

Influence of air-abrasion and subsequent heat treatment on bonding between zirconia framework material and indirect composites

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The purpose of this study was to evaluate the influence of air-abrasion and subsequent heat treatment on the shear bond strength of the bond between indirect composites and a zirconia material. Four surface preparations were employed; ground flat, then heated to regenerate the crystal phase (C); air-abraded with alumina for 10 s (S10), for 20 s (S20), and air-abraded for 10 s and heated (H). Disks were primed with Alloy Primer and bonded either with Estenia or with Gradia composite. XRD analysis suggested that the monoclinic zirconia content was increased by air-abrasion, and decreased by heating. The surface roughness of S10, S20 and H disks was similar. Nevertheless, H groups showed lower bond strengths than the S10 and S20 groups both before and after thermal cycling. Although alumina air-abrasion considerably enhanced bonding between zirconia and indirect composites, subsequent heat treatment had a negative effect on the durability of bond strength.

Keywords: Zirconia, Indirect composite, Bond strength, Air-abrasion, Heat treatment

INTRODUCTION

Yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP, *i.e.*, zirconia) has been applied to post and core systems^{1,2}, implant abutments³ and coping for all-ceramic restorations⁴, because it has an esthetically appealing color, high flexural strength, long-term stability and good biocompatibility. On the other hand, conventional dental restorations may be coped with a variety of materials: metals, ceramics, composite resin or a combination of these. For zirconia materials to be more widely applied as a replacement for metal implants in restorative dentistry, it is necessary for them to be bonded not only to ceramics but also to composite veneering resins (such as required for a composite veneered zirconia crown).

In composite resin veneered prostheses, it is necessary to establish a consistent and durable bond between the veneering material and the zirconia substructure. Komine *et al.*⁵ demonstrated that the use of an acidic functional monomer containing carboxylic anhydride (4-methacryloyloxyethyl trimellitate anhydride), phosphonic acid (6-methacryloyloxyhexyl phosphonoacetate), or phosphate monomer (10-methacryloyloxydecyl dihydrogen phosphate, MDP) could yield a durable, strong bond between Estenia C&B indirect composite and Katana zirconia. However, apart from one study with similar findings⁶, there is little data on the adhesion of indirect composite resins to zirconia.

In contrast, many studies on bonding with luting cements have been reported recently. These studies have focused on the potential benefits of: 1) air-abrasion with alumina particles, 2) tribochemical coating, 3) primers

containing phosphate as adhesive promoters, and 4) resin cement containing adhesive phosphate monomer. Among these four methods, air-abrasion is supposed to roughen the zirconia, increasing the bonding area and modifying the ceramic surface energy and wettability, thus facilitating the formation of resin-zirconia micromechanical interlocks⁷⁻¹⁰. This method is effective not only for improving the bonding strength of luting cements but also in bonding to veneering porcelain^{11,12}.

However, the mechanical stress involved in air-abrasion initiates the phase transition from tetragonal to monoclinic zirconia, subsequently resulting in compressive stress¹³⁻¹⁶. Therefore, heat treatment after air-abrasion is recommended when the zirconia is to be veneered to porcelain, but no information is available about its influence on bonding between zirconia and indirect composites. Furthermore, the effects may differ for different indirect composite materials.

The aim of this study was to investigate the effects of air-abrasion and subsequent heat treatment of the zirconia on the shear bond strengths of bonds between two different indirect composites and a zirconia framework material. The tested null-hypotheses for this study were that the bond strength varies with air-abrasion and heat treatment of the zirconia surfaces and that these effects are influenced by the type of indirect composite.

MATERIALS AND METHODS

Materials used

Information on the materials is summarized in Table 1. The zirconia specimens were milled from zirconia blanks (Cercon Base, Dentsply International, York, PA, USA)

Table 1 List of materials used and their characteristics

System	Batch No.	Main composition ^a	Manufacturer
<i>Zirconia</i>			
Cercon Base	18001606	ZrO ₂ (92%), Y ₂ O ₃ (5%), HfO ₂ (2%), Al ₂ O ₃ , SiO ₂ (1%)	Dentsply International, York, PA, USA
<i>Indirect composites</i>			
Estenia C&B	OA3	00096B	Bis-GMA, methacrylic acid ester, quartz powder, organic hybrid filler
	DA3	0062AB	
Gradia	FO	907092	UDMA, silica nano powder
	OA3	906032	UDMA, silica nano powder
	DA3	911162	UDMA, inorganic-organic composite filler, silica nano powder, glass powder
<i>Primer</i>			
Alloy Primer	0345AA	VTD, MDP, acetone	Kuraray Medical Inc., Tokyo, Japan

UDMA: urethane dimethacrylate; Bis-GMA: bisphenol-A-glycidyl methacrylate; UTMA: urethane tetramethacrylate; VTD: 6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol, or -2,4-dithione tautomer; MDP: 10-methacryloyloxydecyl dihydrogen phosphate.

