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RESEARCH AND EDUCATION

Influence of various airborne-particle abrasion conditions on bonding between zirconia ceramics and an indirect composite resin material

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ABSTRACT

Statement of problem. Indirect composite resins (ICRs) have been suggested as veneering materials for implant-supported zirconia-based fixed dental prostheses; however, obtaining a durable bond between the zirconia ceramic and the ICR is a challenge.

Purpose. The purpose of this in vitro study was to evaluate the influence of airborne-particle abrasion conditions on the bond strength between 2 kinds of zirconia (yttria-stabilized tetragonal zirconia polycrystal [Y-TZP] and ceria-stabilized tetragonal zirconia/alumina nanocomposite [Ce-TZP/A]) and an ICR.

Material and methods. Zirconia disks were prepared by using computer-aided design and computer-aided manufacturing (CAD-CAM) systems. Specimens were airborne-particle abraded with different particle sizes (25, 50, 90, 125 μm) and jet pressures (0.1, 0.2, 0.3, 0.4 MPa). The control group (CO) was not subjected to airborne-particle abrasion. The surface roughness (Ra) of the specimens was measured. Subsequently, the specimens were treated with a primer and bonded with a light-activated composite resin, and the shear bond strength (SBS) was tested. The obtained data were analyzed by using multivariate analysis of variance, the Spearman rank-order correlation, and the Mann-Whitney U test (α=.05). After the SBS test, the interface failure modes were observed by scanning electron microscopy, and X-ray photoelectron spectroscopy (XPS) was used to analyze the chemical changes of the zirconia surface.

Results. The Ra values increased significantly (P<.05) after airborne-particle abrasion with a positive correlation with both particle size and jet pressure. The airborne-particle abraded specimens exhibited significantly higher bond strength after thermocycling (P<.05) than the CO. Nevertheless, the bond strength was not significantly different among different airborne-particle abrasion treatments (P>.05). Additionally, Y-TZP had higher mean bond strength values than Ce-TZP/A. The XPS results revealed that after airborne-particle abrasion, the alumina particles mechanically adhered to the zirconia surface.

Conclusions. Within the limitations of this in vitro study, airborne-particle abrasion improved the bond strength between zirconia and ICR; however, particle size or jet pressure were not influencing factors. (J Prosthet Dent 2019;122:491.e1-e9)

Patient esthetic expectations and concerns about metal allergy have led to an increase in metal-free dental treatments, especially ceramics.[1-3] Moreover, with the development of computer-aided design and computer-aided manufacturing (CAD-CAM) systems, the use of high-strength zirconia has increased.[4-10] Zirconia veneered with feldspathic porcelain became popular in restorative dentistry[11-16]; however, chipping affected clinical performance[17-19] until the laboratory process was optimized with slow cooling.[20-22] Indirect composite resin (ICR)-veneered prostheses have been suggested instead of porcelain to provide esthetics and strength.[23-26]

The bonding methods for silica-based ceramics include hydrofluoric acid-etching and

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Clinical Implications

Airborne-particle abrasion is essential when bonding zirconia ceramics to an indirect composite resin; however, this study identified that different particle sizes or jet pressures of airborne-particle abrasion would not affect the durability of the bond.

subsequent silane treatments,[27-29] and chemical and mechanical preprocessing have been recommended to enhance the bond strength of ICR to zirconia.[30,31] Metal primers containing a phosphoric acid ester monomer (10-methacryloyloxydecyl dihydrogen phosphate, MDP) have been reported to be effective,[32-35] and airborne-particle abrasion treatments have been suggested to improve the mechanical fitting by producing irregularities on material surface.[36-40] However, studies that comprehensively analyzed the effects of alumina particle sizes and jet pressures on the bond strength are lacking.[27,41-48] Therefore, the purpose of this in vitro study was to determine whether airborne-particle abrasion with various alumina particle sizes and jet pressures affected bond strengths and durability of 2 different zirconia ceramics and an ICR. The null hypotheses were that different airborne-particle abrasion conditions would not affect the bond strengths and bond durability between zirconia ceramics and an ICR.

MATERIAL AND METHODS

Details on the materials used in this study are provided in Table 1. Zirconia disk-shaped specimens (10 mm in diameter and 2.5 mm in thickness) were prepared by using CAD-CAM systems. All specimens were ground flat by using 600-grit diamond papers (Diamond Pad; Maruto Instrument Co, Ltd) and cleaned with distilled water (group CO). Some specimens were further treated by airborne-particle abrasion with 4 different alumina particle sizes of 25, 50, 90, and 125 μm for 10 seconds, followed by steam cleaning (Steam Cleaner-Z; SHOFU Inc) and air-drying. The jet pressure during abrasion was 0.3 MPa, and the distance from the orifice to the zirconia surface was approximately 10 mm. Some specimens were airborne-particle abraded with 50 μm grain-sized alumina particles with 4 different jet pressures of 0.1, 0.2, 0.3, and 0.4 MPa.

