

Orthodontic bracket bonding to glazed full-contour zirconia

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Objectives: This study evaluated the effects of different surface conditioning methods on the bond strength of orthodontic brackets to glazed full-zirconia surfaces. **Materials and Methods:** Glazed zirconia (except for the control, Zirkozahn Prettau) disc surfaces were pre-treated: PO (control), polishing; BR, bur roughening; PP, cleaning with a prophyl cup and pumice; HF, hydrofluoric acid etching; AA, air abrasion with aluminum oxide; CJ, CoJet-Sand. The surfaces were examined using profilometry, scanning electron microscopy, and electron dispersive spectroscopy. A zirconia primer (Z-Prime Plus, Z) or a silane primer (Monobond-S, S) was then applied to the surfaces, yielding 7 groups (PO-Z, BR-Z, PP-S, HF-S, AA-S, AA-Z, and CJ-S). Metal bracket-bonded specimens were stored in water for 24 hr at 37°C, and thermocycled for 1,000 cycles. Their bond strengths were measured using the wire loop method ($n = 10$). **Results:** Except for BR, the surface pre-treatments failed to expose the zirconia substructure. A significant difference in bond strengths was found between AA-Z (4.60 ± 1.08 MPa) and all other groups ($13.38 \pm 2.57 - 15.78 \pm 2.39$ MPa, $p < 0.05$). For AA-Z, most of the adhesive remained on the bracket. **Conclusions:** For bracket bonding to glazed zirconia, a simple application of silane to the cleaned surface is recommended. A zirconia primer should be used only when the zirconia substructure is definitely exposed. (*Restor Dent Endod* 2016;41(2):106-113)

Key words: Adhesive; Bonding; Orthodontic brackets; Zirconia

Introduction

Direct bonding in orthodontics has improved esthetics, decreased gingival inflammation and enamel decalcification, and made the placement of orthodontic appliances more comfortable for patients and orthodontists.¹ With the increased demands for adult orthodontic treatment and growing popularity of esthetic dentistry, clinicians are often faced with the problem of bonding orthodontic brackets to different types of restorations as well as to the enamel.² Based on this trend, bond strength of brackets to esthetic pontic materials,³ temporary polycarbonate crowns,⁴ or ceramic surfaces including porcelain^{5,6} has undergone evaluation. Numerous options to improve bracket bonding to such substrates have been suggested, generally combinations of mechanical (e.g., surface roughening by air-particle abrasion, bur grinding, or hydrofluoric acid etching (HF) and chemical (e.g., primer application) conditioning methods.⁷

Recently, zirconia, specifically yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), has become favored as a core material mainly due to its improved fracture resistance by transformation toughening.^{8,9} However, clinical trials have shown high rate of

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porcelain veneer fracture, probably due to the mismatch between the thermal expansion coefficients of the zirconia and the veneered porcelain.¹⁰ To reduce these failures, anatomically contoured (full-contour) zirconia-based dental prostheses have also been developed. The elimination of the veneering porcelain layer has improved the clinical success and reliability of zirconia restorations.¹¹

In practice, many full-contour zirconia crowns are glazed and stained superficially during fabrication to improve their esthetic properties.¹² Hence, the strategy of orthodontic bracket bonding to glazed zirconia restorations should target two different bonding substrates: the thin porcelain glaze layer and the zirconia substructure. For bonding to porcelain, application of silane primer after HF produces a high resin bond strength by forming a covalent bond with both the ceramic and the resin.^{13,14} For bonding to zirconia, in contrast, different approaches, such as air-particle abrasion and the use of zirconia primers or adhesive luting cements, are needed because zirconia is not readily etched by hydrofluoric acid due to its high crystalline content and such silane chemistry is ineffective with zirconia.^{8,15} A review of the relevant literature indicates that little research has been carried out with respect to surface conditioning protocols for bonding of orthodontic brackets to glazed zirconia. It may be necessary to determine adequate surface conditioning methods to ensure a clinically acceptable bond strength of brackets to glazed full-contour zirconia.