^a According to the information provided by the manufacturers

using a CAD/CAM system (Cercon Smart Ceramics, Dentsply International). After firing at 1,350°C for six hours, disk-shaped specimens 10 mm in diameter and 2.5 mm in thickness were prepared. Two types of high-strength indirect composite materials (Estenia C&B, Kuraray Medical Inc., Tokyo, Japan; and Gradia, GC Corp., Tokyo, Japan) originally designed for metal-based or free restoration were chosen for testing as layering materials. A single-liquid primer containing phosphate monomer (Alloy Primer, Kuraray Medical Inc.) was selected as the adhesion promoter.

Surface preparation

All disk specimens were ground flat with a series of silicon-carbide abrasive papers (#80, #240 and #600), and subsequently heated at 1,200°C for 10 min (Jelenko Cerafusion VPF MS-1200, J Morita Corp., Osaka, Japan) to regenerate the crystal phase (group C), because the crystal phase is transformed to monoclinic system by grinding in a way that would not occur in clinical practice. Some specimens were further treated by air-abrasion with 50–70 μm alumina (Hi-Aluminas, Shofu Inc., Kyoto, Japan) for 10 or 20 s (groups S10 and S20, respectively), followed by washing with a steam cleaner for 1 min and air-drying. The supply-side air-pressure during abrasion was 0.3 MPa and the distance from the orifice to the zirconia surface was approximately 10 mm (Jet Blast III, J Morita Corp.). Some of the S10 air abraded specimens were placed in a furnace at 500°C, heated to 1,200°C at a rate of 50°C/min, and then held at 1,200°C for 10 min (group H).

X-ray diffraction (XRD) analyses

The degree of transformation induced by air-abrasion and heat treatment was determined by measuring the peak intensity ratios in the XRD patterns of the disk-shaped specimens ($n=5$ per group). XRD data were collected with a $\theta/2\theta$ diffractometer (XD-D1, Shimadzu, Kyoto, Japan) using Cu-K α radiation at 30 kV and 30 mA. Diffractograms were obtained from 26° to 36° at a scan speed of 1°/min. The monoclinic peak intensity ratio, X_m , was calculated using the Garvie and Nicholson method¹⁷⁾ as follows:

$$X_m = \frac{I_m(\bar{1}11) + I_m(111)}{I_m(\bar{1}11) + I_m(111) + I_t(101)} \quad (1)$$

where I_t and I_m represent the integrated intensity (area under the peaks) of the tetragonal (101) and monoclinic (111) and ($\bar{1}11$) peaks around 30.2°, 31.5°, and 28.2°, respectively. Monoclinic volume content, V_m , was calculated using the method of Toraya *et al.*¹⁸⁾:

$$V_m = \frac{1.311X_m}{1 + 0.311X_m} \quad (2)$$

Surface roughness measurement

The surface roughness of specimens prepared by each different treatment method was measured as the arithmetic mean deviation of the profile (Ra) using a surface roughness tester (Surfcorder SE-3300, Kosaka Laboratory Ltd., Tokyo, Japan). The measurement conditions were: cut-off value 0.8 mm, probe 2 μm,

tracing speed 0.05 mm/s and sampling length 2.5 mm. The value for each specimen was obtained by measuring at three arbitrary points and averaging the values. Three specimens were evaluated for each different treatment method.

Shear bond strength (SBS) testing and failure analysis

16 disk-shaped specimens were prepared for each of the four surface preparations (C, S10, S20, H). A piece of double-sided tape with a circular hole 5 mm in diameter was positioned on the surface of each zirconia specimen to define the bonding area. Then the Alloy Primer was applied to the zirconia surfaces of all 4×16 disks using a sponge pellet.