The surface roughness (Ra) of the specimens prepared in each zirconia group (n=5) was measured as the arithmetic mean deviation of the profile by using a surface roughness tester (Surfcorder SE-3300; Kosaka Laboratory Ltd). Ra was measured at 3 arbitrary points in each specimen, and the values were averaged. Representative specimens were observed by scanning electron microscopy (SEM) (VE-8800; Keyence Corp).

Table 1. List and properties of materials used

Materials/Trade Name (Abbr.)		Main Composition*	Manufacturer	Lot No.
Zirconia ceramic				
P-Nano ZR (Ce-TZP/A)		ZrO_2, Al_2O_3, CeO_2	YAMAKIN Co, Ltd	CD90011076J
Cercon Base (Y-TZP)		ZrO_2, Y_2O_3	Dentsply Sirona	18001606
Indirect composite resin (ICR)				
Gradia	FO	UDMA, silica nano powder	GC Corp	1111152
	OA3	UDMA, silica nano powder	–	1110192
	DA3	UDMA, inorganic-organic composite filler, silica nano powder, glass powder	–	1307171
Primer				
Alloy Primer		VTD, MDP, acetone	Kuraray Medical Inc	0412AA
Alumina particle				
Cobra	25 μm	Al_2O_3, SiO_2	Renfert GmbH	922315
	50 μm	–	–	1054941
	90 μm	–	–	1049807
	125 μm	–	–	942295

Bis-GMA, bisphenol-A-glycidyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; UDMA, urethane dimethacrylate; VTD, 6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol, or -2,4-dithione tautomer. *According to information provided by manufacturers.

Subsequently, 160 disk-shaped specimens were prepared for each zirconia group. A piece of double-sided tape with a circular hole of diameter 5 mm was positioned on the surface of each zirconia specimen to define the bonding area. Then, a metal primer (Alloy Primer; Kuraray Medical Inc) was applied to the zirconia surfaces of all disks. A thin layer of opaque resins and an additional opaque material were applied to each zirconia surface in sequence and then exposed to light polymerization for 60 seconds. After light exposure, the dentin shade of the indirect composite resin material was applied on all specimens, followed by light polymerization for 180 seconds. One hour after preparation, the specimens were immersed in water at 37 °C for 24 hours; this state was denoted as "0 thermocycles."

The 24-hour shear bond strength (SBS) of half the number of specimens in each zirconia group (n=10) was tested at 0 thermocycles. The remaining specimens (n=10) were placed in a thermocycling apparatus (Thermal Cycler; Nissin Seiki Co, Ltd) and cycled between 4 °C and 60 °C in water with a 1-minute dwell time per bath for 2×10^4 cycles. A universal testing machine (Autograph AGS-J; Shimadzu Corp) was used to perform the SBS test. Shear force was applied to the adhesive interface until fracture occurred at a crosshead speed of 0.5 mm/min. After the SBS test, the fractured interfaces were observed by using an optical microscope (S300II; Inoue Attachment Corp) at ×8 magnification to determine the

Table 2. Mean (μm) ±standard deviation (SD) of surface roughness (Ra) values for each zirconia group under different conditions of airborne-particle abrasion

	Y-TZP	Ce-TZP/A	
Conditions	**Mean ±SD**	**Mean ±SD**	**S**
CO	0.05 ±0.01[a]	0.05 ±0.01[f]	–
25 μm M 0.3 MPa	0.20 ±0.02[b]	0.18 ±0.02[g]	S
50 μm M 0.3 MPa	0.24 ±0.02[c]	0.23 ±0.01[h]	–
90 μm M 0.3 MPa	0.35 ±0.02[d]	0.30 ±0.01[i]	S
125 μm M 0.3 MPa	0.43 ±0.03[e]	0.36 ±0.02[j]	S
CO	0.05 ±0.01	0.05 ±0.01[F]	–
50 μm M 0.1 MPa	0.12 ±0.01[B]	0.11 ±0.01[G]	–
50 μm M 0.2 MPa	0.19 ±0.01[C]	0.16 ±0.01[H]	S
50 μm M 0.3 MPa	0.24 ±0.02[D]	0.23 ±0.01[I]	–
50 μm M 0.4 MPa	0.34 ±0.03[E]	0.28 ±0.02[J]	S

S, significant difference between 2 zirconia groups (*P*<.05). Within same column, different letters indicate groups statistically different (*P*<.05).