The purpose of this study was to test the effect of different combinations of surface pre-treatments and primer applications on the shear bond strength of brackets bonded to glazed full-contour zirconia. The null hypothesis tested was that there is no difference among shear bond strengths of the groups tested.

Materials and Methods

Specimen preparation and surface pre-treatments

Disc-shaped zirconia specimens (Zirkonzahn Prettau, Zirkonzahn GMBH, Gais, Italy) with a diameter of 10 mm and a thickness of 1 mm were fabricated (sintered by firing at 1,600°C for 2 hours) and the surfaces were glazed with Initial IQ Lustre Pastes NF (GC America Inc., Alsip, IL, USA). The specimen preparation and glazing procedures were carried out by the same well-trained dental technician in accordance with the manufacturers' instructions. Unglazed zirconia served as the control bonding substrate in this study.

The surface was ultrasonically cleaned in isopropyl alcohol, air-dried, and subjected to one of the following pre-treatments (Table 1): PO (control), the unglazed surfaces were polished with silicon carbide paper up to #2000;⁸ BR, the glazed surfaces were gently roughened using a diamond bur (TR-19 [grit size: 106 - 125 µm], Mani Inc., Tochigi, Japan) in high-speed handpiece;⁴ PP, cleaned for 10 seconds with a rubber prophyl cup and fluoride-free pumice; HF, etched with 4% hydrofluoric acid gel (Bisco Inc., Schaumburg, IL, USA, lot no: 1200011101) for 5 minutes, washed thoroughly with water to remove the residual acid, and air-dried;⁷ AA, air-particle abrasion was performed with an intraoral air abrasion device (Microetcher IIA, Danville, San Ramon, CA, USA) filled with 30 µm aluminum oxide particles from a distance of approximately 10 mm at a pressure of 0.25 MPa for 15 seconds;^{7,8} CJ, the tribochemical silica coating was applied using 30 µm CoJet-Sand (3M ESPE, Seefeld, Germany) in the same device under the same conditions as AA.^{7,16}

Table 1. Surface conditioning (pre-treatment + primer application) methods to glazed zirconia specimens

Group code	Pre-treatment	Applied primer
PO-Z	Polishing unglazed (control) zirconia surface with silicon carbide paper	Z-Prime Plus*
BR-Z	Roughening with a diamond bur (grit size: 106 - 125 µm)	Z-Prime Plus
PP-S	Cleaning with a rubber prophyl cup and pumice	Monobond-S [†]
HF-S	4% hydrofluoric acid etching	Monobond-S
AA-S	Air-particle abrasion with 30 µm aluminum oxide particles	Monobond-S
AA-Z	Air-particle abrasion with 30 µm aluminum oxide particles	Z-Prime Plus
CJ-S	Air-particle abrasion with 30 µm silicon dioxide particles (CoJet-Sand)	Monobond-S

PO, polishing unglazed zirconia; BR, bur roughening; PP, cleaning with a prophyl cup and pumice; HF, hydrofluoric acid etching; AA, air-particle abrasion with aluminum oxide; CJ, silicon dioxide (CoJet-Sand) particles; Z, Z-Prime Plus; S, Monobond-S.

*1 - 2 coats were applied and dried with an air syringe for 3 - 5 sec.

[†]Applied with a brush, allowed to react for 60 sec, then dispersed with a strong stream of air.

Surface characterization

To examine the glaze layer, the glazed (not pre-treated) zirconia specimens were embedded in epoxy resin (Epofix, Struers, Copenhagen, Denmark) for cross-sectioning using a low-speed diamond saw (IsoMet Low Speed Saw, Buehler, Lake Bluff, IL, USA) under continuous water irrigation. The surface was ground, polished, and observed with a field emission-scanning electron microscope (FE-SEM, JSM-6700F, Jeol, Tokyo, Japan) after platinum sputter-coating.

The 6 pre-treated zirconia surfaces were investigated using SEM and additionally electron dispersive spectroscopy (EDS, Incax-sight, Oxford Instruments, High Wycombe, UK) to identify the chemical composition.

