1 MPa	11	0	0	8	3	0
0.2 MPa	11	0	0	8	3	0
0.4 MPa	9	2	0	9	2	0
0.6 MPa	9	2	0	8	3	0

Group CON: as-milled; Group HF: acid-etched with 9.5% hydrofluoric acid gel; Group GRD: wet-ground with 600-grit silicon carbide abrasive paper; Groups 0.05, 0.1, 0.2, 0.4, and 0.6 MPa: airborne-particle abraded at 0.05, 0.1, 0.2, 0.4, and 0.6 MPa pressure, respectively.
A: adhesive failure at the indirect composite material–zirconia interface;
AC: combined adhesive failure and cohesive failure within the indirect composite material;
C: cohesive failure within the indirect composite material.

specimens were examined with a stereomicroscope (StemiDV4, Carl Zeiss Co., Ltd., Jena, Germany) at original magnification ×32 to determine the mode of failure. Failure modes were classified into three categories: A, adhesive failure at the indirect composite material–zirconia interface; AC, combined adhesive failure and cohesive failure within the indirect composite material; and C, cohesive failure within the indirect composite material. Both after surface preparation and shear bond testing, representative specimens were osmium-coated with a sputter coater (HPC-IS, Vacuum Device, Mito, Japan) and observed with a scanning electron microscope (SEM; S-4300, Hitachi High-Technologies Co. Ltd., Tokyo, Japan) operated at 15 kV.

Results

Detailed bond strength values and the results of statistical analysis are shown in Table 2. Mean bond strength varied from 0.1 to 14.2 MPa without thermocycling and from 0.2 to 17.6 MPa with thermocycling. Mean bond strength was significantly lower in the CON and HF groups than in the other groups, regardless of thermocycling. Airborne-particle abrasion at 0.1, 0.2, 0.4, and 0.6 MPa resulted in significantly higher bond strengths among the eight groups, with and without thermocycling. The Mann-Whitney U-test showed no significant difference in shear bond strength after 0 and 20,000 thermocycles, except in the 0.2 MPa group ($P = 0.013$).

Table 3 shows the failure modes after shear bond testing. Without thermocycling, the 0.4 and 0.6 MPa groups exhibited combination adhesive failure and cohesive failure within the indirect composite material. After 20,000 thermocycles, combination adhesive failure and cohesive failure within the indirect composite material was observed in six groups (GRD and 0.05, 0.1, 0.2, 0.4, and 0.6 MPa). In the CON and HF specimens, all failures were completely adhesive failure at the zirconia–indirect composite interface, regardless of thermocycling.

Figures 1A–H show SEM images of the zirconia surface after different surface treatments. The as-milled zirconia surface was moderately rough, with no undercuts (Fig. 1A). An SEM image of the zirconia surface treated with HF acid (Fig. 1B) showed a very similar superficial structure to that of the as-milled surface in Fig. 1A. Figure 1C is an SEM image of a zirconia surface wet-ground with 600-grit silicon carbide paper and shows a smooth surface with scratches caused by the wet grinding. As seen in Figs. 1D–H, the zirconia surface after airborne-particle abrasion with 50-µm alumina at different pressures shows sharp cracks and surface defects. In a comparison of these SEM images, airborne-particle abrasion at 0.1, 0.2, 0.4, and 0.6 MPa (Figs. 1E–H) created a rougher surface than did airborne-particle abrasion at 0.05 MPa (Fig. 1D).

Representative SEM images of fracture interfaces after shear bond testing are shown in Figs. 2 and 3. Figure 2 shows an example of adhesive failure. The composite material is completely separated from the zirconia surface, and the as-milled surface can be seen. In contrast, in a specimen with combined cohesive and adhesive failure, the zirconia surface is partially covered with composite material and/or filler (Fig. 3).