Table 3. Results of 2-way ANOVA on effects of zirconia and different conditions of airborne-particle abrasion on surface roughness (Ra) values

Source	Type III Sum of Squares	Df	Mean Square	F	*P*
Jet pressure	1.12	4	0.279	891.90	<.001
Zirconia	0.03	1	0.028	90.58	<.001
Jet pressure×zirconia	0.02	4	0.004	11.98	<.001
Error	0.04	140	<0.01	–	–
Particle size	2.06	4	0.515	1737.94	<.001
Zirconia	0.03	1	0.034	115.01	<.001
Particle size×zirconia	0.03	4	0.006	20.95	<.001
Error	0.04	140	<.001	–	–

mode of failure. Representative specimens were observed by SEM.

The surfaces of the control and airborne-particle abrasion specimens were analyzed chemically by X-ray photoelectron spectroscopy (XPS) (AXIS-HS; Kratos Analytical Ltd). The measurements were performed under a vacuum condition ($\leq 10^{-7}$ Pa) with Al-Kα monochromatic X-rays at a source power of 150 W. Charge compensation was achieved by using an electron flood gun equipped with the AXIS-HS instrument. Wide- and narrow-scan spectra (Zr 3p, O 1s, C 1s, Al 2s, Y 3p, Hf 4d) were acquired at pass energy of 40 eV and a photoelectric uptake angle of 90 degrees.

The collected data were calculated, and the normality of the distribution was analyzed with the Shapiro-Wilk test. The correlations between Ra values and different conditions were analyzed by using the Spearman rank-order correlation. The result of Ra values was compared with that from 2-way ANOVA, and multiple zirconia group comparisons were performed by 1-way ANOVA with the post hoc Tukey HSD test (α=.05). The SBS results were compared with those from 3-way ANOVA, and multiple zirconia group comparisons were performed with 1-way ANOVA with post hoc Scheffé tests and Mann-Whitney U tests (α=.05). All analyses were performed by using a statistical software program (IBM SPSS Statistics, v24; IBM Corp).

RESULTS

The Ra values of the zirconia surfaces tested are summarized in Tables 2 and 3. For a constant jet pressure (0.3 MPa), the particle size significantly increased the Ra values of the zirconia surface (*P*<.05). For a constant particle size (50 μm), the jet pressure significantly increased the Ra values of the zirconia surface. The Spearman rank-order correlation indicated a significant positive correlation between the Ra values and particle size in both Y-TZP (r=0.98, *P*<.05) and Ce-TZP/A (r=0.98, *P*<.05); likewise, a positive correlation was found between jet pressure and Ra values (Y-TZP: r=0.97, *P*<.05; Ce-TZP/A: r=0.98, *P*<.05).

On observing the surface topography of the zirconia before and after different airborne-particle abrasion treatment conditions (Figs. 1, 2), the group CO revealed a smooth surface in each zirconia group. In contrast, the zirconia surfaces were uneven and rough with scratches after airborne-particle abrasion; these irregularities became distinct with increasing particle size or jet pressure. Furthermore, comparing the Ra values between the 2 zirconia groups, Y-TZP had significantly larger Ra values than Ce-TZP/A (*P*<.05).

The SBS test results are presented in Tables 4-6. At 0 thermocycle, group CO had a significantly lower bond strength than the Ce-TZP/A airborne-particle abraded specimens, whereas these differences were not observed for the Y-TZP specimens. After 2×10^4 thermocycles, airborne-particle abrasion significantly enhanced the bond strength compared with the group CO. However, regardless of whether 0 or 2×10^4 thermocycles were performed, there was no significant difference in the bond strength among the different airborne-particle abrasion treatment conditions (*P*>.05). When the bond strength between the 2 zirconia groups was compared, the difference was not significant at 0 thermocycles (*P*>.05), but after 2×10^4 thermocycles, the bond strength of Y-TZP was significantly higher than that of Ce-TZP/A (*P*<.05).

Regarding the failure mode (Tables 4 and 5), for 0 thermocycles, adhesive failure was not observed in any specimens; most specimens in the group CO showed combination failure; cohesive failure occurred in the posttreatment specimens. SEM images of a representative thermocycled and debonded zirconia/ICR interface are seen in Figure 3. Most specimens in the group CO showed adhesive failure after 2×10^4 thermocycles and combination failure under other conditions.