Surface roughness

The surface roughness R_a of pre-treated specimens ($n = 10$ per group) was measured using a previously calibrated profilometer (Surftest SV-400, Mitutoyo Corp., Kawasaki, Japan) at a stylus speed of 0.1 mm/sec, a cutoff of 0.8 mm, and a range of 600 μm . The R_a of each specimen was recorded as the average of the five readings.

Bracket bonding

From preliminary tests, the sample size ($n = 10$) was determined with a power analysis to provide statistical significance at 80% power. Prior to pre-treatments, the specimens were embedded in round silicone rubber molds using acrylic resin, ensuring the surface to be bonded remained uncovered. The surface was ultrasonically cleaned in isopropyl alcohol, air-dried, and subjected to the pre-treatments described above. Z-Prime Plus (Z, Bisco Inc., lot no: 1200011069) or Monobond-S (S, Ivoclar Vivadent, Schaan, Liechtenstein, lot no: P57807) was applied to the pre-treated surfaces according to the manufacturers' instructions, yielding 7 groups (PO-Z, BR-Z, PP-S, HF-S, AA-S, AA-Z, and CJ-S, $n = 10$ per group). The pre-treatment and primer application procedures are summarized in Table 1.

Stainless steel brackets designed for mandibular incisors (Gemini series, 3M Unitek, Monrovia, CA, USA) were bonded to the surfaces using a light-curing orthodontic bonding system (Transbond XT, 3M Unitek). The average surface of the orthodontic bracket base was 10.5 mm². The excess material was removed from the bracket margin with a hand scaler and light-cured from the mesial and distal aspects for 5 seconds each (total = 10 seconds) using a light-emitting diode curing unit (Bluephase G2, Ivoclar Vivadent) with an output intensity of 1,200 mW/cm² (high mode) as measured with a built-in radiometer. The specimens were stored in water for 24 hours at 37°C and then thermocycled at 5 and 55°C for 1,000 cycles.^{17,18}

Shear bond strength testing

Shear bond strength tests were performed by the wire loop method previously described by Mojtahedzadeh *et al.*¹⁹ The bonded specimens were mounted in a jig attached to a universal testing machine (3343, Instron Inc., Canton, MA, USA). A 0.018 × 0.025 inch stainless steel wire was engaged under tie wings. The shear load (pull of the steel wire) was applied at a crosshead speed of 0.5 mm/min until failure. The results were calculated in MPa.

After debonding, the residual composite on the zirconia surface was assessed based on an adhesive remnant index (ARI) score under an optical microscope (SMZ800, Nikon Corp., Tokyo, Japan) at x10 magnification. Each specimen was scored according to the amount of composite remaining on the surface as follows: 0 = no composite remaining, 1 = less than 50% of composite remaining, 2 = more than 50% of composite remaining, and 3 = all composite remaining, with a distinct impression of the bracket base.²⁰

Statistical analysis

Statistical analyses was carried out using SPSS 17.0 for Windows (SPSS Inc., Chicago, IL, USA) at a level of significance of $\alpha = 0.05$. The Shapiro-Wilks normality test and Levene's variance homogeneity test were applied to the surface roughness and bond strength data. As the roughness data were normally distributed but showed inhomogeneity of variances between groups ($p < 0.001$), they were analyzed using Welch's variance-weighted one-way analysis of variance (ANOVA) and the Games-Howell *post hoc* test. The bond strength data, which met both the normality and variance homogeneity assumptions, were analyzed using one-way ANOVA and Tukey *post hoc* test. The ARI scores were tabulated and analyzed using the Fisher's exact test.

Results

Figure 1 shows the representative cross-sectional SEM image of the glazed (not pre-treated) zirconia. The thickness of the glaze layer was found to be approximately 0.1 mm. The topographic SEM images of the pre-treated surfaces and their corresponding EDS spectra are presented in Figure 2. The surface roughness is also presented in Table 2. BR showed the roughest surface among the groups, followed by AA and CJ, which shared significantly similar R_a values ($p = 0.283$). HF exhibited a roughened surface with some striation formation. The EDS spectrum of BR showed Zr peaks (no Si peaks), whereas those of HF, AA, and CJ exhibited Si peaks (no Zr peaks).