Discussion

The present study evaluated the effect of different surface treatments on initial and durable bond strength between an indirect composite material and zirconia ceramics. The first null hypothesis, that there would be
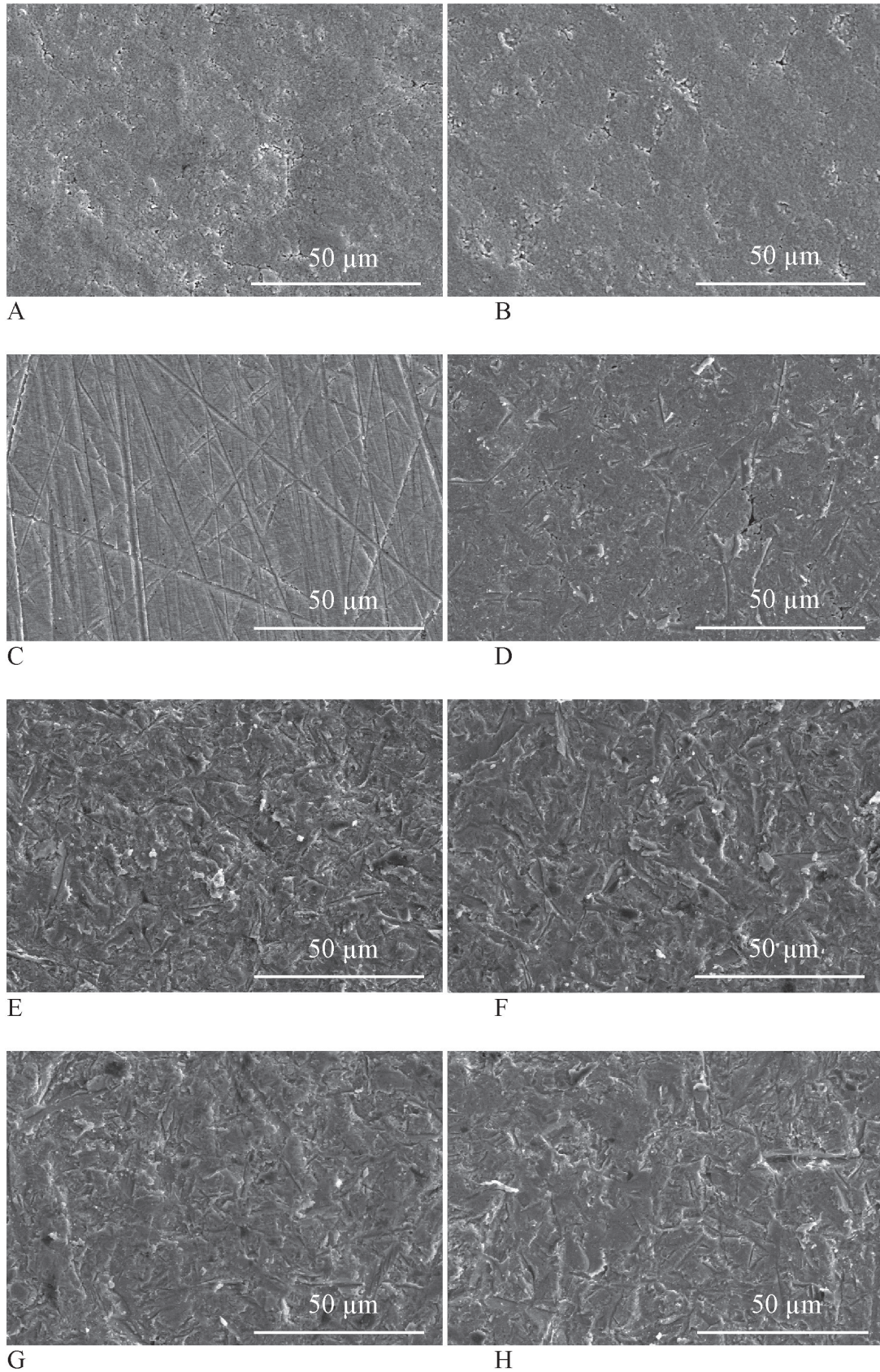
Fig. 1 SEM image of (A) zirconia surface as milled, (B) zirconia surface treated with hydrofluoric acid, (C) zirconia surface wet-ground with 600-grit silicon carbide paper, and (D E, F, G, and H) zirconia surface airborne-particle abraded at 0.05, 0.1, 0.2, 0.4, and 0.6 MPa, respectively (Original magnification ×1,000).
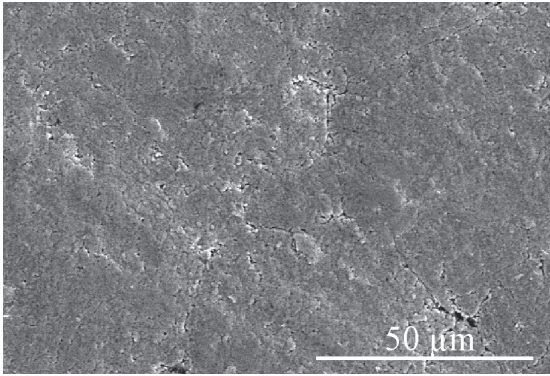
Fig. 2 SEM image of the zirconia surface of a specimen from the CON group after shear bond testing and aging with 20,000 thermocycles (Original magnification ×1,000).
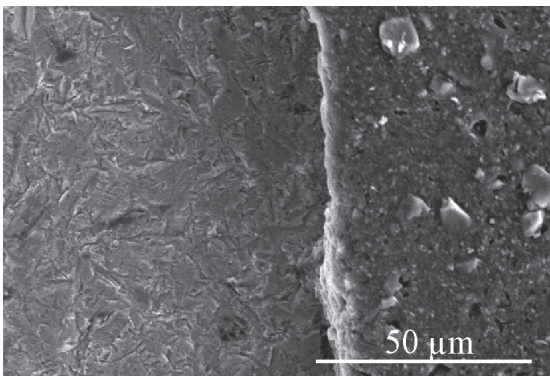