Narrow-scan XPS spectra of the Al 2s region of Y-TZP with or without airborne-particle abrasion are presented in Figure 4. An Al 2s peak was observed at an expected

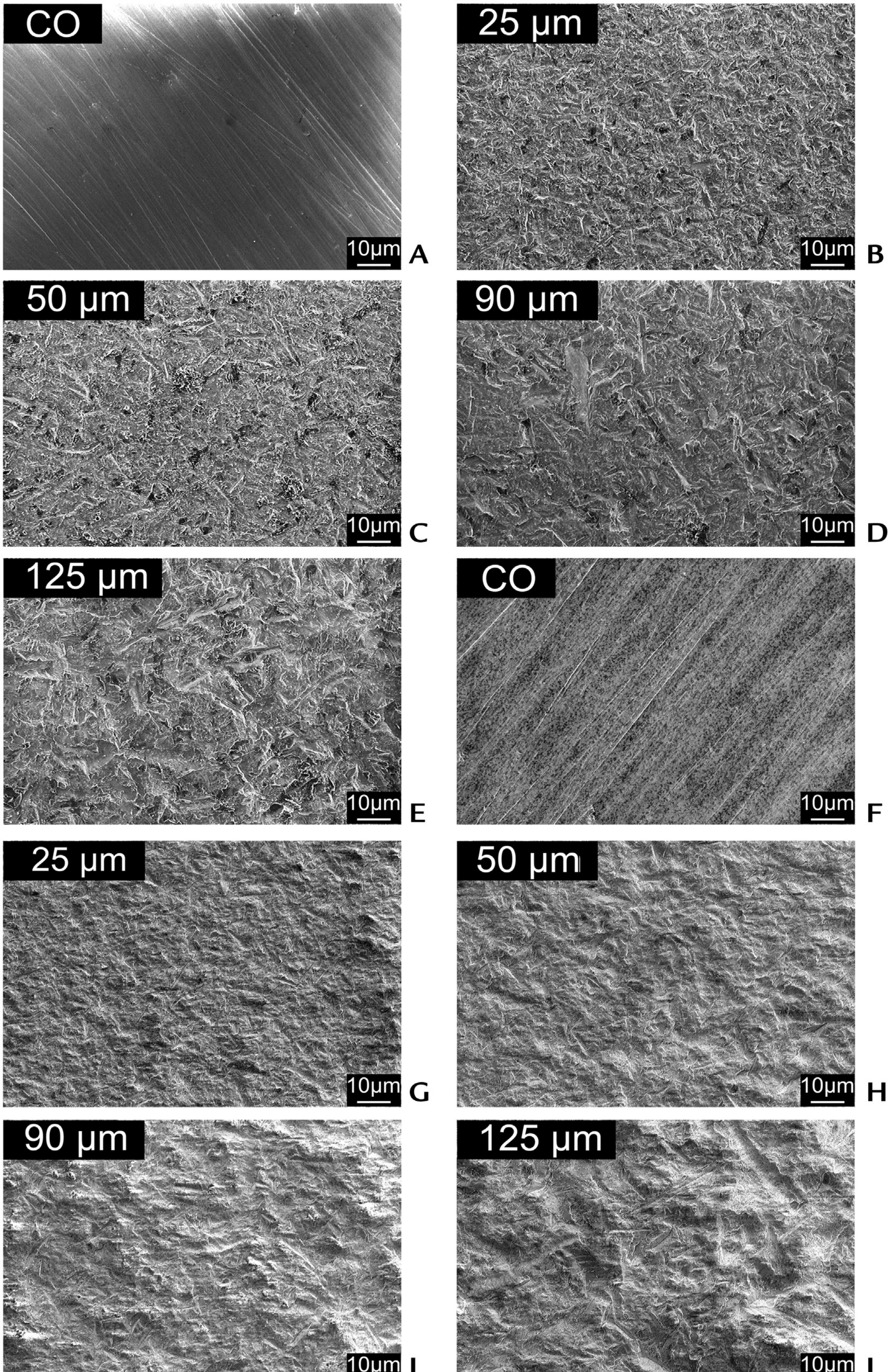

Figure 1. Scanning electron micrographs of surface roughness after airborne-particle abrasion with 0.3 MPa of jet pressure and different particle sizes. A-E, Y-TZP group. F-J, Ce-TZP/A group. Original magnification ×1000.