Table 3 presents the mean shear bond strength values, standard deviations, and ranges of all groups. ANOVA and

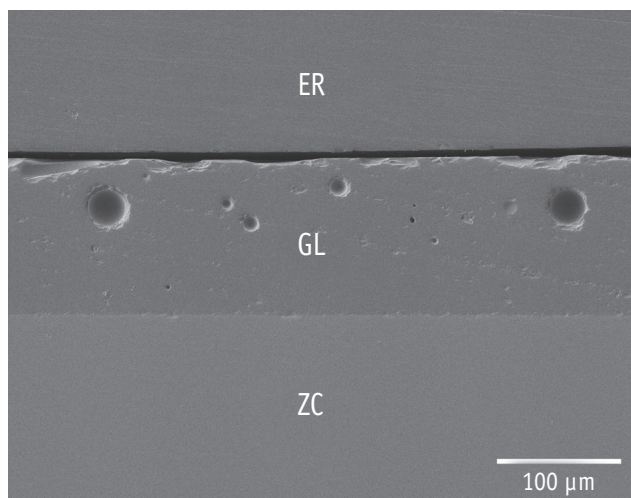


Figure 1. Representative cross-sectional SEM image of the glazed full-zirconia ceramic (original magnification $\times 200$, bar represents $100\ \mu\text{m}$). ER, epoxy resin for embedding; GL, glaze layer; ZC, zirconia ceramic.

Tukey *post hoc* test revealed a significant difference in values between AA-Z ($4.60 \pm 1.08\ \text{MPa}$) and the remaining groups (ranging from 13.38 ± 2.57 to $15.78 \pm 2.39\ \text{MPa}$, $p < 0.05$).

Table 4 presents the residual composite on the surfaces as evaluated by ARI scores. The Fisher's exact test indicated significant differences among the groups ($p < 0.001$). For AA-Z, most of the adhesive remained on the bracket (scores 0 and 1), indicating failure at the ceramic-adhesive interface. Except for AA-Z, most of the adhesive remained on the ceramic surfaces (scores 2 or 3), indicating failure at the bracket-adhesive interface. No ceramic fractures were observed in this study.