A thin layer of one of two opaque resins (Gradia FO, GC Corp., or Estenia C&B OA3, Kuraray Medical Inc.) was applied to each zirconia surface and exposed to light for 60 s (Gradia) or 90 s (Estenia C&B) in a polymerizing unit (Hi Light power, Heraeus Kulzer GmbH, Wehrheim, Germany). An additional opaque material (Gradia OA3, GC Corp., Estenia C&B OA3, Kuraray Medical Inc.) was applied twice on top of the primary opaque resin, each of which was exposed to light for the same duration as the first layer. After light-exposure, a brass ring (6 mm inside diameter, 2 mm length, and 1 mm wall thickness) was placed surrounding the opaque resin. The ring was filled with a dentin shade of the indirect composite material (Gradia DA3, GC Corp., Estenia C&B DA3, Kuraray Medical Inc.). All specimens were then light cured with the polymerization unit for 300 s, and the Estenia specimens received a further final heat-polymerization at 110°C for 15 min in an oven (KL-100, J. Morita Corp.).

Thirty minutes after preparation, the specimens were immersed in water at 37°C for 24 h, and this state was defined as thermal cycle 0. Each set of 16 disks was then divided into two groups of 8 specimens: half (four sets of 8 specimens) were tested to determine the 24-h SBS without thermal cycling. The remaining specimens were placed in a thermal cycling apparatus (Thermal Cycler, Nissin Seiki Co. Ltd., Hiroshima, Japan) and cycled between 4°C and 60°C water, with a 1-min dwell time per bath, for 20,000 cycles.

Prior to SBS testing, the specimens were positioned in steel molds, and seated in a shear-testing jig. SBS were determined by means of a mechanical testing device (AGS-J, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm/min. The fractured interfaces were observed with an optical microscope (×8; S300II, Inoue Attachment Corp., Tokyo, Japan) and the failure modes were recorded as either: adhesive failure at the zirconia-resin interface (A), a combination of cohesive and adhesive failures (AC), or cohesive failure within the resin (C). Representative specimens were observed with a scanning electron microscope (SEM, VE-8800, Keyence Corp., Osaka, Japan).

Statistical analysis

For all tests, the mean and standard deviation (SD) for each set of surface treatments was calculated. The

results of the monoclinic zirconia content derived from the XRD and the surface roughness values were initially analyzed with the Levene test for the evaluation of equality of variance. The means of each group were also analyzed by one-way ANOVA and Scheffé or Tukey HSD post hoc pairwise comparison tests ($\alpha=0.05$).

The SBS results were compared with three-way analysis of variance (ANOVA) after the Levene test for the evaluation of equality of variance. Then, for each thermocycling status, the values of each treatment-indirect composite were compared by one-way ANOVA, and *post-hoc* Scheffé tests were performed with the value of statistical significance set at $\alpha=0.05$. In addition, to analyze the influence of the thermal cycling, the results of the samples from an identical surface treatment subjected to 0 or 20,000 thermal cycles were compared using Mann-Whitney's U tests with the value of statistical significance set at $\alpha=0.05$ for each treatment. All analyses were carried out using IBM SPSS 19.0 (IBM Japan Inc., Tokyo, Japan).

RESULTS

XRD results

The XRD patterns after grinding followed by heating to regenerate the crystal phase (C), air-abrasion for 10 or 20 s (S10 or S20), and heat treatment after abrasion for 10 s (H) are shown in Fig. 1. The diffraction peaks

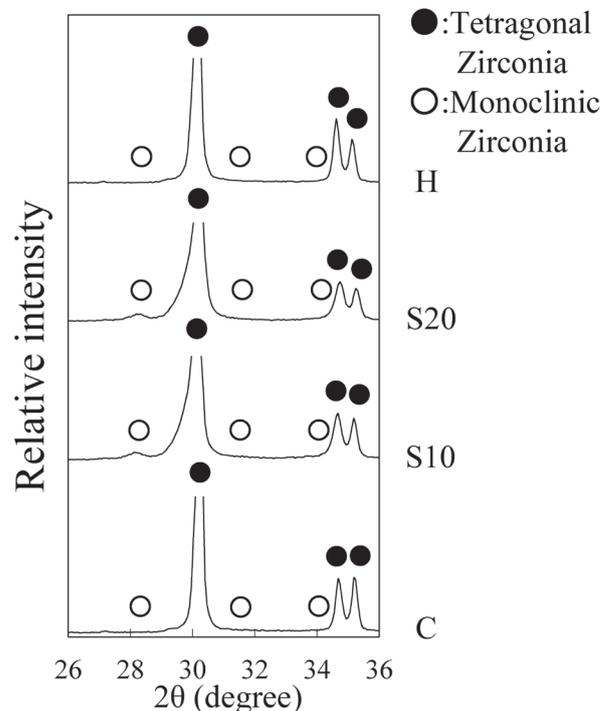


Fig. 1 XRD patterns of zirconia after each treatment. (C) ground flat; (S10) air-abraded with alumina for 10 s; (S20) for 20 s; (H) air-abraded for 10 s and heated.