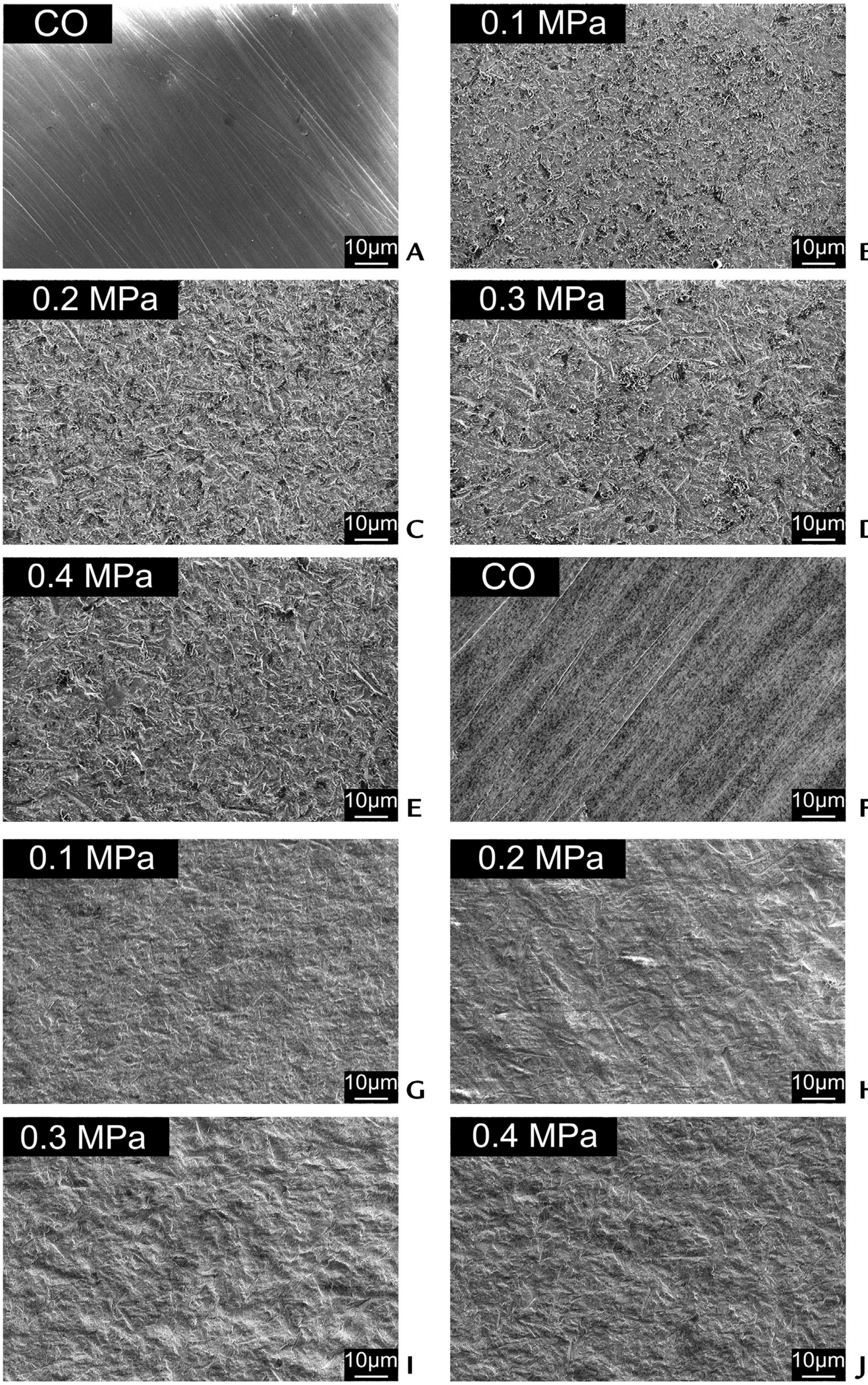

Figure 2. Scanning electron micrographs of surface roughness after airborne-particle abrasion with 50-μm-sized particles and different jet pressures. A-E, Y-TZP group. F-J, Ce-TZP/A group. Original magnification ×1000.

Table 4. Mean values of bond strengths (MPa) and failure modes for each zirconia group after airborne-particle abrasion with 0.3 MPa of jet pressure and different particle sizes (μm)

		0 Thermocycle				20 000 Thermocycles					
Groups	**Conditions**	**Mean ±SD**	**A**	**AC**	**C**	**Mean ±SD**	**A**	**AC**	**C**	**S**	**Reduction**
Y-TZP	CO	25.1 ±10.3^{a}	0	9	1	0.7 ±0.3^{A}	10	0	0	S	97.4%
	25 μm M	28.7 ±3.4^{a}	0	6	4	25.5 ±5.3^{B}	0	10	0		11.1%
	50 μm M	33.1 ±6.1^{a}	0	3	7	30.1 ±3.9^{B}*	0	10	0		9.2%
	90 μm M	29.7 ±2.7^{a}	0	3	7	24.6 ±4.7^{B}	0	10	0	S	17.2%
	125 μm M	32.7 ±5.8^{a}	0	1	9	28.5 ±6.6^{B}+	0	10	0		13.0%
Ce-TZP/A	CO	20.7 ±3.0^{b}	0	10	0	1.9 ±3.3^{C}	9	1	0	S	91.1%
	25 μm M	30.0 ±4.3^{c}	0	1	9	23.1 ±6.0^{D}	1	9	0	S	22.8%
	50 μm M	31.4 ±2.7^{c}	0	1	9	24.2 ±1.8^{D}*	0	10	0	S	23.0%
	90 μm M	31.2 ±5.9^{c}	0	1	9	22.6 ±8.1^{D}	0	8	2	S	27.7%
	125 μm M	28.6 ±6.0^{c}	0	0	10	21.5 ±6.0^{D}+	0	9	1	S	25.0%