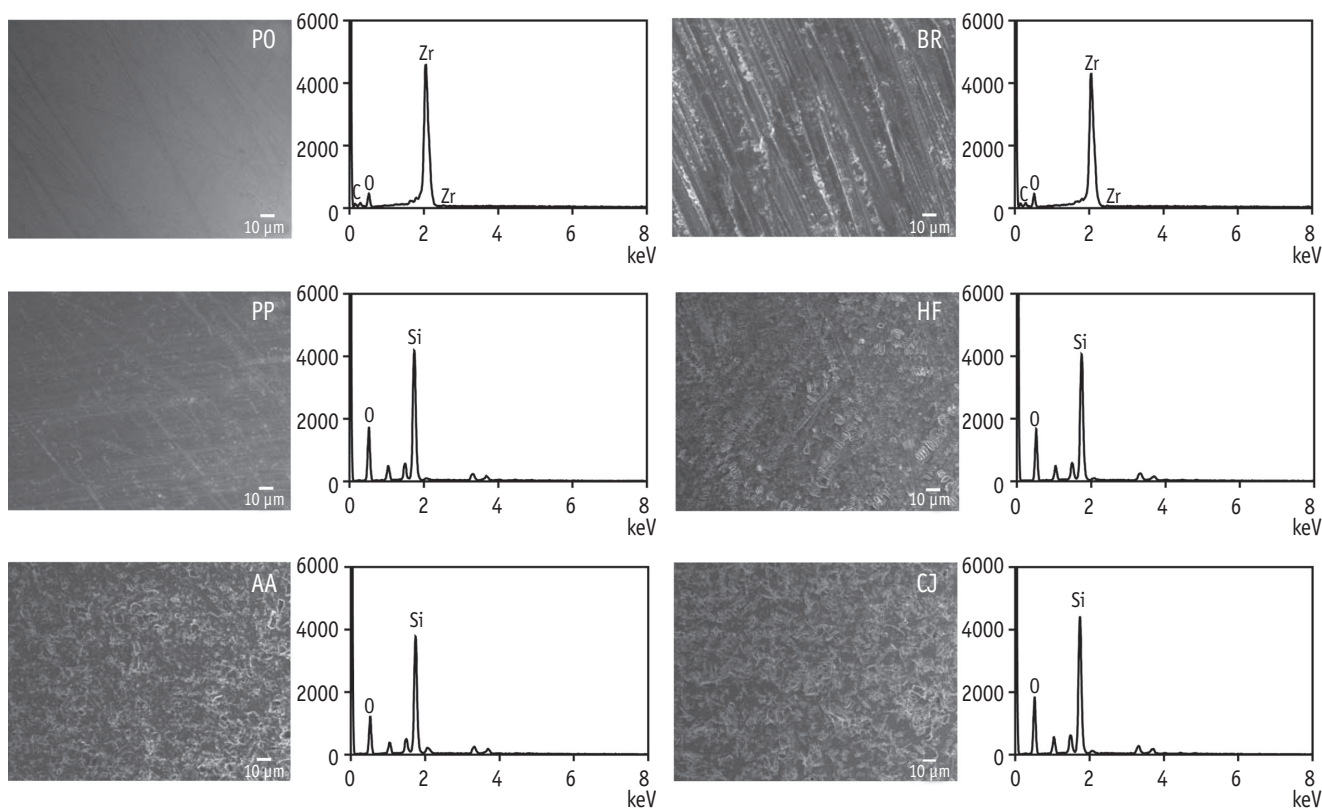


Figure 2. Topographic SEM images and corresponding EDS spectra of the surfaces subjected to 6 different pre-treatments. Note that only the PO and BR spectra show Zr peaks. PO, polishing unglazed zirconia; BR, bur roughening; PP, cleaning with a prophy cup and pumice; HF, hydrofluoric acid etching; AA, air-particle abrasion with aluminum oxide; CJ, silicon dioxide [CoJet-Sand] particles.

Table 2. R_a surface roughness (μm) after surface pre-treatment

Surface pre-treatment	<i>n</i>	Mean	SD	Min	Max
PO (polishing unglazed zirconia)	10	0.06 ^a	0.02	0.05	0.09
BR (bur roughening)	10	1.25 ^e	0.24	0.85	1.64
PP (cleaning)	10	0.24 ^b	0.05	0.16	0.34
HF (hydrofluoric acid etching)	10	0.32 ^c	0.05	0.24	0.39
AA (alumina air-abrasion)	10	0.51 ^d	0.09	0.39	0.67
CJ (CoJet-Sand air-abrasion)	10	0.60 ^d	0.08	0.40	0.69

Welch's variance-weighted one-way ANOVA showed a significant difference in mean R_a value among the groups ($p < 0.001$); the Games-Howell *post hoc* test showed that means with the same superscripted letter were not significantly different ($p > 0.05$). SD, standard deviation; Min, minimum value; Max, maximum value.

Table 3. Shear bond strength values (MPa) for each group

Group	<i>n</i>	Mean	SD	Min	Max
PO (polishing unglazed zirconia)-Z*	10	13.38 ^a	2.57	9.75	18.22
BR (bur roughening)-Z	10	15.48 ^a	3.15	10.68	19.92
PP (cleaning)-S [†]	10	14.90 ^a	2.75	10.93	18.91
HF (hydrofluoric acid etching)-S	10	15.24 ^a	3.36	8.49	20.16
AA (alumina air-abrasion)-S	10	15.78 ^a	2.39	11.23	19.02
AA (alumina air-abrasion)-Z	10	4.60 ^b	1.08	2.76	5.98
CJ (CoJet-Sand air-abrasion)-S	10	14.81 ^a	2.91	10.01	19.75

*Z-Prime Plus; [†]Monobond-S.

One-way ANOVA showed a significant difference in mean shear bond strength among the groups ($p < 0.001$); the Tukey *post hoc* test showed that means with the same superscripted letter were not significantly different ($p > 0.05$).

SD, standard deviation; Min, minimum value; Max, maximum value.