due to monoclinic zirconia increased after both of the air-abrasion treatments (S10 or S20) as compared with C group samples, and the peaks for tetragonal zirconia decreased conversely. In contrast, the XRD pattern of H group samples showed an increase in the peaks corresponding to tetragonal zirconia. The peaks of the H group samples after heat treatment were nearly identical to those of the C group before air-abrasion.

The monoclinic zirconia contents derived from the XRD patterns using equations (1) and (2) are shown in Table 2. The monoclinic zirconia contents of S10 and S20 groups were significantly larger than those of the C and H groups ($p < 0.05$). There was no significant difference in monoclinic content between H group and C group ($p > 0.05$).

Surface roughness results

The surface roughness values of the zirconia surfaces tested are shown in Table 2. Air-abraded groups (S10,

S20, H) showed significantly higher Ra values than the control group (C) ($p < 0.05$). The surface roughness increased with increasing air-abrasion time, with the S20 group showing significantly higher Ra than the S10 group ($p < 0.05$). Comparison between samples with or without heat treatment after air-abrasion for same time (10 s, *i.e.* the S10 group and the H group) showed that there was no significant difference in Ra value ($p > 0.05$).

SBS and failure results

Means and standard deviations of SBS in MPa for the different sample groups and percentages of each failure mode are shown in Table 3. The SBS values for both indirect composites, with both thermal cycling statuses, were significantly higher for the S10 and S20 groups than the C groups ($p < 0.05$). There was no significant difference between S10 and S20 groups ($p > 0.05$). However, the H group showed significantly lower bond strength than S10 and S20 groups ($p < 0.05$). Some differences were seen with thermal cycling - the strengths of C (Gradia) and H (Estenia, Gradia) groups at 20,000 thermal cycles were significantly lower than the corresponding 0 thermal cycle data ($p < 0.05$). The number of combination adhesive and cohesive failures (AC) was reduced for all groups by application of thermal cycling. All specimens in the H groups for both indirect composites showed adhesive failure (A) after thermal cycling.

Electron micrographs of representative thermally cycled and debonded composite-zirconia surfaces are shown in Fig. 2. When Gradia was used after air-abrasion (S10, S20 groups), composite residues are identified on the zirconia surface (Figs. 2f and g). Complete detachment from the zirconia surface occurred in almost all specimens except for the abovementioned groups (Figs. 2 a–e, and h).

Table 2 Means and standard deviations of monoclinic content and surface roughness values (Ra) for each group

Groups	Monoclinic content (SD) (vol%)	Surface roughness (SD) (μm)
C	0.74 (0.15) ^A	0.05 (0.01) ^a
S10	4.61 (1.02) ^B	0.45 (0.02) ^b
S20	5.44 (0.49) ^B	0.50 (0.03) ^c
H	0.99 (0.25) ^A	0.46 (0.03) ^b

Within the same column, different letters indicate groups that are statistically different ($p < 0.05$).

Table 3 Means (MPa) and standard deviations of shear bond strengths

Composites	Groups	0 thermal cycle				20,000 thermal cycles			
		Mean (SD)	A	AC	C	Mean (SD)	A	AC	C
Estenia	C	1.1 (0.1) ^A	100%	–	–	1.2 (0.4) ^a	100%	–	–
	S10	13.7 (2.2) ^C	100%	–	–	12.4 (3.0) ^c	100%	–	–
	S20	13.6 (2.1) ^C	75%	25%	–	13.9 (3.3) ^c	87.5%	12.5%	–
	H	6.3 (0.8) ^B	87.5%	12.5%	–	3.1 (1.8) ^{a,b *}	100%	–	–
Gradia	C	18.3 (1.0) ^D	–	100%	–	1.4 (0.2) ^{a *}	87.5%	12.5%	–
	S10	24.6 (3.0) ^E	25%	75%	–	22.4 (2.1) ^d	37.5%	62.5%	–
	S20	23.0 (1.5) ^E	25%	75%	–	21.5 (3.4) ^d	50%	50%	–
	H	19.7 (2.0) ^D	37.5%	62.5%	–	6.9 (2.8) ^{b *}	100%	–	–

Within the same column at each composite, mean with the different letters are statistically different ($p < 0.05$), *: significant difference from 0 thermal cycle bond strength at the same group ($p < 0.05$). A: adhesive; AC: combination of cohesive and adhesive; C: cohesive.