A, adhesive failure; AC, combination of cohesive and adhesive failure; C, cohesive failure; Reduction, rate of reduction; S, significant difference between before and after thermocycling (*P*<.05); SD, standard deviation. Within same column, different letters indicate groups statistically different (*P*<.05). *+, significant difference between 2 zirconia groups (*P*<.05).

Table 5. Mean values of bond strengths (MPa) and failure modes for each zirconia group after airborne-particle abrasion with 50-μm-sized particles and different jet pressures (MPa)

		0 Thermocycle				20 000 Thermocycles					
Groups	**Conditions**	**Mean ±SD**	**A**	**AC**	**C**	**Mean ±SD**	**A**	**AC**	**C**	**S**	**Reduction**
Y-TZP	CO	25.1 ±10.3^{a}	0	9	1	0.7 ±0.3^{A}	10	0	0	S	97.4%
	0.1 MPa	31.8 ±4.7^{a}	0	2	8	28.8 ±4.3^{B}*	0	10	0	—	9.4%
	0.2 MPa	31.7 ±4.8^{a}	0	3	7	31.4 ±5.1$^{B\xi}$	0	10	0	—	0.8%
	0.3 MPa	33.1 ±6.1^{a}	0	3	7	30.1 ±3.9^{B}+	0	10	0	—	9.2%
	0.4 MPa	33.7 ±6.4^{a}	0	1	9	29.3 ±4.1^{B}	0	10	0	—	13.0%
Ce-TZP/A	CO	20.7 ±3.0^{b}	0	10	0	1.9 ±3.3^{C}	9	1	0	S	91.1%
	0.1 MPa	29.6 ±4.4^{c}	0	0	10	23.6 ±3.1^{D}*	0	10	0	S	20.3%
	0.2 MPa	29.4 ±5.7^{c}	0	0	10	27.3 ±3.7$^{D\xi}$	0	10	0	—	7.2%
	0.3 MPa	31.4 ±2.7^{c}	0	1	9	24.2 ±1.8^{D}+	0	10	0	S	23.0%
	0.4 MPa	32.3 ±3.9^{c}	0	0	10	26.9 ±4.3^{D}	0	10	0	—	16.7%

A, adhesive failure; AC, combination of cohesive and adhesive failure; C, cohesive failure; Reduction, rate of reduction; S, significant difference between before and after thermocycling (*P*<.05); SD, standard deviation. Within same column, different letters indicate groups statistically different (*P*<.05). *$^{\xi}$+, significant difference between 2 zirconia groups (*P*<.05).

Table 6. Results of 3-way ANOVA on effects of zirconia, thermocycles, and different conditions of airborne-particle abrasion on shear bond strength (SBS)

Source	**Type III Sum of Squares**	**df**	**Mean Square**	**F**	***P***
Particle size	8147.46	4	2036.86	72.76	<.001
Thermocycle	3936.15	1	3936.15	140.59	<.001
Zirconia	273.59	1	273.59	9.77	.002
Particle size×thermocycle	2066.78	4	516.69	18.46	<.001
Particle size×zirconia	204.89	4	51.22	1.83	.13
Thermocycle×zirconia	37.50	1	37.49	1.34	.25
Particle size×thermocycle×zirconia	170.84	4	42.71	1.53	.12
Error	5039.28	180	27.99	—	—
Jet pressure	9557.13	4	2389.28	106.54	<.001
Thermocycle	2402.70	1	2402.70	107.14	<.001
Zirconia	573.28	1	573.28	25.56	<.001
Jet pressure×thermocycle	2817.06	4	704.27	31.41	<.001
Jet pressure×zirconia	48.99	4	12.25	0.55	.70
Thermocycle×zirconia	0.43	1	0.43	0.02	.89
Jet pressure×thermocycle×zirconia	202.05	4	50.51	2.25	.07
Error	4036.57	180	22.43	—	—