Table 4. Distribution of ARI scores for each group

Group	<i>n</i>	ARI score [‡]				Fisher's exact test
		0	1	2	3	
PO (polishing unglazed zirconia)-Z*	10	0	3	2	5	$p < 0.001$
BR (bur roughening)-Z	10	0	1	2	7	
PP (cleaning)-S [†]	10	0	4	2	4	
HF (hydrofluoric acid etching)-S	10	0	3	2	5	
AA (alumina air-abrasion)-S	10	0	2	1	7	
AA (alumina air-abrasion)-Z	10	8	2	0	0	
CJ (CoJet-Sand air-abrasion)-S	10	0	1	1	8	

*Z-Prime Plus; [†]Monobond-S.

[‡]ARI, adhesive remnant index, 0, No composite left on surface; 1, less than half of the composite left on surface; 2, more than half of the composite left on surface; 3, all composite left on surface, with a distinct impression of the bracket mesh. No ceramic fractures were observed in this study.

Discussion

In this study, 6 experimental (glazed, pre-treated, and primer-applied) groups were tested to simulate clinical situations (Table 1). Bond strength of orthodontic brackets should be high enough to resist accidental detachment during treatment but also low enough that excessive force need not be applied during debonding at the end of the treatment.⁷ Thus, 6 to 8 MPa has been suggested as clinically adequate bond strength for metal bracket to enamel.^{7,20} In this study, except for AA-Z, the mean shear bond strengths of metal brackets to glazed zirconia surfaces submitted to 5 different surface conditioning procedures fell within this range or exceeded these limits and therefore could be considered sufficient for clinical applications (Table 3). Thus, the null hypothesis was partially rejected. BR produced effective bond strength with the subsequent application of a zirconia primer (Z-Prime Plus), whereas PP, HF, AA, and CJ performed well with silane (Monobond-S).

It has been reported that a combination of air-particle abrasion and resin composites with organophosphate ester monomers results in a relatively strong and durable bond to zirconia.¹⁵ Another approach to chemical bonding with zirconia is to use various primers, which seems more suitable for use with non-adhesive orthodontic bonding systems such as Transbond XT. Among various zirconia primers, Z-Prime Plus, which is based on organophosphate/carboxylic acid monomers, has been reported to be highly effective in achieving durable resin bonding to air-abraded zirconia surface.^{15,21} The EDS analysis showed that only bur grinding easily removed the thin (approximately 0.1 mm, Figure 1) glaze layer and exposed the zirconia substructure (Figure 2). A high bond strength (15.48 ± 3.15 MPa) for BR-Z indicates that the application of the zirconia primer to exposed and roughened zirconia by diamond bur grinding is effective for orthodontic bracket bonding. In this study, PO (control) showed comparable shear bond strength (13.38 ± 2.57 MPa), suggesting that the primer works well chemically even with a polished zirconia surface.

The EDS analysis indicated that the bonding substrate in most of the 6 experimental groups (except for BR) was virtually the porcelain glaze layer (Figure 2). Despite the inconvenience of its intraoral manipulation, hydrofluoric acid etching followed by silane application to silica-based ceramic such as porcelain has been reported to be an efficient conditioning method.^{17,22} Strong acids such as 9.6% hydrofluoric acid are commonly used to etch porcelain.⁴ However, highly corrosive hydrofluoric acid can cause severe trauma to soft tissues and tooth substance when used intraorally.^{4,13} In this study, therefore, a low concentration (4%) of hydrofluoric acid was used with a longer application time (5 minutes). Despite a significantly larger R_a value than PP (Table 2), HF showed a relatively smooth surface structure with some

striation formation (Figure 2), rather than the amorphous spongy-like microstructure found on the conventional veneering ceramic surface.²³ This reduced etching effect may be attributable to the use of a low hydrofluoric acid concentration.²³ In terms of bond strength, there was no significant difference between HF and PP (Table 3). These findings suggest that chemical bonding by silane treatment exceeds the mechanical retention provided by hydrofluoric acid, although both steps contribute to final bond strength.⁶