Fig. 3 SEM image of the zirconia surface of a specimen from the 0.6 MPa group after shear bond testing and aging with 20,000 thermocycles (Original magnification ×1,000).

no difference in shear bond strength between an indirect composite material and a zirconia ceramics after various surface treatments, must be rejected. The strengths of the bond between an indirect composite and zirconia ceramics were affected by the type of surface treatment in the present study. However, there was no significant difference in the shear bond strength between 0 and 20,000 thermocycles, except in the 0.2 MPa group. Thus, the results of the present study partially confirm the second null hypothesis, ie, that artificial aging by means of thermocycling would not affect bond strength between an indirect composite material and zirconia ceramics.

As shown in Table 2, specimens that underwent airborne-particle abrasion at a pressure of 0.1, 0.2, 0.4, and 0.6 MPa had significantly higher bond strengths than the other four groups, with and without thermocycling. These findings indicate that airborne-particle abrasion is necessary to achieve superior and durable bond strength between an indirect composite material and zirconia

ceramics. Previous studies of adhesion between resin cements and ceramics found that mechanical surface-conditioning with airborne-particle abrasion improved resin–ceramic bonding by increasing surface roughness and bonding area, improving the wetting behavior of adhesives, and chemically activating bonding surface by removing organic contaminants from the ceramic surface (30-32). However, in the present study, specimens that were airborne-particle abraded at 0.05 MPa had significantly lower bond strengths than did specimens abraded at other pressures. SEM images showed that abrasion at a pressure of 0.1, 0.2, 0.4, and 0.6 MPa resulted in a rougher surface as compared with a pressure of 0.05 MPa. These results demonstrate that mechanical surface-conditioning with airborne-particle abrasion at a pressure of 0.1 MPa or higher results in a more retentive surface and achieves superior and durable bond strengths between an indirect composite material and zirconia ceramics. Our findings disagree with those of a previous study showing that low-pressure airborne-particle abrasion (0.05 MPa) provided sufficient surface conditioning (29). We speculate that the disparity is due to differences in bonding materials and testing methods.

There was no significant difference in shear bond strength between the CON and HF groups regardless of thermocycling, which suggests that HF acid etching does not sufficiently enhance bond strength between an indirect composite and zirconia ceramics. It has been reported that HF acid etching was not effective for surface treatment of zirconia ceramics due to the low amount of glass phase (26). In the present study, primer with MDP was used in all surface treatment groups. However, there was no difference in adhesion between the indirect composite material and zirconia ceramics in the CON and HF groups. Thus, when airborne-particle abrasion or polishing was combined with MDP-containing primer, a superior and durable bond between the indirect composite material and zirconia ceramics was achieved. Yang et al. (31) reported that the surface activation and cleaning effects of airborne-particle abrasion were needed for chemical bonding.

The shear bond strength between the composite material and zirconia ceramics increased slightly after thermocycling in all groups except in the CON and HF groups. As shown in Table 3, combined adhesive failure and cohesive failure within the indirect composite material was observed in two groups at 0 thermocycle. However, combined adhesive failure and cohesive failure within the indirect composite material was observed in six groups that underwent thermocycling. These findings are consistent with those of a study (18) which noted that

polymerization of 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.

In conclusion, the present study verified that airborne-particle abrasion at a pressure 0.1 MPa or higher yields satisfactory initial and durable bond strengths between an indirect composite material and zirconia ceramics.

Acknowledgments

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