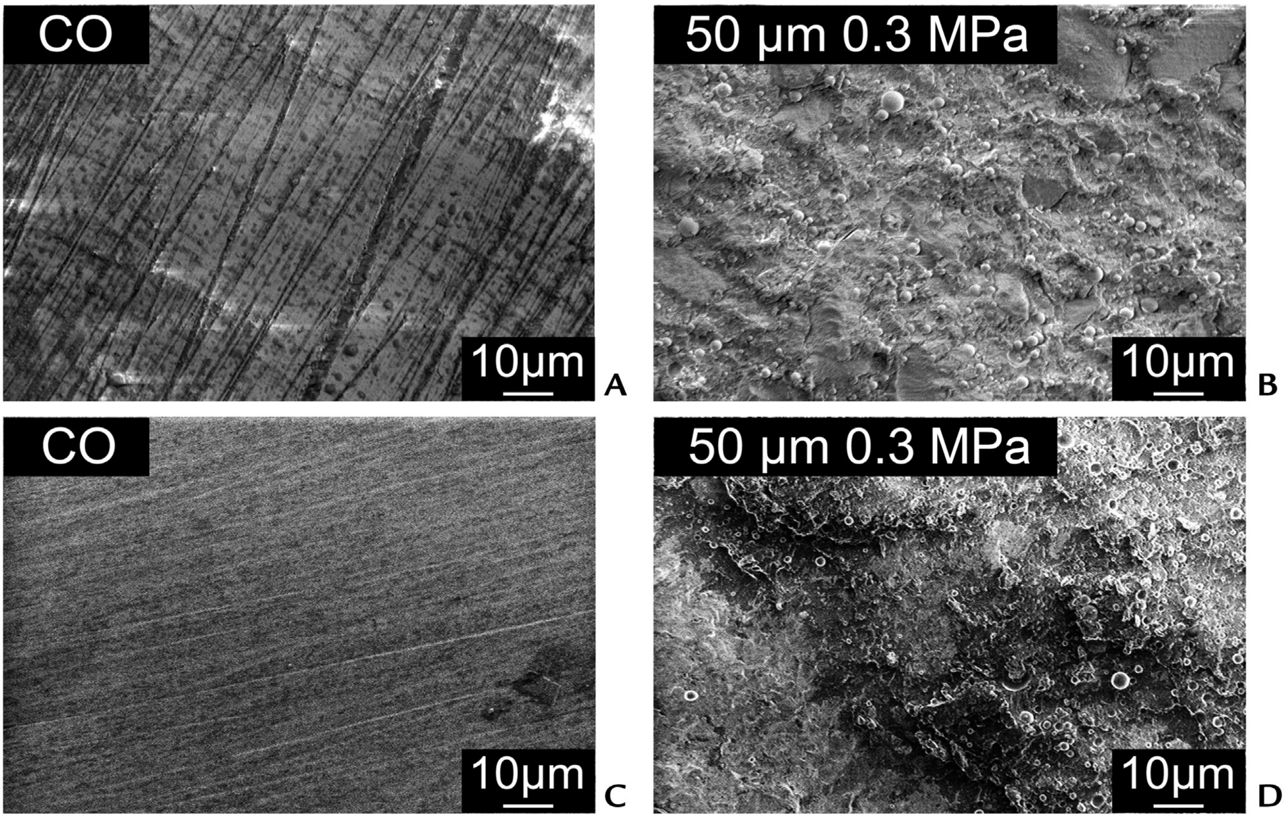

Figure 3. Representative scanning electron micrographs of debonded zirconia-ICR interface after thermocycling. A-B, Y-TZP group. C-D, Ce-TZP/A group. Original magnification ×1000.

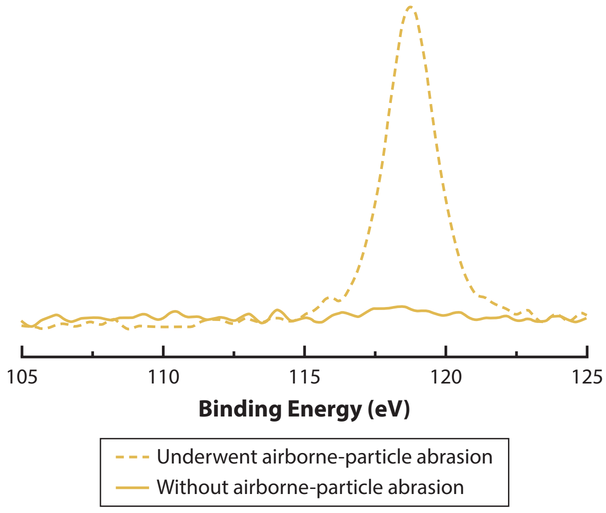

Figure 4. X-ray photoelectron spectroscopy of narrow-scan spectra of the Al 2s region of yttria-stabilized tetragonal zirconia polycrystal.

binding energy of approximately 119 eV in the airborne-particle abraded specimens, but not in the group CO specimens. The atomic ratios of [Al]/[Zr], analyzed from the narrow-scan spectra, were higher for the airborne-particle abraded specimens than for the group CO specimens.