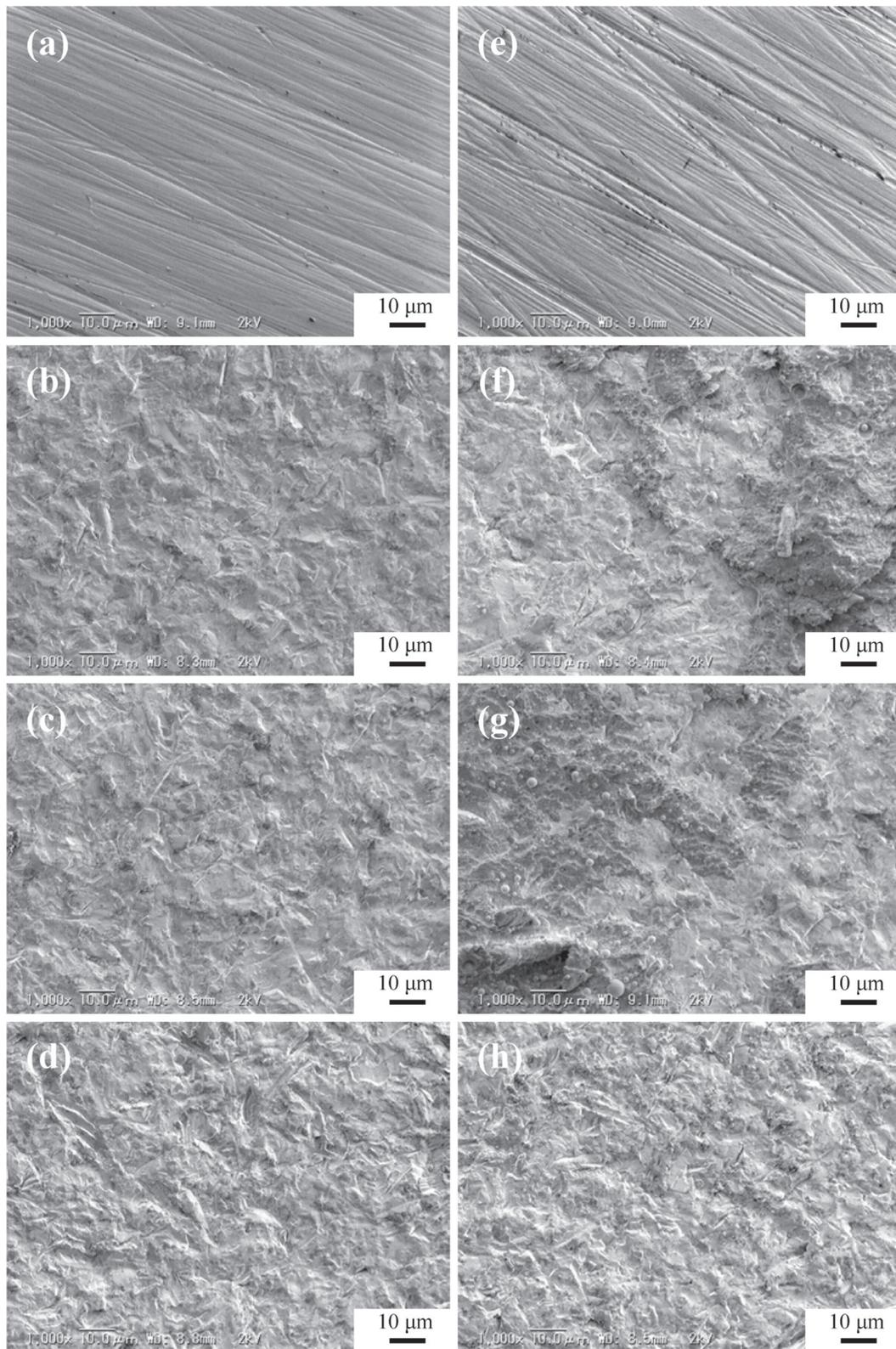


Fig. 2 Scanning electron micrographs of representative thermally cycled and debonded composite-zirconia fracture surfaces:
 (a) Estenia-C group; (b) Estenia-S10 group; (c) Estenia-S20 group; (d) Estenia-H group; (e) Gradia-C group; (f) Gradia-S10 group; (g) Gradia-S20 group; (h) Gradia-H group.