In this study, air-particle abrasion was performed with 30 μ m aluminum oxide particles (used for intraoral) on the glazed zirconia, producing a randomized roughened glazing porcelain surfaces (Figure 2).⁴ Although AA produced a significantly rougher surface than HF, no gain in bond strength was observed even when silane was subsequently applied (AA-S and HF-S), confirming that chemical adhesion by silane treatment might have a greater effect than surface roughening (Tables 2 and 3).⁶ To simulate a possible clinical situation in which a zirconia primer is applied to glazed full-contour zirconia restorations, Z-Prime Plus was applied to the air-abraded surface (AA-Z), yielding a poor shear bond strength (4.60 ± 1.08 MPa). This finding indicates that acidic monomer-containing primer is incompatible with porcelain even though the surface has been significantly roughened (AA vs. PP).^{13,15} The surface of glazed full-contour zirconia restorations might be altered or removed mainly by the dentist's proximal contact adjustment procedures before insertion or by occlusal wear within the first six months after insertion.²⁴ The glazed layer over the labial/buccal surface of a glazed zirconia restoration may also be worn off by long-term tooth brushing. As shown in AA-Z, a zirconia primer should not be used in clinical practice when the porcelain glaze layer appears to remain (Table 3). When it is unclear whether the surface to be bonded with bracket is a porcelain glazed layer or zirconia substructure, thoroughly exposing the zirconia by bur grinding and then applying a zirconia primer would be preferable.

It has been reported that tribochemical silica coating with subsequent silanization enhances the bond between the ceramic surfaces and the resin composite, the silica layer left on the ceramic surface by such coating providing a basis for silane.⁷ In terms of bond strength, the current study found no additional effect of this treatment compared to air-particle abrasion (Table 3). As the porcelain glaze layer was already rich in Si, such tribochemical silica coating did not considerably increase the amount of silane-reactive Si (Figure 2).

Since no recommendations or standards for glazing dental zirconia are found in the literature at present,²⁵ the quality (particularly the thickness) of the glaze layer may vary depending on the glazing technique used. Nonetheless, the silane chemistry remains effective on porcelain glaze

layer irrespective of thickness.¹³ In this study, brackets for the mandibular incisors were used because their flat bases ensured optimal adaptation to the flat glazed or unglazed zirconia surface,⁷ but only metal brackets were employed. The use of ceramic or polycarbonate orthodontic brackets is more desirable on zirconia-based restorations in esthetic terms. Thus, the bond strength of such tooth-colored brackets to zirconia ceramic should be further investigated.

In this study, shear bond strength was tested using the wire loop method. Mojtahedzadeh *et al.*¹⁹ suggested that this method might more closely reproduce clinical loads than the shear blade method. In addition, thermocycling (1,000 cycles) was applied prior to debonding to approximate clinical reality.¹⁷ When the specimens were stored only in water without thermocycling, the bond strengths to ceramics and the incidence of cohesive ceramic fractures were found to be excessively high.¹⁹ In this study, no ceramic fractures were found, probably indicating that the exposure to water and temperature changes involved in thermocycling mainly affected the bond of the resin composite to the metal bracket base (Table 4).¹⁸ However, de Oliveira *et al.*²⁶ reported that the bracket bond strength to enamel was significantly improved after thermocycling. Further studies are required to determine whether longer thermocycling regimens are needed.

This study suggests that prior to orthodontic bracket bonding, a simple surface cleaning of the zirconia glaze layer with a prophyl cup and pumice followed by silane application suffices. Additional roughening of the glaze layer did not improve the shear bond strength of the brackets. Moreover, a roughened surface can be expected to promote bacterial accumulation if it is not well-covered by a bracket after bonding.²⁷ Such simple surface cleaning may be replaced by phosphoric acid conditioning, which produces no morphological changes to porcelain, only removing its smear layer.⁶ Nonetheless, the former would be a safer choice of surface cleaning than the latter (i.e., intraoral use of an acid). Recently, multi-function primer for working on all types of restoration surfaces and universal adhesives for treatment of tooth surfaces as well as restoration surfaces have been launched. These materials were not tested in this study; further tests should be performed to prove their effectiveness in orthodontic bracket bonding to glazed zirconia.

Conclusions

It was found that the most simple and effective surface conditioning method for orthodontic bracket bonding to glazed zirconia was a silane application to the cleaned surface in the clinic.

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Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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