DISCUSSION

The airborne-particle abrasion specimens showed significantly higher bond strengths than the group CO, and the different airborne-particle abrasion conditions were not significantly related to bond strengths (Tables 4 and 5). Therefore, the null hypothesis was rejected.

The application of zirconia-based ceramics has increased rapidly in clinical dentistry during the past decade.[2,5] When zirconia is used as a framework for a fixed dental prosthesis, feldspathic porcelain has been generally used as the veneering material; however, chipping of the porcelain has been reported.[17,26] ICRs may be more suitable as a veneering material, expanding the application of metal-free treatments. A limitation to using ICRs is the durability of the bond between the zirconia and the ICR.[27,41,42] However, the present study found that airborne-particle abrasion improved the bond between the zirconia ceramics and the ICR.[43,46,48] The aluminum oxide particles produce irregularities and shallow pits in the zirconia surface, thereby enhancing mechanical retention and the adhesive area, consequently improving bond strength.[38,39] Airborne-particle abrasion also cleans and activates the zirconia surface by removing organic contaminants from the surface.[40]

As shown in Tables 4 and 5, most specimens in the group CO suffered adhesive failure after 2×10^4

thermocycles, but the others experienced combination failure. In addition, the SEM images (Fig. 3) indicated the presence of a part of or all the residual opaque resin on the zirconia surface. The airborne-particle abrasion specimens did not fail because the bond strength could not withstand the load in the SBS test; rather, the reasons for failure are related to the physical properties of the ICR. When in an aqueous environment, the bond strength of an ICR decreases owing to its physical properties such as wettability or filler size; this leads to combination failure without a further decrease in the bond strength.[25]

The SEM images (Figs. 1, 2) revealed that the surface roughness became distinct and the Ra values increased (Table 2) with increasing alumina particle size and jet pressure. However, these factors did not significantly (P>.05) affect the bond strength in either zirconia (Table 4 and Table 5). This might be because the Vickers hardness of the zirconia materials used in this study was greater than 1100 Hv[12]; hence, the irregularities formed from airborne-particle abrasion were superficial, with only minimal undercuts. The bond strength would not increase even if the particle sizes and jet pressures increased, and these results are consistent with those of other reports.[37,43,46,48] Nevertheless, airborne-particle abrasion with a large particle size or high jet pressure might cause chipping and damage in a thin portion of the prostheses; therefore, it is not necessary to airborne-particle abrasion with a large particle size or high jet pressure.[31,36] To avoid damaging zirconia and simultaneously achieve optimum bond durability, airborne-particle abrasion should be with 50 μm-sized particles and 0.2 or 0.3 MPa of jet pressure.

XPS was performed to analyze the chemical changes of the zirconia surface before and after airborne-particle abrasion. Al 2s peaks in the XPS narrow-scan spectra were identified near a binding energy of 119 eV in the Y-TZP airborne-particle abrasion specimens (Fig. 4). This indicated that alumina (Al_2O_3) particles mechanically adhered to the Y-TZP surfaces.[35] As Ce-TZP/A already contains Al_2O_3, this effect could not be analyzed; nevertheless, the Ce-TZP/A surface was expected to also acquire alumina particles because of airborne-particle abrasion. Hence, it could be inferred that airborne-particle abrasion affects bond durability.

On comparing the 2 zirconia ceramics and only when subjected to a larger particle size or jet pressure, Ce-TZP/A showed smaller Ra values than Y-TZP (Table 2). This variation may have been because the homogeneous dispersion of alumina in the Ce-TZP matrix suppressed grain growth and increased the hardness and flexural strength of zirconia; accordingly, Ce-TZP/A exhibits higher fracture toughness than Y-TZP.[4,6,7,9,15,16] In addition, the results of this study, in accordance with previous studies, indicate that the bond strength of most Ce-TZP/A specimens significantly decreased after thermocycling.[44,45,47]

The valid parameters of airborne-particle abrasion (alumina blast) most commonly used for bonding between restorative materials were clarified; yet, the properties of thermal expansion and hygroscopicity for indirect composite resin materials must be considered in future experiments to evaluate more effective mechanical maintenance.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Airborne-particle abrasion effectively improved the bond strength between zirconia ceramics and ICR.
2. Y-TZP exhibited a higher bond strength with ICR than Ce-TZP/A.
3. The size of alumina particles or the jet pressures did not primarily influence the bond strength.

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