DISCUSSION

Air-abrasion of the zirconia surface is an important treatment method to get a strong bond to veneering porcelain. However, the mechanical stress initiates a phase transition to monoclinic zirconia, and the phase transition gives rise to volume expansion and reduction in coefficient of thermal expansion. Therefore, the phase transition needs to be reversed to the tetragonal zirconia by heat treatment. In contrast, the effect of heat treatment after air-abrasion on the bonding of indirect composites to zirconia is not known.

In this study, at first, the monoclinic zirconia contents produced by air-abrasion and subsequent heat treatment were determined by calculation from XRD data. The monoclinic zirconia content was increased by air-abrasion stress (S10 and S20 groups), in accordance with air-abrasion time (Table 2). However, the monoclinic content induced by air-abrasion stress was largely reversed by heat treatment (H group). These results were very similar to previous studies¹⁶⁻¹⁸⁾. Then, the surface roughness resulting from four different surface preparations (C, S10, S20, H) was examined in terms of Ra (Table 2). The values for the air-abraded groups were significantly higher compared with the ground flat group, and the roughness also increased in accordance with air-abrasion time. However, in this case, the S10 group was not significantly different from the H group in surface roughness. These two groups had the same air-abrasion time. This means that the surface roughness is not altered by the temperatures used in our heat treatment¹⁵⁾. These results show that the air-abrasion pressure can increase the monoclinic zirconia content with increase of surface roughness. However, the heat treatment only reverses the increase in monoclinic zirconia content, and does not affect the surface roughness.

The results of the present study show that the bond strengths of samples in the H groups were lower than S10 and S20 groups for both indirect composites regardless of thermal cycling, and that these strengths were also significantly decreased by thermal cycling (Table 3). As shown above, S10 and S20 groups had higher monoclinic contents than H group. The coefficient of thermal expansion of monoclinic zirconia ($7.5 \times 10^{-6}/^{\circ}\text{C}$)^{19,20)} is significantly lower than that of tetragonal zirconia ($10-10.5 \times 10^{-6}/^{\circ}\text{C}$)²¹⁾. However, this cannot be regarded as an influence on strength of bonds to indirect composites, because the coefficients of thermal expansion of the Estenia ($20.7 \times 10^{-6}/^{\circ}\text{C}$) and Gradia ($59.6 \times 10^{-6}/^{\circ}\text{C}$)²²⁾ used in this study are significantly different from those of both monoclinic and tetragonal zirconia. Although the reason for this decrease in bond strength upon heat treatment remains unclear, it is probably due to a decrease in the adsorbed hydroxide on the zirconia surface after heat treatment²³⁾. With respect to comparison of two indirect composites, the Gradia composite was relatively higher bond strength than Estenia (Table 3). There is difference in kind of monomer, content of inorganic filler and polymerization procedure between these composites²⁴⁻²⁶⁾.

Thus, it seems that difference in bond strength was affected by these factors.

Regardless of the thermal cycling status, many groups showed a high percentage of adhesive failures, and no cohesive failures were seen in any group (Table 3, Fig. 2). The result is different from previous studies which showed mostly cohesive failures using phosphate monomer and resin cement^{8,10,27)}. This indicated that there is a problem at the interface between zirconia and the composites, which is likely to be because of the physical properties of the composite such as wettability or filler size. In addition, the H group after thermal cycling showed only adhesive failures with both indirect composites. These results suggest that although the bond strength values vary with the type of indirect composite, the influences of air-abrasion and subsequent heat treatment are not changed.

Usually, the transformation from tetragonal to monoclinic zirconia induced by air-abrasion expands their volume to generate compressive stress on the surface, resulting in an increase in strength, and subsequent regeneration of tetragonal phase by heat treatment reduces the compressive stress on the surface, resulting in a decrease in strength²⁸⁾. Furthermore, neither of the coefficients of thermal expansion of tetragonal or monoclinic zirconia is compatible with those of indirect composites¹⁹⁻²²⁾. Thus, within the limitations of this study it was possible to conclude that air-abrasion is an effective method to enhance bonding between zirconia and indirect composites, whereas heat treatment after air-abrasion is not necessary.

CONCLUSION

Within the limitations of this study, it was possible to conclude that air-abrasion is highly effective in improving bonding between indirect composite and primed zirconia (Y-TZP). However, heating the zirconia after air-abrasion decreases the bond strength with the indirect composites studied.

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